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ON GENERAL AND APPLIED CHEMISTRY

BOOK OF ABSTRACTS

IN 7 VOLUMES

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XXII MENDELEEV CONGRESS ON GENERAL AND APPLIED CHEMISTRY

*Dedicated to the 190th anniversary
of D.I. Mendeleev and the 300th anniversary
of the Russian Academy of Sciences*

BOOK OF ABSTRACTS

Volume 2

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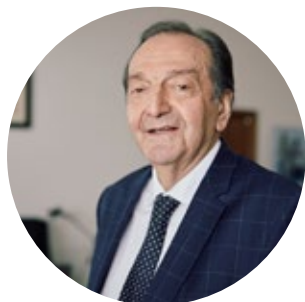
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SECTION 3

PHYSICO-CHEMICAL BASES OF METALLURGICAL PROCESSES

BEHAVIOR OF CHEMICAL ELEMENTS DURING STEELMAKING IN ELECTRIC FURNACES

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Modern steelmaking processes take place in two stages, oxidizing in the main steelmaking unit, converter or electric arc furnace (EAF) and reducing in ladle processing.

The analysis of large-tonnage EAF heats showed that as a result of oxidation refining, the yield from metallic charge to a liquid metal is 84-93% and depends on the operating practice of a particular furnace and, to a lesser extent, on the steel grade. The coefficients of assimilation of elements for low- and medium-carbon grades along the technological chain from EAF to CCM were estimated. The instability of the scrap composition in the EAF charge makes a great contribution to the dispersion of the results obtained, variations in the scrap composition are illustrated by the copper content in the metal at the metal tapped from EAF¹.

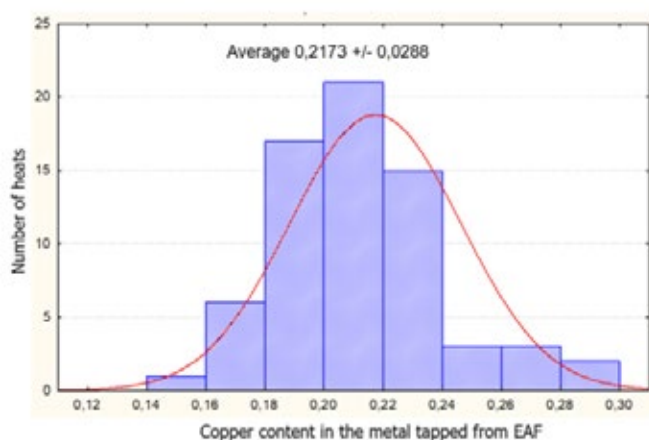


Figure 1. Distribution of copper concentration in the metal tapped from EAF.

The obtained coefficients of assimilation of elements in EAFs are estimated, but more accurate determined for ladle processing and casting, for example, for manganese: EAF – 0.11; ladle furnace - 0.50 and continuous casting – 0.98.

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MODERN METHODS OF CONTROL AND ADJUSTMENT OF STEEL PRODUCTION TECHNOLOGIES

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The development and optimization of industrial metallurgical technologies for steel production is a complex process that requires significant material and time costs. One of the most important controlled parameters of steel quality is the purity of impurities and non-metallic inclusions with the determination of their composition, morphology and size distributions. A method was developed for analyzing and optimizing technologies for the production of high-quality steels, including the use of fractional gas analysis (FGA), scanning electron microscopy with X-ray microanalysis and mathematical computer modeling. Mathematical computer modeling of technologies, which makes it possible to adequately describe real high-temperature chemical and physical processes in metallurgical units, significantly reduces the cost of optimizing existing and developing new steel production technologies. Therefore, the development of complex dynamic models of smelting and after-furnace processing units, including models of individual units, is a very relevant area of research. The use of developed methods of control and optimization of technologies will ensure an increase in the quality of steel and a reduction in its cost, high stability of production and the competitiveness of the enterprise in the metal market.

The report will show the possibilities of combined use of PGA methods, microprobe analysis and PDA optical emission spectrometry to control the cleanliness of steel. The possibilities of the developed method for optimizing out-of-furnace steel processing technologies will be discussed. Examples of analysis and adjustment of technologies for smelting and ladle processing of steel of various grades under existing production conditions will be given.

THE EFFECT OF HYDROGEN ON THE MECHANICAL PROPERTIES OF PIPE STEELS

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The growing interest of industry and transport decarbonization induces search of alternative energy sources for development a green energy segment. An important aspect of this concept is the utilization of hydrogen, which does not contribute to the formation of greenhouse gases. However, study on the applicability of materials for hydrogen production, transportation, storage, and direct usage is required due to special physical and chemical parameters.

The report presents generalized data on various types of the properties degradation of metals during interaction with hydrogen such as high-temperature hydrogen attack and hydrogen embrittlement. The key mechanisms of hydrogen influence on the various tube steels are discussed.

The results of the study on the behavior of materials interacting with a hydrogen-containing environment obtained at the research centers of TMK (RusNITI and TMK R&D) are presented. The regularities of metal hydrogen saturation from the gas phase were determined as a function of applied stress levels and plastic deformation using low-carbon steel. An assessment of the resistance to hydrogen embrittlement of steel for large-diameter pipe was conducted under a hydrogen pressure of 10 MPa. The effect of hydrogen charging on the mechanical properties of medium-carbon pipe steels under hydrogen sulfide corrosion was determined. The relationship between the degree of mechanical properties degradation and the strength level was established under intensive electrolytic hydrogen charging of medium-carbon steel. Concept of alloying were determined to achieve an optimal combination of high strength with resistance to hydrogen embrittlement.

A draft standard for seamless steel pipes for transporting gaseous hydrogen and for steel cylinders for its storage, transportation, and utilization has been developed by the Technical Committee on Standardization TC357 "Steel and Cast Iron Pipes and Cylinders" based on the analysis of contemporary knowledge and research results. In the development process, the requirements of ASME 31.12 "Hydrogen piping and pipelines", IGC DOC 121/14 "Hydrogen pipeline systems", and several other international standards were taken into account, along with the results of our own research.

PHYSICO-CHEMICAL FEATURES OF THE DEVELOPMENT NEW GENERATION OF FERROALLOYS

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The main products of ferroalloy plants are standard ferroalloys. It often do not have all the necessary service characteristics. Therefore, the developing progressive technology of steelmaking is forced to adapt to the existing range of ferroalloys, standards for which have not been updated for several decades. In addition, the sources and markets of ferroalloy raw materials have changed. Their quality and the content of the leading elements have decreased, which makes it difficult or impossible to obtain ferroalloys according to existing standards. In this regard, it is necessary to create more efficient ferroalloys of a new generation, suitable for progressive processes in developing areas of metallurgy and smelted from available mineral and man-made raw materials.

The Institute of metallurgy the UB of the RAS has developed a comprehensive approach to assessing the physicochemical characteristics of ferroalloys, that responsible for the assimilation of elements in liquid steel and its quality. It including the determination of: the temperatures of the beginning and end of melting; density; kinetics of oxidation; the amount and shape of non-metallic inclusions formed in the processed melt; the thermal effect of interaction of ferroalloys with an iron-carbon melt; the duration of melting ferroalloy, its mechanical and thermal characteristics.

The work demonstrated that it is possible to create new processes and combinations of different types of ferroalloys from non-traditional domestic ore raw materials based on deep physico-chemical and technological research, which are not inferior in their technical and economic indicators to products obtained from rich imported materials.

The scheme to developing ferroalloys of a new generation is based on a comprehensive assessment of the metallurgical characteristics of existing mineral and man-made raw materials; the development of new resource- and energy-saving chemical-technological and metallurgical processes; the study of the physico-chemical characteristics of the ferroalloys obtained and the assessment of the quality of the steel process.

The work was carried out according to the state assignment of the Institute of metallurgy the UB of the RAS

PHYSICAL AND CHEMICAL BASICS OF CREATION OF INNOVATIVE TECHNOLOGICAL SOLUTIONS FOR THE PRODUCTION OF NEW GENERATION STRUCTURAL STEEL WITH A COMPLEX OF INCREASED PROPERTIES

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Among the priority tasks for the development of the country's metallurgical complex, the problem of improving the quality and reducing the cost of metal products remains relevant. Improving the quality characteristics of structural steels is carried out at all technological stages of steel production. The thermodynamics of the phosphorus oxidation reaction, macrokinetics of oxidation processes, phase composition, structure and physicochemical properties of multicomponent slags of the $\text{CaO-SiO}_2\text{-FeO-MnO-P}_2\text{O}_5\text{-MgO}$ and $\text{CaO-SiO}_2\text{-B}_2\text{O}_3\text{-Al}_2\text{O}_3$ system, including viscosity, equilibrium interphase distribution of sulfur and boron during out-of-furnace processing of steel.

The results of fundamental research form the basis for the development of innovative technological solutions that provide:

- smelting of intermediate steel in oxygen converters and modern EAFs under magnesium slag of rational composition with a guaranteed low phosphorus content and high durability of the refractory lining of steel-smelting units;
- deep desulfurization and direct microalloying of structural steel grades with boron in ladle-furnace installations using environmentally friendly boron-containing slag.

The introduction of developed innovative technological solutions ensured the production of low-carbon boron-containing structural steels of a new generation, sparingly alloyed with manganese, with low phosphorus and sulfur content and a complex of increased mechanical properties, incl. for large-diameter pipes of strength category X80 without heat treatment with the prospect of reaching strength category X100-X120.

INFLUENCE OF SLM PROCESS TECHNOLOGICAL PARAMETERS ON TEXTURE OF THE AUSTENITIC ALLOY 03CR17NI14MO3

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It is known that the technological parameters of laser powder bed fusion or selective laser melting (L-PBF/SLM) affect the structural characteristics of material, including its texture¹. In this work, the effect of a protective atmosphere on the structure of the austenitic alloy 03Cr17Ni14Mo3 after L-PBF was studied.

Steel samples were synthesized from powder with a dispersion of $20 \div 50 \mu\text{m}$ on Trumpf TruPrint 1000 installation with a laser power of 110 W, a scanning speed of 750 mm/s, a distance between laser tracks of $50 \mu\text{m}$, and a chess melting strategy. N_2 was used as a protective atmosphere for the first batch of samples, and Ar for the second.

Samples obtained in various protective atmospheres were subjected to comparative structural analysis using metallography, scanning electron microscopy, and X-ray diffraction methods, and their microhardness was also determined.

As a result, it was revealed that the only significant difference between the structures formed by L-PBF in different protective atmospheres is their different texture: in the samples synthesized in the N_2 environment, the laser scanning plane predominantly coincided with the crystallographic plane of $\{100\}$ type, and in the Ar environment with the plane type $\{110\}$ (Fig. 1).

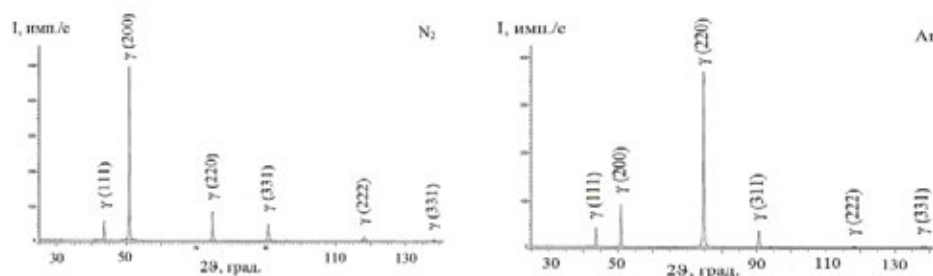


Figure 1. Diffraction patterns of 03Cr17Ni14Mo3 steel samples after L-PBF in N_2 and Ar.

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FEATURES OF INTERMETALLIDE FORMATION WITH TRANSITION AND RARE-EARTH METALS

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Aluminum alloys alloyed and modified with transition metals along with rare earth metals are increasingly finding their application for strategic materials. The latest technologies use the unique physical properties of rare earth metals (REM) in conjunction with transition metals. Currently, complex alloys of light metals are obtained by direct smelting of components, aluminothermic reduction of oxides, fluorides, nitrides and halides.

The process of producing aluminum alloys synthesized by reducing REM compounds and transition metals in an aluminum-magnesium alloy with the formation of intermetallic compounds of given composition has been studied. With the help of special developed computer programs, thermodynamic conditions were determined for the formation of intermetallic compounds with REM and transition metals in the melts of halides with the necessary relation of each element.

Studies of the endogenous process of producing aluminum ligatures with transition and rare metals show that intermetallic compounds are synthesized in a shape approximately rectangular, and in their central part there is a free space filled with matrix material containing 0.2-1.0% of transition metal. In case of presence of erbium and ytterbium compounds in the charge, coagulated crystals consisting of Mn_3Er and Mg_3Yb are synthesized. By changing the composition of the initial reagents, additives of alloying rare elements, the selection of various process modes (temperature, ultrasound, mixing, etc.) and it is possible to predict in advance the structure, technological and operational characteristics of the synthesized aluminum-based alloys.

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METAL REFINING AT ESR

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An effective technology for obtaining steels and alloys with a low concentration of non-metallic inclusions is EAF, where the refining medium is a working flux. The process involves three boundaries of interaction of liquid metal with refining medium: metal film on the surface of the melted electrode - flux, a drop of electrode metal - flux, the mirror of the liquid metal bath - flux. The degree of refining at these boundaries is not equal. The main refining takes place on the melted surface of the electrode, when the liquid metal film is formed. Reduction of inclusions concentration on this boundary is proportional to their size in the liquid metal film - the thinner the film, the more complete the refining. First of all, the inclusions, the size of which is equal to or exceeds the film thickness, are eliminated. The size of the liquid electrode metal film on the melted surface of the electrode is not the same, its thickness increases as it approaches the axis of the remelting electrode. That is, the areas of the remelting electrode most contaminated with inclusions are in the worst refining conditions. It is necessary to provide thinning of the liquid metal film on the sent surface of the consumable electrode, especially in the area of the highest contamination without reducing the already low productivity of the process. It is also necessary to reduce the size of electrode metal droplets and increase the time of their contact with the working flux, to exclude the delivery of droplets into the liquid bath along the axis of the formed ingot. To create conditions for ensuring a non-deep, uniform in height metal bath, which will allow to create an axial crystalline structure and exclude blocking of inclusions floating. Among the studies aimed at solving the problem under consideration, it is worth mentioning the technology with rotation of the consumable electrode, which is well-proven in production conditions. By controlling the centrifugal forces arising in this case, it is possible to solve the problem of reducing the concentration of non-metallic inclusions with the size of 10 microns and less, while retaining all the advantages of electroslag technology. Obtaining a shallow flat liquid metal bath creates favorable conditions for the formation of multilayer billets, which can serve as a new area of technology development.

INFLUENCE OF MICROALLOYING ELEMENTS ON PHASE PRE-CIPITATION DURING THERMAL DEFORMATION OF HIGH-STRENGTH LOW-CARBON STEELS

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Nowadays, global and domestic automotive industry is enjoying an upward trend in the consumption of hot-rolled and cold-rolled high-strength microalloyed steel. Active research is ongoing in various areas to develop cost-effective alloying options ensuring the targeted mechanical properties. Special focus is given to studying how chemical composition and parameters of thermal deformation treatment influence the formation of a system of nanoscale phase precipitates of different types and the resulting mechanical properties.

The paper summarizes the research data on hot-rolled and cold-rolled high-strength low-carbon steels microalloyed with Ti, Nb, V, Mo in various combinations. Depending on the microalloying system and thermal deformation treatment parameters, nanosized carbide (carbonitride) precipitates of several types are present in the microstructure of rolled steel in varying quantities. Austenitic precipitates cause the formation of fine-grained micro-structure, while ferritic precipitates contribute to precipitation hardening. Interphase precipitates generated in the $\gamma \rightarrow \alpha$ transformation process contribute to both hardening mechanisms.

To analyse the type and quantity of nanoscale phase precipitates present in rolled steel, transmission electron microscopy and thermodynamic calculation of temperature dependences of equilibrium phase composition of steels were used. It was found that titanium and vanadium have the highest susceptibility to the generation of interphase precipitates. The presence of molybdenum in steel composition accelerates nucleation of carbide (carbonitride) precipitates, but restrains their growth. It has been demonstrated that titanium has advantages over vanadium in the development of a cost-effective microalloying system.

PHYSICO-CHEMICAL AND THERMOPHYSICAL FOUNDATIONS OF DIGITAL TWINS OF AGLO- COKE-BLAST FURNACE PRODUCTION AND THEIR APPLICATION IN PRACTICE

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The role of mathematical models in improving the technology of blast furnace smelting is shown. Examples of new developments of the Institute of Metallurgy of the Ural Branch of the Russian Academy of Sciences in the field of digital models of blast furnace production are given, in particular, one-dimensional, two-dimensional and three-dimensional mathematical models of various zones of the blast furnace, including analysis and prediction of phenomena of gas dynamics, heat transfer, reduction in the blast furnace¹; monitoring of the condition of refractory lining and filling of the furnace hearth; prediction of silicon content in cast iron and slag based on neural network technologies; optimization of the composition and improvement of the metallurgical characteristics of iron ore raw materials (agglomerate, pellets) and coke in order to increase the efficiency of blast furnace smelting and analysis of the efficiency of blast furnaces based on the calculation of the minimum coke consumption^{2,3}. These models are based on the physico-chemical and thermophysical phenomena occurring in a blast furnace, taking into account their two-dimensionality inherent in setting a task. New developments in the field of analysis and control of phenomena occurring in various zones of the blast furnace allow us to raise the technology and controlling methods of blast furnace smelting to a fundamentally new level, allowing us to save fuel and energy resources. The possibility of using a digital model at a pace with the process when using sensor readings through the database management system of the blast furnace workshop of the metallurgical plant is shown.

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FORMATION OF STRUCTURE AND PROPERTIES OF WELDED JOINTS OF STEEL PIPES

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Systematic studies of the structure of the base metal and welded joints of pipes \varnothing 60–1420 mm from low-carbon microalloyed steels produced using various types of welding (submerged arc and high-frequency) were carried out; the main connections between their structure and properties were established, microstructural parameters were determined and ranked mechanisms that determine the resistance to destruction of the base metal and welded joints during the studied types of welding.

An increase in the cooling rate of the coarse-grained HAZ region during welding is accompanied by an increase in the density of high-angle boundaries in the microstructure, which is due to the replacement of granular bainite by lath bainite and an increase in the contribution of grain boundaries of former austenite.

The influence of the parameters of the thermal cycle of welding and the corresponding change in the microstructure on the impact toughness of the HAZ of steels can be limited in the case of exposure to stronger embrittlement factors, for example, non-metallic inclusions that act as sites of initiation of brittle cracks.

It has been shown that groups of adjacent ferrite grains (crystallographic clusters), in which the $\{001\}$ planes are close to the potential fracture surface ($\pm 20^\circ$), contribute to the formation of transgranular cleavage. In a high-frequency welded joint, a decrease in impact toughness and cold resistance is associated with a 2–3-fold increase in the proportion of $\{001\}$ clusters along the fusion line in comparison with the base metal, while the size of the largest fraction of clusters is approximately 5 times larger than the size of the largest grains α –phases.

In arc welding, the greatest negative effect on cold resistance is exerted by large $\{001\}$ clusters up to 160 μm in size, located in the center of the weld in areas of grain-boundary ferrite.

Ways to improve the structure and properties of welded joints have been proposed, new and improved technologies and steels have been developed, tested and introduced, mass production of new types of pipe products with a unique combination of operational properties has been mastered.

STUDYING DIRECT PROCESSES RESTORATION OF IRON ORE CONCENTRATES IN A HYDROGEN ENVIRONMENT

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One of the promising directions for making the process of producing iron in various units more environmentally friendly is the expansion of the use of hydrogen in carrying out technological processes.

In order to assess the possibility of direct reduction of iron ore concentrate with hydrogen, a set of laboratory studies was carried out. Magnetite iron ore concentrate and magnetite concentrate pre-oxidized at a temperature of 800°C were used as starting materials for studying the process of direct reduction of iron ore concentrate with hydrogen.

During the research, the chemical and granulometric composition of the concentrates was studied, and the microstructure was analyzed. The reduction process was studied on an SDT Q600 thermal analyzer with a constant supply of argon at a flow rate of 100 ml/min and hydrogen at a flow rate of 6 l/h. Reduction was carried out by heating to 1000°C at a constant rate of 20°C/min.

Granulometric analysis showed that the oxidation of the initial magnetite concentrate in a static furnace at a temperature of 800°C leads to an increase in the average particle size from 19.0 μm to 26.6 μm, which indicates the enlargement of concentrate particles during oxidative roasting. Analysis of the microstructure showed significant differences in the size of the concentrate particles, it should be noted that the particles of the original concentrate have sharp particle edges, while the oxidized concentrate has smooth edges and some of the particles have merged, possibly due to fusion.

The thermogravimetric analysis showed the possibility of carrying out the process of direct reduction of both the original magnetite concentrate and the one previously oxidized at a temperature of 800°C.

The analysis established that, both from the point of view of energy costs and the speed of the process of direct reduction of iron ore concentrate with hydrogen, it is most advisable to use pre-oxidized concentrate.

MATERIALS SCIENCE APPROACH TO THE DEVELOPMENT OF NEW TECHNOLOGIES AND STEELS FOR PIPE PRODUCTION

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Scientific developments for the production of both welded and seamless pipes are associated, first of all, with the search for optimal compositions of alloying elements, taking into account the production technological chain.

Within the framework of this work, approaches to the creation of new pipe steels are considered, taking into account production technology, from laboratory research to industrial testing. Using examples of laboratory experiments using metallurgical approaches to structural studies at various stages of production, the possibility of producing welded and seamless pipes with increased resistance to hydrogen cracking and increased cold resistance based on low-carbon low-alloy steels with various micro-alloying systems is shown. It has been revealed that it is possible to achieve promising characteristics in welded pipes both with and without the use of volumetric heat treatment of pipes. In seamless pipes, the optimal combination of corrosion and mechanical properties in low-alloy steels is achieved after extensive heat treatment consisting of quenching and tempering. Microstructural studies and thermodynamic calculations have shown that the use of niobium and vanadium as microalloying additives, although it can lead to comparable effects in terms of influence on precipitation hardening, however, the mechanisms of influence on the final structure of pipes can differ significantly.

Part of the work was carried out as part of a comprehensive project on the topic “Development and implementation of integrated technologies for the production of seamless pipes from new generation steels with controlled corrosion resistance under difficult operating conditions for the fuel and energy complex of the Russian Federation” within the framework of Agreement No. 075-11-2023-011 dated 02/10/2023 according to Decree of the Government of the Russian Federation No. 218 dated 04/09/2010

MODERN DIRECTIONS OF POWDER METALLURGY

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The work examines modern and promising trends in the field of powder metallurgy, applied to heat-resistant alloys used for the manufacture of the most critical and heavily loaded structural elements. Powder metallurgy technology includes the following main operations: obtaining initial powder materials; formation of powders into workpieces with specified dimensions; Heat treatment.

One of the first powder metallurgy is granular metallurgy, which combines the solidification of the melt in the form of microingots - granules - with a high rate of crystallization and their subsequent consolidation in forming equipment to a pore-free state. To compact granules, methods of hot isostatic pressing (HIP), vacuum hot pressing, plastic deformation, or a combination of these (isostatic stamping) are used. One of the main advantages of granular metallurgy is the ability to control phase and structure formation, and, consequently, the mechanical characteristics of alloys, through the use of powder inclusion of a pulsed granulometric composition process, and the conclusion of HIP modes and heat treatment conversion.

A promising and actively developing direction in the production of products from powder materials is additive manufacturing (AM). AM represents the integrity of the technology for creating three-dimensional physical bodies based on data from initial electronic models by layer-by-layer addition of material using selective underwater energy (laser or electron beam, electric arc). Interest in AM technology is driven by the production of complex-profile parts with high structural thickness and relatively low surface roughness. In addition, AM technology makes it possible to realize the possibilities of topological optimization of parts and assemblies, which ensures wide-dimensional and design characteristics of products. In the process of layer-by-layer synthesis, melting of the powder material occurs and subsequent crystallization at high cooling rates. As a result, the material is subjected to significant thermal stresses, structural anisotropy, and the possible presence of defects in the form of pores and cracks. To eliminate these shortcomings, various thermal post-processing is used, including HIP, annealing and hardening followed by aging.

SHS TECHNOLOGY FOR PROCESSING DUSTS OF FERROALLOY PRODUCTION

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Russia is a major producer of ferroalloys (Fig.1), during the manufacture of which a significant amount of various waste and substandard materials are generated that require recycling.¹ Fine ferroalloys in the form of cyclone and aspiration dusts and various powdery materials do not find consumers and are difficult to recycle using traditional methods. At the same time up to 50 thousand tons of cyclone dusts of various ferroalloys are formed annually in Russia, as well as up to 400 thousand tons of other powdery fractions in the form of screenings, sweeps, spills, etc. For their efficient processing, a metallurgical SHS process² has been proposed, characterized by high energy efficiency and environmental friendliness. As a result of laboratory and industrial tests, the metallurgical SHS was recommended for processing cyclone dusts and screenings of large and small-tonnage ferroalloys, with the production of commercial nitrided ferroalloys and composite materials based on nitrides, borides and carbides for the steel and refractory industries.

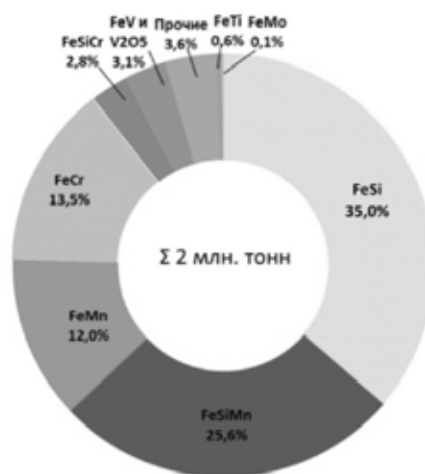


Figure 1. Structure of ferroalloy production in Russia (2023)

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SYNTHESIS AND PROCESSING OF TUNGSTEN-BASED POWDERS IN THERMAL PLASMA

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The paper describes plasma technologies and equipment developed at IMET RAS for the production of nanopowders W, W-5Ni-2Fe, W-20Cu, W-7C, W-7C-10Co, as well as micro-powders of these materials consisting of spherical particles.

All nanopowders were obtained in the flow of hydrogen-containing thermal plasma of an electric arc plasma torch from metal oxide powders. Nanoparticles in plasma chemical synthesis are formed as a result of chemical condensation from the gas phase during cooling of a plasma jet propagating in the cooled volume of the reactor.

The resulting nanopowders consist of aggregated nanoparticles whose size does not exceed 100 nm. Pseudoalloy nanoparticles have a core (W) –shell (Ni-Fe/Cu) structure due to the strong difference in condensation temperatures of individual metals.

The obtained nanopowders W, W-Ni-Fe, and W-Cu were used to prepare aqueous nanosuspensions, from which the corresponding nanostructured microgranules were obtained by spray drying. The yield of microgranules of a fraction of 20-60 microns during spray drying was more than 50%.

Nanostructured microgranules were processed in the thermal plasma of an electric arc plasma torch for densification and spheroidization. As a result, micro-powders W, W-Ni-Fe, W-Cu, (W-C)-Co consisting of dense spherical particles were obtained. The microstructure of the particles is characterized by grain sizes mainly in the micron size range.

Plasma technologies make it possible to produce tungsten-based powders both in the form of nanopowders with a particle size of less than 100 nm and micro-powders consisting of particles of 20-50 microns in size to obtain compacts by various methods, including modern additive technologies.

The work was carried out with the financial support of the RSF, project 22-19-00112.

FORMATION OF A RATIONAL SLAG COMPOSITION IN THE LADLE-FURNACE UNIT IN ORDER TO IMPROVE DESULFURIZATION AND REDUCE THE CONSUMPTION OF SLAG-FORMING MATERIALS DEPENDING ON THE STEEL GRADE

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The paper considers the issues of determining the rational composition of the slag formed in the ladle-furnace unit, which has maximum desulfurizing properties.

Refining properties of slags are determined by the presence of "free" oxygen anions (O^{2-}).^{1,2} In the slag formed in the ladle furnace unit, oxides are present, which are sources of « O^{2-} » (CaO and MgO) and the oxides that absorb them (SiO_2) through education. Additionally, amphoteric oxide Al_2O_3 . Therefore, to determine the basicity index, the formula (1) was proposed in which oxides exhibiting basic properties are summed up, and oxides exhibiting acidic properties are subtracted, the influence of amphoteric oxide Al_2O_3 is additionally taken into account.

$$\Lambda^* = \sum_{i=1}^n (X_i \cdot \Lambda_i^*)_{OCH} - \sum_{i=1}^n (X_i \cdot \Lambda_i^*)_{KHC} + X_{Al_2O_3} \cdot Y_{Al_2O_3} \quad (1)$$

Taking into account the above and as a result of the conducted research, the composition of the slag was determined, which, in the conditions of technological operations of JSC EVRAZ NTMK, has rational refining properties: CaO – 53,3%; Al_2O_3 – 17,8%; SiO_2 – 18,8%; MgO – 8,0%; $FeO+MnO < 1,5\%$. As a result, the following results were achieved: 1) The total consumption of slag-forming materials was reduced by 7.5% 2) Desulfurization for various groups of steel grades was improved from 7.6 to 18.8%.

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PHYSICOCHEMICAL PROPERTIES OF FLUORIDE MELTS, PROMISING FOR NUCLEAR POWER

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One of the key tasks of nuclear power is the industrial-scale reprocessing of spent nuclear fuel (SNF) from nuclear power plants, aimed at minimizing radiation hazards, separating useful substances (U, Pu) for their further use, as well as the safe disposal of unused components that are highly radioactive. For efficient processing of minor actinides, it is proposed to use molten salt nuclear reactors (MSR), which have a number of economic and environmental advantages, as well as increased safety. Promising media for ensuring the operation of MSR are molten fluoride salts based on the FLiNaK (LiF-NaF-KF eutectic) and FLiBe (LiF-BeF₂ eutectic) systems, which are characterized by good thermophysical, neutronic and physicochemical properties.

The paper presents the results of systematic studies of the basic physicochemical properties of molten fluoride mixtures based on the molten eutectic FLiNaK containing fluorides of rare earth metals (REMs). In a wide temperature range, the influence of the REM fluorides additives on the liquidus temperature (measured by thermal analysis methods), density and viscosity (hydrodynamic and rotational methods), and the thermal conductivity and the thermal diffusivity (laser flash method) was determined.

The temperature and concentration dependences of the physicochemical properties of the FLiNaK-(P3M)F₃ systems are in a good agreement with those available in the literature, however, at the same time, the majority of the data were obtained for the first time.

The experimental results attained on model molten systems can be used as the basis for mathematical modeling applying molecular dynamics or quantum mechanical calculations, which will make it possible to determine the optimal compositions of molten mixtures and technological modes, and to evaluate the influence of the heat release of fissile materials on the physicochemical and thermophysical properties.

IMPROVING THE QUALITY OF IRON ORE FEEDSTOCK IS A PROMISING FOCUS AREA FOR RAISING THE EFFICIENCY OF BLAST FURNACE IRONMAKING

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The main quality indicator of any burden material is the stability of all of its characteristics. Of all the characteristics of sinter, cold crushing strength and the content of -5 mm size are mainly considered. The share of -5 mm size in blast furnace burden is reduced by mixing sinter with pellets in the system of loading the blast furnace.

Sinter size distribution largely determines the behaviour of physical and chemical processes in the blast furnace. Coarse size (+25 mm) determines segregation, while the unstable content of this size in the burden makes it difficult to optimize the distribution of ore load over the furnace radius. Fine particles may fill the spaces between coarse particles thus reducing gas permeability of the layer. For a stationary layer, the critical ratio of fine to coarse particle diameter is 0.15, while for a moving layer it can be as high as 0.30.

The larger the diameter of particles, the lower the intensity of heat transfer and recovery. The dependence of the total heat transfer coefficient and recovery rate on the particle diameter is inversely proportional. At the same time, the degree index at the diameter of ore material is 1.3-1.6 (depends on the thermal conductivity coefficient and lump porosity).

Using CFD simulation for the upper zone of the furnace, heat transfer and recovery patterns, it was found that the optimum diameter of iron ore particles is 5 to 20 mm. If blast furnaces are operated with such size distribution, specific consumption of total fuel carbon may be reduced by 3-5 kg per ton of pig iron.

Another way to reduce specific consumption of total fuel carbon is to improve stability of burden chemistry. Reducing the RMS Fe content in iron ore feedstock and basicity by half will curb the consumption of total fuel carbon by at least 5 kg per ton of pig iron.

TEMPERATURE AND CONCENTRATION DEPENDENCES OF VISCOSITY OF Fe-(B,Si) AND Co-(B,Si) MELTS

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Fe(Co)-B-Si systems are the basis for the industrial production of amorphous and nanocrystalline alloys used as soft magnetic materials. The structure and properties of such alloys largely depend on the structural state of the initial liquid phase. When studying the structural features of liquid alloys and analyzing the interatomic interaction in them, indirect methods are often used, based on measuring the temperature and concentration dependences of their structure-sensitive properties, which include viscosity.

The temperature and concentration dependences of the kinematic viscosity of melts of binary and ternary Fe-(B,Si) and Co-(B,Si) systems were studied in this work using the oscillating-cup method.

For all studied liquid alloys, the temperature dependences of viscosity obtained in heating and cooling regimes coincide and are satisfactorily described by the exponential Arrhenius equation.

The concentration dependences of the viscosity of binary systems (Fe-B, Fe-Si, Co-B and Co-Si) have a non-monotonic dome-shaped appearance, indicating a strong chemical interaction in melts between atoms of different types. The features of viscosity isotherms are indirect confirmation of the structural changes occurring in the melt with varying composition and the possibility of implementing short-range ordering type compounds in liquid alloys.

When studying three-component systems, the concentration dependences of quasi-binary sections $\text{Fe(Co)}_x\text{Si}_{(100-x)}$ - $\text{Fe(Co)}_x\text{B}_{(100-x)}$ were constructed. The viscosity isotherms of melts of each quasi-binary system have a monotonic form and are close to the additive sum of the viscosity values of its constituent binary systems. Apparently, in three-component Fe(Co)-B-Si melts a chemically micro-inhomogeneous structure is realized, the components of which are atomic microgroups with short-range ordering characteristic of alloys of the Fe(Co)-B and Fe(Co)-Si binary systems.

SCHEME FOR PRODUCING FERRONICKEL AND CAST IRON FROM ORE OF THE SAKHARINSKOE DEPOSIT BY COMBINED CARBON-HYDROGEN METHOD

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The results of research on the development of a technological scheme for the processing of Sakharinskaya nickel-containing ore with the production of hydrogen ferronickel, cast iron and waste slag were obtained. Using previously obtained calculation data in laboratory conditions, the implementation of each stage of the proposed technology from the enrichment of feedstock to the production of ferronickel, cast iron and waste slag was confirmed¹. Studies have shown that the reduction of nickel from ore with hydrogen ends at temperatures of 700-900 °C. Under these conditions, iron oxides are reduced mainly to FeO, partially (5-15%) to metallic iron.

When studying the process of reduction of iron and nickel from an oxide melt, the possibility of separating them into various products by precise dosage of the reducing agent was shown.

As a result of laboratory studies on the processing of Sakharin ores using the method of selective hydrogen solid-phase reduction, ferronickel and ferrous slag were obtained. From ore with a nickel content of 0.4-0.8% Ni, ferronickel was obtained with a nickel content in the alloy of 4-15%. It has been shown that nickel recovery reaches 80-85%. The ferrous slag was then subjected to liquid-phase carbothermic reduction to produce cast iron with a carbon content of 4.12%, silicon - 0.86%, chromium - 0.65% and waste slag.

The implementation of the technology at the country's metallurgical enterprises will allow a significant amount of off-balance nickel-containing ores of the Ural region to be included in the active balance and will provide the domestic industry with ferronickel and high-quality charge.

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STRUCTURAL TRANSITIONS IN LIQUID COMPLEX CAST IRONS Fe-25Cr-3C AND TEMPERATURE TREATMENT OF THE MELT

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Original experimental data were obtained on the temperature dependences of kinematic viscosity $\nu(T)$, electrical resistivity $\rho(T)$, density $d(T)$ and surface tension $\sigma(T)$ of the liquid alloy Fe-25%Cr-3%C-2%Ni (liquid cast iron grade ИЧХ28Н2) and Fe-25%Cr-3%C-4%V (liquid cast iron grade ИЧ300Х30Ф4Н5КБ) in the temperature range from the liquidus temperature to 1950K. Liquid cast iron was understood as a micro-inhomogeneous melt undergoing a temperature induced liquid–liquid structure transitions (TI-LLST) at temperature T^* .¹ Analysis of $\nu(T)$, $\rho(T)$, $d(T)$ and $\sigma(T)$ of liquid cast iron made it possible to determine $T^* = 1740$ K (cast iron grade ИЧ300Х30Ф4Н5КБ) and $T^* = 1760$ K (cast iron grade ИЧХ28Н2). Based on $\nu(T)$ of liquid cast iron, the size of dispersed (colloidal) particles was determined to be ~ 25 nm (melt ИЧХ28Н2), ~ 3 nm (melt ИЧ300Х30Ф4Н5КБ). The cluster size $D(T)$ was determined from $\rho(T)$ of liquid cast iron; $D(1700) = 3.4$ nm and $D(1900) = 1.7$ nm for the liquid cast iron grade ИЧХ28Н2 and $D(1700) = 1.7$ nm and $D(1900) = 1.2$ nm for the liquid cast iron grade ИЧ300Х30Ф4Н5КБ.² It was previously discovered that that temperature treatment of a melt of hypereutectic chromium cast iron at 1800 K leads to the destruction of compositionally stable clusters and provides a change in the structure and properties of the ingot³, which confirms the authors' conclusion about the value of T^* .

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FORMATION OF NON-METALLIC INCLUSIONS DURING BUCKET PROCESSING OF 08H18N10T GRADE STEEL

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The work analyzed the technology of smelting, ladle treatment and casting of corrosion-resistant steel 08H18N10T in order to identify factors that adversely affect the quality and pour capacity of steel. The overgrowth of steel filling cups occurs due to the high content of non-metallic inclusions (NMI) in steel, such as oxides of chromium, manganese, silicon, aluminum, and titanium.

To determine the type of non-metallic inclusions (NMI), a study of selected metal samples was carried out by fractional gas analysis (FGA). The FGA allows you to quickly obtain information about the oxygen content distributed in various types of NMI. To determine the chemical composition of non-metallic inclusions and the main types of NMI, a scanning electron microscope with X-ray microanalysis Jeol JHA ISP 100 EPMA was used.

During the work, samples of four 08H18N10T corrosion-resistant steel melts were analyzed, melts No. 1 and No. 2 before changing the technological parameters of steel production, and melts No. 3 and No. 4 after corrective actions.

It is shown that the studied steel samples are dominated by such NMI as: aluminates, oxides of chromium, manganese, silicon and titanium.

It was found that the change in the composition of the coating slag led to a decrease in the size of non-metallic inclusions (smelters No. 3 and No. 4). The morphology and composition of the inclusions have not changed. Aluminum oxide inclusions are almost completely removed from the metal (smelters No. 3 and No. 4). Titanium nitrides (VD3 – VD4 samples) are also removed into the slag.

The presence of extended film inclusions based on titanium oxides in the metal was detected only in metal samples taken during casting, which may be related to the processes of secondary oxidation of the melt. This is probably due to air suction or insufficient work of the coating slag to assimilate inclusions. The number and sizes of film inclusions in the metal were smaller at smelters No. 3 and No. 4. There was no overgrowth of glasses at smelters No. 3 and No. 4.

QUALITY MANAGEMENT OF DUPLEX STAINLESS STEELS

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Duplex stainless steels (DSSs) possess a unique combination of corrosion resistance and high strength. The increasing demand for components made of these steels presents metallurgists with the task of mastering and improving them. Therefore, the aim of this study was to develop an approach to selecting the chemical composition of these steels, taking into account the peculiarities of phase formation during melting, solidification, and subsequent heat treatment to control their operational properties. To achieve this goal, the task of quantitatively evaluating the phase composition of DSSs considering secondary phases was solved; critical points of phase equilibria in the alloy and permissible deviations from them were determined; based on these critical points, an optimization model for selecting the chemical composition of the alloy depending on property requirements was developed.

The methodology for quantitative assessment of the phase composition of DSS is based on selective etching with Beraha reagent and metallographic evaluation. The applied etching method allows for accurate assessment of the amount of austenite, ferrite, determining their sizes, as well as evaluating the volume fraction of σ -phase and carbonitrides. It is shown that the accuracy of this assessment is significantly higher compared to X-ray diffraction method.

Critical points of phase equilibria of DSSs have been determined, the control of which allows obtaining the required combination of mechanical and corrosion properties. The first point, $T_{50/50}$, is the temperature at which austenite and ferrite are in equal quantities. The second and third points, T_{σ}^0 and T_{Cr2N}^0 , respectively, are the temperatures at the beginning of σ -phase and chromium nitride formation, catastrophically reducing corrosion and mechanical properties. The influence of the chemical composition of DSSs of different grades on these points has been studied, and the permissible deviations of critical points from their "ideal" values have been experimentally determined, allowing the production of DSSs with the required properties.

An optimization model has been developed, which is a system of equations linking the proposed critical points of the alloy with its chemical composition, mechanical, and corrosion properties. Examples of practical application of the optimization model for managing the performance of existing and developing DSSs are provided.

ANALYTICAL METHODS IN CHEMISTRY FOR SOLVING METALLURGICAL PRODUCTION PROBLEMS: CONTROL OF TECHNOLOGICAL PROCESSES AND PRODUCT QUALITY

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Determining the qualitative and quantitative composition of steels and alloys is an important part of product quality management at all stages: the conformity of the raw materials to the required brand, the development of new formulations, manufacture, and operation. Methods of analysis of steels and alloys to confirm the chemical composition are regulated according to GOST, while single-element spectrophotometric and atomic absorption methods (usually flame) for sample solutions are more often used. A modern alternative is multi-element methods based on excitation in inductively coupled plasma mass spectrometry or optical emission detection (ICP MS and ICP OES) in combination with gas analysis methods. Using the developed approaches to sample preparation, we obtained results for the determination of impurities in steels and alloys with a metrological assessment of their reliability. The search for approaches to direct analysis of solid samples remains relevant. Therefore, the practical implementation of the method of high-resolution electrothermal atomic absorption spectrometry with a continuous radiation source (HR-CS AAS) is of interest, especially for the determination of metals and nonmetals along molecular lines. In particular, for the determination of S, P along the lines of PO 246.40 nm, sulfur along the line of CS 258.056 nm. For the determination of halogens, GaF, AlCl, AlBr and BaI molecules are used along the lines of 211.248 nm, 261.42 nm, 278.914 nm and 538.308 nm, respectively. In addition, multielement analysis is possible, for example, Cr, Fe and Al (425.433 nm, 425.076 nm and 425.315 nm, respectively). For the control of technological processes and finished products, the non-destructive X-ray fluorescence method has been applied in new technical solutions – with total reflection (TXRF) and micro-XRF with polycapillary optics. Approaches for establishing a database of reference materials have been proposed.

INVESTIGATION OF THE EFFECT OF REFILLING THE MELT INTO THE PROFITABLE PART OF THE INGOT ON THE SOLIDIFICATION PROCESS AND STRUCTURE FORMATION

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Improving the quality of cast metal of large ingots is impossible without knowledge of the mechanism and conditions under which the formation of internal defects of cast metal occurs. One of the easily implemented and reproducible ways to study the processes occurring during the solidification of ingots is physical modeling, which allows us to determine the intensity of the advance of solid and solid-liquid phases, convective mixing and, as a result, the influence of these processes on the degree of development of internal defects.¹

The paper presents the results of a laboratory study of the solidification process and the development of ingot defects when refilling the profitable part with melt, after a certain time interval after pouring the ingot body at different temperatures of the refilled melt. The research was carried out by the method of physical modeling on a laboratory installation (mold mold) of an ingot model weighing 19.6 tons. The melt was poured from above. Sodium sulfuric acid was used as a modeling solution. It was found that refilling the melt into profit contributed to the replenishment of the "interdendritic" space in the ingot body and continued growth of the columnar crystal zone, and as a result led to a dense structure of model ingots. Profit-topping ingots differ from conventional ingots by a dense defect-free structure in the body of the ingot. The shrinkage sink is concentrated in the volume of profit. The results obtained make it possible to develop a technology for differentiated casting of ingots when filling their profits with melt after a certain time interval after pouring the ingot body, which will affect the process of forming the metal structure and reducing defective zones.

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NICKEL AND COBALT EXTRACTION FROM LEACHING SOLUTIONS OF OXIDIZED NICKEL ORES

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Oxidized nickel ores (ONO) account for about 80% of the world nickel reserves. Autoclave sulfuric acid leaching of ONO produces complex polycomponent solutions, which, in addition to nickel and cobalt, contain a significant amount of impurities. The typical composition of the solution is given below, g/l: Ni-3÷6; Co-0.3÷0.6; Zn-0.1÷0.2; Mn-2.0÷3.0; Ca-0.4÷0.6; Mg-6.0÷8.0. Extraction processes are the most effective for separating Co and Ni from these solutions.

The extraction of these metals with mixtures of bis(2,4,4-trimethylpentyl)dithiophosphinic acid (Cyanex 301; HR) with individual amines of various structures (primary, secondary and tertiary) was studied. Mixtures of HR and trialkylamine (TAA) are of greatest practical interest, both from the point of view of effective extraction of cobalt and nickel, and (especially) their re-extraction with sulfuric acid solutions.

The optimal composition of the extractant for the specified conditions is a solution of 0.4 mol/dm³ Cyanex 301; 0.5 mol/dm³ TAA and 20v/o tributyl- phosphate (TBP) in an aliphatic diluent. The main parameters for the group extraction of Co and Ni from leaching solutions of ONO were determined (kinetics of extraction/re-extraction, the effect of acidity on the extraction of Co and Ni, data on the main extraction equilibria were obtained). The extraction of Co and Ni from solutions is at least 98%.

Based on the data obtained, a conceptual technological scheme was developed and recommendations were issued for group separation of nickel and cobalt from sulfate leaching solutions of ONO containing impurities of calcium, magnesium, manganese and zinc.

The proposed process is of undoubted interest in the processing of nickel sulfide raw materials, in particular, for autoclave and bio-leaching solutions, offering a new path in the hydrometallurgy of nickel and cobalt.

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DEVELOPMENT OF HIGH-STRENGTH HEAT-RESISTANT MATERIAL FOR THE HALLS OF PROMISING VVER-SKD TYPE REACTORS

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Improving the efficiency and safety of nuclear energy requires the development of new types of reactors with high efficiency, efficient use of nuclear fuel, long service life, high reliability and safety. A promising direction is the creation of generation IV+ water-to-water power reactors with supercritical coolant parameters.

The analysis of scientific sources and the results of previous studies allowed us to determine the main directions for the development of the structural material of the VVER-SKD reactor vessel, namely a fine-grained composition with a high nickel content based on the Cr-Ni-Mo-V-Nb system with an ultra-low content of harmful and impurity elements. 10KHN5MFBA-A steel was chosen as the candidate material.

The conducted complex of mechanical tests and structural studies made it possible to evaluate the properties of the resulting metal, as well as the features of the microstructure. Conclusions are drawn about the influence of the content of chemical elements: the composition with a nickel content of 5,1% has high characteristics. The fine-grained structure is the main factor ensuring a high level of performance.

At the industrial site of the AEU equipment manufacturer, a technology was developed for the production of experimental step stamping from this steel grade, simulating different wall thicknesses of the reactor vessel shells.

The resulting experimental billet has high strength and viscosity properties and meets the requirements for ensuring a strength category of at least KP65 at a temperature of 450 °C, meets the requirements for metallurgical quality, the content of harmful impurity elements and a score of non-metallic inclusions: no more than 1,0 points by type of point oxides and non-deformable silicates, brittle silicates were not detected. The results of evaluation tests for the long-term strength of forged metal made of 10KHN5MFBA steel with a duration of 500,000 hours showed that the required value of the conditional limit of long-term strength of the metal was provided at a level of more than 300 N/mm² at 450 °C.

INFLUENCE OF THE QUALITY OF CALCIUM-CONTAINING WIRE FILLER ON THE CONTENT AND COMPOSITION OF OXIDE NON-METAL INCLUSIONS IN THE PRODUCTION OF HIGH-CARBON STEEL

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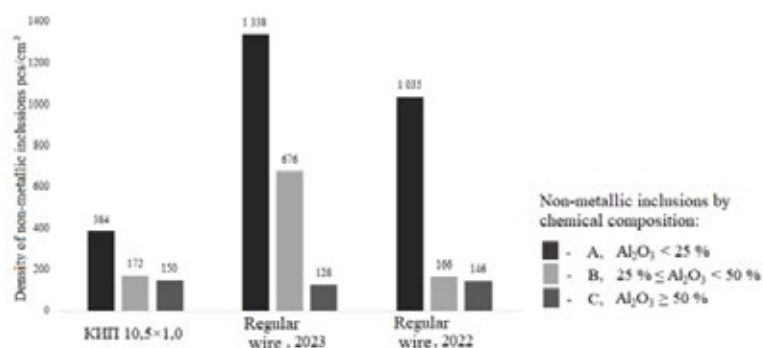
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The mechanical and operational properties of metal products, as a rule, are determined by the quality of the continuously cast billet, which largely depends on the presence of oxide non-metallic inclusions. The composition of such non-metallic inclusions and $[O]_{\text{total}}$ depends on a large number of factors, the main ones of which are the quality of the deoxidizers and modifiers used, the sequence, quantity and method of their introduction¹.

Comparative studies of the density and chemical composition of non-ferrous materials in high-carbon steel not deoxidized by aluminum have been carried out. The experimental melts were treated with calcium injection wire, which is a steel shell with a monolithic filler of various chemical purities - calcium from the electrolytic and aluminothermic methods of production. Samples from liquid steel grades 70/80 were used as research objects. Qualitative and quantitative analysis of oxide non-metallic inclusions was carried out using a scanning electron microscope; the content of total oxygen was determined by the method of reduction melting.

According to the results of the study, it was established that when using calcium by the electrolytic production method, a significant reduction in the total density of non-metallic inclusions and $[O]_{\text{total}}$ is achieved.



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THE USING BORON-CONTAINING MATERIALS IN THE SMELTING OF STAINLESS STEEL BY THE AOD PROCESS

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The main method of producing stainless steel is a duplex process. It consists of smelting a carbon semiproduct in an arc furnace and its subsequent processing in an argon-oxygen decarburization unit (AOD). At first stage the metal is subjected to carbon oxidation, and then the reduction of chromium from the slag with followed by bringing the steel to given chemical composition. The decarburization process is accompanied by partial oxidation of chromium, resulting in slags from the reduction period having a high melting point and viscosity.¹ To improve the fluid mobility of these slags, fluorspar (CaF_2) is used. Its introduction, in addition to reducing viscosity, promotes the formation of harmful volatile compounds. Therefore, it is rational to use other environmentally friendly additives as flux, such as boron-containing materials (colemanite/calcium borate).

In laboratory conditions, an experimental assessment was conducted on the effect of replacing fluorspar with boron-containing material (colemanite) on the physical and chemical characteristics of the slag. Slag composition (40-50% CaO , 25-30% SiO_2 , 17,5% Cr_2O_3 , 8,4% MgO и 3,2% Al_2O_3 , 3-6% B_2O_3) was used in an amount of 14% by weight of steel. It was observed that the formation of slags with 4-6% B_2O_3 provides good fluidity. Additionally, along with a high degree of chromium reduction, steel is microalloyed with boron in an amount of 0.005-0.006%, which positively impacts the operational properties of the metal.

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THERMODYNAMIC MODELING OF THE PROCESS OF MODIFICATION OF NON-METALLIC INCLUSIONS WITH CALCIUM IN LOW-ALLOY STEEL DEOXIDIZED WITH ALUMINUM

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In the conditions of modern steelmaking, the products of steel deoxidization - non-metallic inclusions, do not have a sufficient time reserve for complete flotation and removal from the metal melt. The important technological and operational properties of steel depend on the quantity, composition and morphology of non-metallic inclusions, in particular: the castability of the metal melt, the level of surface and internal defects of rolled steel, the corrosion and mechanical properties of metal products, etc. An effective way to control the chemical and phase composition of non-metallic inclusions is processing melt steel with calcium-containing modifiers¹. The formation of liquid-phase inclusions makes it possible to eliminate cases of clogging of casting refractory and avoid the formation of dangerous macro-accumulations of non-metallic inclusions, speeding up the process of steel refining. The calcium concentration in the steel melt, which is in equilibrium with the liquid phase of non-metallic inclusions, is not constant and depends on a number of factors². Within the framework of this report, the thermodynamic dependences of the target range of calcium content in the melt of low-alloy aluminum-deoxidized steels on the chemical composition and temperature of the melt when treating steel with calcium are considered. The results of theoretical calculations are compared with industrial data on the castability of steel melt and laboratory studies of steel samples.

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STUDY OF MICROLIQUATION HETEROGENEITY AND DISTRIBUTION OF CHEMICAL ELEMENTS IN LOW-ALLOYED STEEL INGOTS AND CONTINUOUSLY CAST BILLETS

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Problems of chemical heterogeneity today are relevant and significant because the development of microliquetation processes is accompanied by the formation of zonal segregation in various parts of the ingot and continuously cast billet (CCB), which leads to a decrease in the quality of the metal and the acceptance characteristics of the billets. Particular difficulties are caused by the presence of microliquetation heterogeneity in the CCB, taking into account the peculiarities of the redistribution of impurities and their solubility under conditions of accelerated cooling.

We studied the features of the formation of microliquetation heterogeneity in laboratory ingots weighing 40 kg from 15KhN2FA steel, cast under different cooling conditions, and in a vacuum ingot weighing 24.2 tons from steel 38KhN3MFA. It has been established that in laboratory ingots the determining factor affecting microliquetation processes is the initial chemical composition, in particular the sulfur content, an increase in the concentration of which promotes microliquetation processes for almost all elements. In a large ingot, changes in the concentration of elements are closely related to the peculiarities of the formation of the macrostructure. A more stable distribution of elements is observed on the lower horizon of the ingot, which is due to accelerated crystallization processes.

When studying the dendritic segregation of round CCB steel 26KhGMFA, it was found that, according to the degree of segregation, the elements are in the following sequence: Mo, Mn, Cr, V, Si. It was found that the segregation coefficients in the ingot are 1.2-1.6 times higher than in the continuous casting, which is due to the peculiarities of thermal processes at the end of solidification of the ingot. To reduce the microliquetation heterogeneity of ingots and continuous castings and reduce possible problems during their heat treatment, it is necessary to create conditions for more intense heat removal of the melt both at the stage of crystallization and in the secondary cooling zone.

The research was carried out at the expense of the funds of the development program of VSTU "Priority 2030", within the framework of scientific project No. 6/643-24.

DEVELOPMENT OF TECHNOLOGY FOR THE USE OF BUCKET SLAG BRIQUETTES AS A PARTIAL REPLACEMENT FOR LIME IN STEEL SMELTING IN EAF

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The purpose of the work: to confirm the possibility of replacing a part of metallurgical lime with slag-forming briquettes based on bucket slag, without deterioration of steel quality and smelting parameters in EAF.

The implementation of this technology will solve the following main tasks:

- reduce the cost of liquid steel by partially replacing lime with slag-forming briquettes;
- eliminate the formation of bucket slag (illiquid material) as a separate type of by-product [1].

During the first stage of the experiment, briquettes of the following sizes were produced 40*30*20 millimeters. Briquettes are manufactured on a briquetting production line using a roller press.

At the second stage of the experiment, experimental smelting was carried out, where slag-forming briquettes were used as a slag-forming flux replacing part of the metallurgical lime of their own production.

During the experiment, there was no deterioration in the parameters of steel smelting in EAF.

A comparison of the chemical composition of the steel of experimental and comparative smelters is shown in Table 1.

Average carbon	Mass fraction of chemical elements, %			
	C	P	S	O ppm
Comparative	0,057	0,004	0,030	691
Experienced	0,056	0,005	0,041	680

Table 1. Chemical composition of steel of experimental and comparative smelts

A comparison of the chemical composition of the slag of experimental and comparative smelters is shown in Table 2.

Average carbon	Mass fraction of slag components, %									
	S	P ₂ O ₅	FeO	MnO	Cr ₂ O ₃	SiO ₂	Al ₂ O ₃	MgO	TiO ₂	CaO
Comparative	0,074	0,41	39,45	6,80	1,25	13,35	3,10	2,11	0,46	26,58
Experienced	0,069	0,36	39,16	6,15	1,39	13,63	5,10	2,99	0,50	25,10

Table 2. Chemical composition of EAF slag before steel production

The slag composition of the experimental smelters does not lead to a deterioration of the dephosphorization process of steel. An increase in sulfur in steel does not affect the final content in the rolled product.

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INVESTIGATION OF EXPLOSION WELDING TECHNOLOGY WITH SIMULTANEOUS EXPOSURE TO ULTRASOUND TO CREATE NEW COMPOSITE MATERIALS

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An urgent direction in the creation of composite materials for various purposes is the search for new ways to form their structure and properties using various combined methods based on the effects of several energy sources or combining various methods of its supply. A comprehensive study of the combined effects of several types of highly concentrated energy sources will reveal the fundamental mechanisms of their influence on the characteristics and behavior of materials under extreme conditions, which creates the possibility of obtaining unique physical, mechanical and operational properties of both the materials themselves and their compounds, which cannot be achieved by external energy sources of a different nature. New prospects for the creation of such materials open up when using ultrasonic action on a substance in both a solid and a liquid state¹.

The introduction of additional ultrasound energy during explosion welding makes it possible to improve the quality of joints, manifested in changes in the structure of the joint zone, an increase in strength, and a significant decrease in the amount of molten metal compared with explosion welding without the use of ultrasound. The technology of explosion welding with simultaneous exposure to ultrasound makes it possible to significantly expand the area of weldability of various metal pairs, which makes it possible to obtain equal-strength joints with minimal development of structural and mechanical inhomogeneities in low-intensity welding modes².

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EVOLUTION OF THE STRUCTURE AND PROPERTIES OF MATERIAL FROM AZhK ALLOY OBTAINED BY THE METHOD OF SELECTIVE LASER ALLOYING IN COMBINATION WITH POST-PROCESSING

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Laser powder bed fusion (LPBF) is one of the promising technologies for manufacturing complex-shaped parts from Superalloy. This is mainly due to the lack of need for subsequent complex mechanical post-processing of products made from metals, which are characterized by poor machinability, and, as a consequence, a reduction in material and labor costs for the manufacture of products. The essence of the LPBF process is the layer-by-layer selective fusion of powder material on a metal plate according to a previously formed 3D model. A feature of the layer-by-layer synthesis process is the crystallization of the material in a metastable state, as well as the possible formation of defects in the form of pores, cracks, and local non-fusion. In addition, during the LPBF process, the precipitation of strengthening phases (carbides, γ' -phase, etc.) in the can be suppressed. To eliminate these disadvantages and improve performance properties, the synthesized material is subjected to various types of post-processing. The most common scheme for processing LPBF material is annealing to relieve thermal stresses, hot isostatic pressing (HIP), which reduces the number of continuity defects in the material, and hardening heat treatment (HT) such as hardening followed by aging.

The object of study of this work was the AZhK alloy, intended for the manufacture of heavily loaded body parts with an operating temperature of up to 800 °C.

It has been established that the maximum level of properties of LPBF samples from the AZhK alloy is achieved through HIP and HT operations, which is associated with the elimination of the characteristic LPBF structure (melt pool boundaries, laser tracks), a decrease in volumetric porosity and the release of the strengthening γ' -phase and carbides in volume and along grain boundaries. As a result of post-processing, the tensile strength of LPBF samples increased by more than 30% while maintaining a high ductility index of over 20%.

DETERMINATION AND STUDY OF THE MAIN DIRECTIONS FOR THE REMOVAL OF CARBON FROM THE MELT IN A CIRCULATING VACUUM UNIT

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Obtaining an ultra-low carbon content in steel (less than 0.002%) is an important practical and theoretical task.

Considering the process of decarbonization of the melt in a circulating vacuum unit, several factors can be identified that affect the rate of carbon removal and the achievement of minimum concentrations of this element in the melt:

1. Technological factors;
2. Work and exploitation of the equipment;
3. The composition of refractory materials;
4. The design of the circulation vacuuming unit.

To achieve the required carbon concentration in steel, it is necessary to study and take into account each of the factors and determine the relationships between them.

The paper considers the influence of the fourth factor on the decarbonization of the melt. It is shown that when the lining of the inlet snorkel wears out, the conditions for carbon removal change, therefore it is necessary to make changes to the technological parameters of steel processing.

The technological and structural parameters of the circulating vacuum unit have been determined, which make it possible to achieve the best performance in terms of the intensity of carbon removal from the melt. The best performance is achieved when the flow rate of the transporting gas is in the range of 66.0-90 m³/hour and the inner diameter of the inlet snorkel is 0.43-0.50 m.

Theoretical calculations show that for a 160 circulating vacuum unit, as the lining breaks out, it is necessary to gradually increase the gas consumption to values of 105-110 m³/hour.

PHYSICAL-CHEMICAL AND TECHNOLOGICAL BASICS OF MELTING SEMI-PRODUCTS IN ARC STEEL MELTING FURNACE UNDER MAGNESIA SLAG

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Currently, in the structure of steelmaking there is a steady trend towards an increase in the share of steel smelting in arc steel-smelting furnaces, which reaches more than 30%. One of the main directions for improving technological and technical-economic indicators is the operation of modern arc steel-smelting furnaces under magnesium slag. The results of theoretical and experimental studies formed the basis for the development of a technology for the formation of magnesium slag of a rational composition based on smelting periods in DSP-135 furnaces. The implementation of the developed technological modes ensured a high level of basic technological and technical and economic indicators of the process. The operating time of the furnace under current decreased by an average of 1.5 minutes, and the specific energy consumption decreased by an average of 15 kWh/t. A record durability of the furnace refractory lining (RHI) has been achieved, exceeding more than 2000 heats per campaign. There was a reduction in the consumption of refractory materials for lining and maintenance of the furnace (gunning masses, filling materials) by an average of 3.76 kg/t. At the same time, the formed slags retain high refining properties, providing an average phosphorus content in the metal at the end of the oxidative period of smelting at the level of 0.005%.

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INDUSTRIAL STUDIES OF DEEP DESULFURIZATION OF STEEL PIPE GRADES IN THE CONDITIONS OF THE FOUNDRY AND ROLLING COMPLEX OF VSW

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In the production of pipe grades of steel, one of the main technological operations is metal desulfurization, the purpose of which is to ensure the production of a sulfur content of less than 0.001%.

According to our estimates, in the course of steel production along the chain: chipboard-Release-automatic transmission, the desulfurization rate is:

Production stages	EAF	Release	LF
Desulfurization rate, ppm/мин	3,5		6

Table 1. The degree of desulfurization by production stages.

It was found that the maximum desulfurization rate of steel corresponds to the maximum concentration of sulfur in the metal and is about 12 ppm/min, then when the sulfur content decreases below 0.005%, the rate drops by more than 10 times. The minimum sulfur removal rate is fixed in the range of its ultra-low concentrations from 0.0013 to 0.0010% and is 0.2 ppm/min.

It is generally believed that the concentration of sulfur in steel during extraction under refining slag in the bucket-furnace unit can be estimated with sufficient accuracy by applying the known ratio 1:

$$\frac{[\%S] - [\%S]_p}{[\%S]_n - [\%S]_p} = \exp(-k \times t)$$

Where: [%S], [%S]_n, [%S]_p - are the current, initial and equilibrium sulfur concentrations in the metal, respectively;

t - the duration of the desulfurization process (min);

k - the mass transfer coefficient (min⁻¹).

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IMPROVEMENT OF STEEL MODIFYING TECHNOLOGY IN OUT-OF-FURNACE PROCESSING

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The most common modifier currently used for steel treatment in the secondary furnace process is calcium, which is most often introduced into the melt as part of SK-30(40) grade wire with a powdered filler.

Inoculation of steel with a two-component component does not always ensure the complete transformation of oxide inclusions from refractory to low-melting calcium aluminates, which worsens the properties of finished metal products [1, 2]. For example, the use of SK-30 in the production of sulfur-containing assortment worsens the spillability of steel at continuous casting machines due to the excessive formation of CaS sulfides, which lead to overgrowth of the walls of the steel-pouring path [3].

In the study, schemes for introducing the modifier at the final stage of secondary treatment (before and after vacuuming) were tested. It has been established that the moment of introduction of the modifying additive has a significant impact on the process of formation of low-melting aluminate inclusions. The use of a three-component inoculant containing additional barium for inoculation of the melt makes it possible to influence the morphology and phase composition of non-metallic inclusions to a greater extent due to its effect on the modifying properties of calcium.

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METAL POWDER COMPOSITIONS OF HEAT-RESISTANT INTERMETALLIC ALLOYS BASED ON TITANIUM ALUMINIDES FOR ADDITIVE MANUFACTURING

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Chemical composition, structure, and technological properties have been investigated for metal powder compositions (MPCs) of a new six-component TiAl-based alloy with Gd microadditions: Ti–31.0Al–2.5V–2.5Nb–2.5Cr–0.4Gd, wt.% (Ti–44.5Al–2V–1Nb–2Cr–0.1Gd, at.%).¹ Three MPCs fractions (10–63, 40–100, 80–120 μm) were produced by electrode induction melting and inert gas atomization technique (table 1) and targeted for the additive manufacturing of parts by selective electron beam melting (SEBM).

Fraction, μm	Elongation, $l_{\text{max}}/l_{\text{min}}$	Form factor, P/\sqrt{S}
10 – 63	$1,09 \pm 0,01$	$3,89 \pm 0,01$
40 – 100	$1,10 \pm 0,01$	$3,96 \pm 0,01$
80 – 120	$1,08 \pm 0,01$	$3,94 \pm 0,01$

Table 1. Particle elongation and shape parameters for different MPC fractions

It is shown that the chemical composition of the MPCs for the main elements corresponds to that of the electrode. In contrast, a 1.5-fold increase of the oxygen content in the MPCs was observed, which is being the result of natural oxidation of powder particles upon air environment due to developed specific surface. It has been determined that the phase composition of the MPCs ($\gamma+\alpha(\alpha_2)+\beta$) differs from the equilibrium phase composition of the electrode ($\gamma+\alpha_2+\beta_0/B2$) and corresponds to a rapidly quenched metastable state, which indicates high solidification rates in the atomization process, exceeding critical cooling rates of the alloy. The technological properties, specifically the powder flowability, were found to be improved for 40–100 and 80–120 μm fractions, making them applicable for additive synthesis by SEBM.²⁻³

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IMPROVING THE QUALITY OF CRYOGENIC STEEL

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The inevitable process of high-temperature heating is the formation of oxide scale on the steel surface, which is fully applicable to cryogenic steel, largely due to the peculiarities of its chemical composition¹. Due to the addition of Ni to cryogenic steels, a complex multi-component scale is formed on the surface during hot working, including an outer oxide layer and an additional inner oxide layer².

The outer layer grows by diffusion of iron outward, the inner layer grows by diffusion of oxygen inward. As oxidation proceeds, an irregular scale/steel interface is formed³.

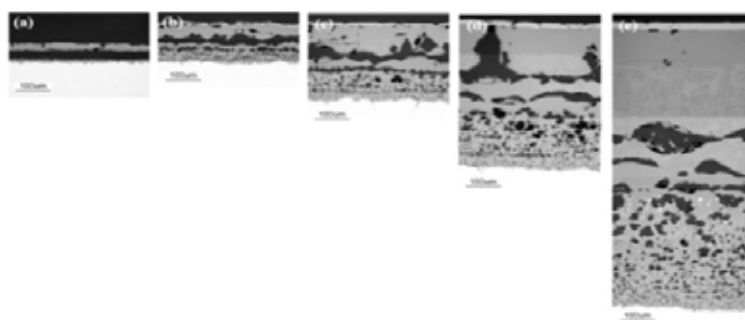


Figure 1. Cross-sectional microstructure of Fe-5% Ni alloy oxidized at 1200 °C for (a) 1, (b) 5, (c) 15, (d) 30, and (e) 60 min.⁴

In our study, the critical analysis of scale layer growth was carried out with the help of mathematical modeling tools and artificial intelligence, resulting in recommendations for adjusting the significant parameters of cryogenic steel heating before rolling.

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INVESTIGATION OF THE LEAD AND ZINC SELECTIVE EX-TRACTION POSSIBILITY FROM EAF DUST IN A REDUCING MEDIUM

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The electric arc furnace dust formed during the production of steel in electric arc furnaces and contains iron (35–55 wt. %), zinc (15–25 wt. %) and lead (up to 3 wt. %), which according to the concept of a closed-loop economy should be returned to production.

The study object is EAF dust from baghouse. The elements content, by wt. %: Fe–41,4; Zn–14,5; Ca–6,2; Na–2,8; Mn–2,5; Cl–1,7; C–1,7; Mg–1,5; Si–1,3; K–1,1; Pb–1,0; S–0,74; Cr–0,2; Cu–0,2; Al–0,2; Ti–0,1; P–0,1; the rest is presumably oxygen. The main phases were determined by the X-ray diffraction method. Basic phases are (Zn,Mn,Fe) O₄, Fe₃O₄, ZnO.

The zinc and lead behavior were studied when heating 5 g of EAF dust in a horizontal tubular furnace in an argon medium with an excess of reducing agent (soot) of 20 wt. % with stepwise heating. The first stage: heating up to 1100 °C with an exposure time of 1 hour. Second stage: heating up to 1200 °C with an exposure time of 1 hour. В ходе эксперимента осуществляли отбор отходящих газов. During the experiment, the waste gases were selected. After cooling, a thin metal foil was found on the gas outlet pipe inside the furnace. The resulting products and residue were studied on a JEOL JSX ISO 100 raster electron probe microanalysis.

Heating and holding the sample at 1100 °C led to the joint removal of lead and zinc from the EAF dust. Upon subsequent heating to 1200 °C and exposure for 1 hour, zinc without the presence of lead was detected in the sublimates.

According to the results of determining the chemical composition and elemental mapping of areas on the JEOL JSX ISP 100 microanalyzer, the sublimates cooling outside the furnace led to the formation of lead and zinc oxides. At the same time, cooling the sublimates directly in the furnace in an inert medium allows you to obtain the product in a metallic form.

Phase analysis of the residue of EAF dust showed the iron-containing phase reduction to α -Fe, the presence of the Fe₃C phase was noted, which is associated with an excess of the reducing agent; the lead extraction degree was 95 rel. %; the zinc extraction degree was 99.8 rel. %.

As a result, the technique for the lead and zinc selective separation when EAF dust heating has been developed and the mathematical model has been constructed that allows to pre-evaluate the selective separation of these colored metals under different conditions.

DEVELOPMENT OF A MATHEMATICAL MODEL OF THE FORMATION OF NON-METALLIC INCLUSIONS DURING THE LADLE PROCESSING OF METAL

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One of the main parameters of the metallurgical quality of steel is the amount of non-metallic inclusions (NMI) it contains. It is known that NMI have an extremely negative effect on the properties of steel, being sources of such defects such as the formation of cracks, captivities and discontinuities, and are the cause of overgrowth of steel-pouring equipment. Therefore, it is extremely important to control the level of inclusions during ladle treatment. To this end, to predict the formation of NMI and determine the possibility of their removal, a mathematical model was developed that allows to calculate the number of primary oxide inclusions formed during ladle processing of steel and determine their residual amount.

Information about the mass, temperature and chemical composition of the metal, as well as its oxidation, is specified as the initial data for the algorithm of the formation of NMI. At the output of the algorithm, information can be obtained about the types and volume fraction of inclusions that can be formed under given process parameters on the ladle-furnace processing.

To determine the number of removed non-metallic inclusions, information on the types and sizes of inclusions, the viscosity of the liquid metal and the intensity of purging with an inert gas is indicated as initial information. At the output of the algorithm, information can be obtained about the possibility of removing of NMI, the time during which they can be removed and their residual quantity in the ladle-furnace.

The developed mathematical model of NMI formation and removal is implemented as a software application. In this work, the method of fractional gas analysis (FGA) was used to study metal samples taken at the beginning of processing on a ladle-furnace unit (LAF) for pipe steels grades K-56, 09G2S, KEI-55, corrosion-resistant austenitic steel 08X18H10T and steel grade IF-BH. The total oxygen content, characteristic types of NMI and their quantities were determined. The results of calculations of the NMI content in selected metal samples according to the developed program were compared with the results of FGA. Satisfactory agreement between the calculated values and the experimental data was obtained.

DEVELOPMENT OF A NEW METHODOLOGY FOR EXPERIMENTAL SMELTING

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The development of new steel compositions with specified properties is costly due to the expense of smelting a series of trial compositions. The economical methodology of such works is offered. For smelting of compositions on the basis of steel Cr13 with additives N, Mn, Mo, V, Nb on the basis of one large melting of the basic composition were obtained several ingots with different alloying. From the metal of the basic melting Fe-C-Cr-Mn made sampling (MS) for analysis and casting ingot (CI) No. 1. After continued melting of the metal in the furnace and introduced an additional alloying element (Mo); after the MS was made CI No. 2, the system Fe-C-Cr-Cr-Mn-Mo. Then N, Nb, V were successively introduced, obtaining castings of the required compositions (Figure 1).

Nitrogen alloying of steel increases its strength and corrosion resistance. To assess the solubility of nitrogen in solid metal, among other things, the coefficient of compositional stability K_s is important. It reflects the distribution of nitrogen between liquid and solid phases¹. For the Fe-13Cr-(N, Mn, Mo, V, Nb) composition, K_s was determined using a new methodology. Scheme of work: smelting of base metal, MS, CI No. 1 with %N₁. Step 2 – introduction of a nitrogen-containing ligature according to calculation, MS after nitrogen assimilation, CI No. 2 with %N₂ = N₁ + 0,01%, etc. Crystallization was observed until the appearance of “growth” after CI. It indicated that %N_i in the previous defect-free ingot was compositionally stable. So, without using a series of melts, the effective K_s was determined.



Figure 1. Templates of Fe-13Cr-(N, Mn, Mo, V, Nb) steel ingots when determining K_s

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THE RAPID HEATING EFFECT ON THE TEXTURE FORMATION IN THE HIGH-PERMEABILITY GRAIN-ORIENTED STEEL

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Rapid heating of the high-permeability grain-oriented steel prior to decarburizing annealing allows to obtain better magnetic properties¹. Improvement of magnetic properties is based on increasing the intensity of the Goss texture $\{110\}<001>$ in the primary recrystallization matrix².

Investigation was implemented on the high-permeability grain-oriented steel samples of two compositions: steel A containing larger amount of tin (0,08-0,1% (wt.)) and steel B containing lesser amount of tin (0,03-0,05% (wt.)). The physical simulation of the heating stage was carried out according to the next three regimes 1) heating at standard rate of 25-30°/s or 2) heating at 150°C/c.

Texture analysis was carried out by means of EBSD and diffractometry. Both methods demonstrate close values of Goss texture with misorientation of 7° and similar results that rapid heating increases the Goss grains area fraction. According to the EBSD results, rapid heating increased the Goss grains area fraction by 7,5 times in steel A and by 12 times in steel B. According to diffractometry results, there is a similar trend of increasing the Goss intensity due to the rapid heating. It is also demonstrated by the diffractometry results as well as by the EBSD results that steel B contains more Goss texture.

The research has also been conducted on the effect of the rapid heating on such texture components as $\{411\}<148>$, $\{311\}<136>$, $\{111\}<112>$ and texture fibers α , α' and γ after primary recrystallization.

The effect of alloying by tin was evaluated. It was shown that tin affects the structure and texture formation in the rapid heating process.

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PHYSICOCHEMICAL PROPERTIES OF DESULFURIZATION PERIOD SLAGS OF THE AOD PROCESS

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Using the simplex lattice method of experiment planning, the physicochemical characteristics of slags of the $\text{CaO-SiO}_2\text{-B}_2\text{O}_3\text{-2\%Cr}_2\text{O}_3\text{-3\%Al}_2\text{O}_3\text{-8\%MgO}$ system with a basicity of 1.0-2.5 and a content of 0-6% B_2O_3 were studied. The results of experimental studies of slag viscosity are presented graphically in the form of a composition - property diagram (Figure 1). It was established that the introduction of up to 6% of boron oxide into the slag, despite the increase in the complexity of its structure, provides a sufficiently high liquid mobility of the melt in the considered basicity range. The viscosity of slag varies from 0.12 Pa·s in the basicity range of 1.0-1.3 to 0.2 Pa·s with a basicity of 2.5. At the same time, the formed slags with a viscosity of 0.2-0.3 Pa·s in the basicity range of 2.0-2.5, containing 4.0-6.0% boron oxide, retain the possibility of sufficiently deep desulfurization of the metal, providing sulfur content in it at the level of 0.003-0.004%. The formed slags in the considered region of chemical composition at temperatures during the desulfurization period are in a homogeneous region, having a crystallization onset temperature not exceeding 1500 °C.

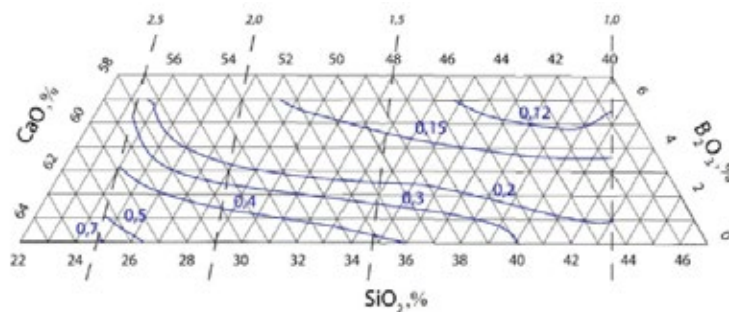


Figure 1. Viscosity diagram for the $\text{CaO-SiO}_2\text{-B}_2\text{O}_3\text{-2\%Cr}_2\text{O}_3\text{-3\%Al}_2\text{O}_3\text{-8\%MgO}$ slag system at a temperature of 1600 °C: solid lines — viscosity, Pa s; dotted lines — basicity

The work was carried out according to the state assignment for IMET UB RAS.

MODERN TECHNOLOGIES OF METAL SMELTING WITH TVO IN HEAVY-DUTY ARC STEELMAKING FURNACES

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Metal smelting with thermally advanced steel processing (TVO) was developed by the schools of Samarina A.M., Baum B.A., Elansky G.N. and Kudrin V.A.. Currently, this has been widely developed in the Evraz-ZSMK electric steelmaking workshop ¹. There, the optimal metal temperature in the furnace was developed before melting, the time of metal processing in the furnace was determined, the temperature range of the beginning and end of metal processing at the automatic transmission was found, the time of metal processing, the modes of vacuuming and casting of metal were estimated¹.

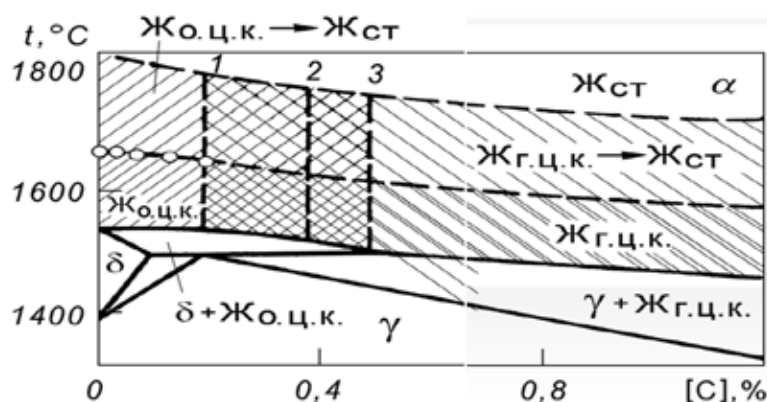


Figure 1 - The structure of the short-range order of Fe-C melts. ²

Similar work is currently being carried out at the VMZ. The results of the research allow us to state that overheating of the melt of a tubular steel grade to the temperature of an anomaly can cause a restructuring of the entire structure of the melt.

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ANALYSIS OF FRACTURES IN LOW-ALLOY STEELS USING COMPUTER VISION

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Testing samples of low-alloy steels in impact bending is used to assess the quality and reliability of the metal. During testing, information is obtained about the fracture process both integrally and in stages in the form of a dynamic curve in force-deflection coordinates, as well as information about the fracture structure. Manual fracture analysis is often subjective, inaccurate, and time-consuming¹. Essentially, identifying fractures in images and different areas on the fracture is a task of semantic segmentation, which can be solved using modern computer vision algorithms based on neural networks. The aim of this work was to develop efficient methods for automatic analysis of fractographic information from digital images of fractures in samples of low-alloy steels.

Images of fractures in base metal and welded joints of steels 09G2S, 17G1S after hot rolling and after heat treatment were used for the study. Fractures in samples under impact bending were obtained in the ductile-to-brittle transition range and contained two types of fracture surfaces - ductile dimpled and brittle faceted.

A comparison of two approaches to fracture analysis was conducted. The first approach involved extracting textural features from images and using them as input signal vectors for a fully connected feedforward neural network (Multilayer Perceptron, MLP). A sliding window method was used to obtain spatial information about the fracture structure. The second approach involved using a convolutional neural network with an encoder-decoder architecture (U-Net). Fragments of the analyzed image were used as input signals with subsequent merging. It was shown that the use of the U-Net architecture allows for a more accurate model compared to MLP (98.88% on the test set versus 92.43%). Additionally, the MLP architecture is suitable for systems with low performance without GPUs required for fine-tuning and using U-Net.

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INVESTIGATION OF THE EFFICIENCY OF REFINING RAIL STEEL FROM NON-METALLIC INCLUSIONS WHEN VARYING THE PARAMETERS OF INERT GAS PURGING IN A STEEL LADLE

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The operational stability of railway rails largely depends on the type, concentration and location of non-metallic inclusions¹. At the same time, based on the physical nature of the processes occurring in the production of rail steel in modern steelmaking workshops^{2,3}, the concentration of non-metallic inclusions in it and, accordingly, in finished rails is significantly determined by the modes of purging the melt with inert gas in a steel ladle.

The paper presents the results of comprehensive studies of the refining process of rail steel from non-metallic inclusions when it is purged with argon on the ladle furnace unit in relation to the conditions of the electric steelmaking workshop of EVRAZ ZSMK JSC. These studies included mathematical modeling of the efficiency of mixing and diffusion processes in the melt of rail steel when purging with inert gas in a steel ladle through bottom tuyeres, field experiments to determine the contamination of rail steel with non-metallic inclusions of various types, depending on the modes of its purging with argon.

According to the results of the conducted research, the regularities of the influence of the parameters of purging of rail steel with inert gas during processing on the bucket furnace unit on the content of non-metallic inclusions in it have been established and purging modes have been developed to ensure an increase in the purity of the rail electric steel for non-metallic inclusions.

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REM OXIDES AND THEIR INFLUENCE ON THE PHYSICAL AND CHEMICAL PROPERTIES OF LADLE METALLURGY SLAG

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The development of effective technological methods for controlling non-metallic inclusions is a promising direction for improving the complex of properties and quality characteristics of steels. One of the factors regulating the quantity, morphology and distribution of sulfide inclusions over the metal volume is the sulfur content. To organize the production of steel with low (up to 0.003-0.005%) sulfur content, the desulfurization process is carried out in ladle-furnace installations with the formation of the main slags of the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ system and deep deoxidation of steel with aluminum. At the same time, one of the main oxide inclusions in steel deoxidized with aluminum is corundum (Al_2O_3), which deteriorates the properties of steel and leads to “overgrowth” of the inner surface of the immersion nozzle during continuous casting. The negative effect of corundum in steel can be neutralized by removing it into the main liquid slag formed in the ladle-furnace by reducing the activity of Al_2O_3 . However, in practice, an excessive increase in the basicity of refining slag to reduce the activity of Al_2O_3 is usually accompanied by heterogenization of the slag, an increase in its melting temperature and a decrease in refining properties. One of the promising directions for reducing the activity coefficient of Al_2O_3 in basic refining slags may be the use of rare earth metal oxides. The use of REM oxides ensures a decrease in their melting point, an increase in fluid mobility, an increase in the coefficient of interphase distribution of sulfur and a decrease in the coefficient of interphase distribution of REM. The paper presents the results of a study of the influence of cerium oxide in the slags of the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-Ce}_2\text{O}_3$ system on the physicochemical properties. The positive influence of the temperature factor, slag basicity and cerium oxide content in the studied range of chemical composition on the process of cerium reduction is shown.

The research was supported by a grant from the Russian Science Foundation № 22-29-00975, <https://rscf.ru/project/22-29-00975/>.

COMPOSITIONAL APPROACH TO PREVENT NITROGEN POROSITY OF AUSTENITIC-FERRITIC STEEL INGOTS

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The crystallization mechanism of austenitic-ferritic steels (AFS) is determined by the balance of concentrations in the melt of groups of elements - ferritizers (Cr, Mo, Nb, Si, Al) and austenizers (Ni, N, Mn, C), expressed through the ratio of chromium and nickel equivalents. Crystallization proceeds through 100% δ -ferrite¹ at $C_{req} / Ni_{eq} > 2$ (Figure 1).

The nitrogen content in the AFS is above 0.2% wt. during crystallization, it can cause the release of excess nitrogen into the gas phase with the formation of pores, which significantly complicates the production of forgings and blanks and reduces the yield. The report reveals the essence of the compositional approach - optimization of the content of alloying elements within the grade composition of steel UNS S32750, which allows to manage ingot quality. The proposed methodology is based on the solution of a system of regression equations describing the conditions for achieving direct and indirect quality indicators depending on the chemical composition of steel. Along with the use of certain technological techniques², this approach makes it possible to prevent the formation of nitrogen pores during ingot crystallization.

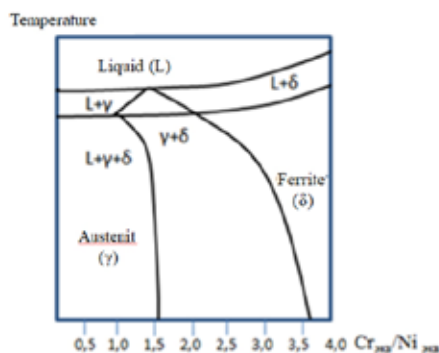


Figure 1. High-temperature region of the pseudobinary diagram for chemical compositions of stainless AFS steel¹

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MODELING AND SIMULATION OF METALLURGICAL PROCESSES IN THE DEVELOPMENT OF STEEL FOR CASING PIPES

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During the development of technology for the production of rolled 30G steel for casing pipes, process modeling is used, laboratory and industrial experiments are carried out. The banded ferrite-pearlite structure that occurs during rolling of low- and medium-carbon steels is an unfavorable structural factor that increases the anisotropy of mechanical properties in the rolling. Banding is a consequence of dendritic segregation in steel¹. The view of the deformed dendritic structure after simulated rolling on a dilatometer is shown in Figure 1.



Figure 1. Type of deformed dendritic structure

The mathematical modeling of solidification processes in Thermo-Calc is a tool for predicting dendritic segregation². The dendritic structure in 30G grade steel is a picture of the distribution of Si and Mn elements.

In order to reduce dendritic segregation and improve the microstructure obtaining the required mechanical characteristics, the content of chemical elements in rolled steel 30G was adjusted.

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STUDY OF THE INFLUENCE OF SINTER BASICITY ON STRUCTURE AND METALLURGICAL PROPERTIES

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The mechanical strength, reducing strength, reducibility, and softening interval of sinter depend primarily on their chemical, phase, and mineralogical compositions. The basicity of sinter can vary widely, and altering it can produce sinter with desired characteristics. This study examined samples of iron ore sinter with basicity ranging from 1.2 to 3.0. The agglomerates undergo several phases, including magnetite, hematite, calcium, and aluminium silicoferrite with variable composition (SFCA), as well as two-calcium silicates.¹ Increasing the basicity of the sinter results in a higher amount of SFCA phase, with a corresponding increase in Ca content. The study also investigated the changes in phase composition of sinter during heating with a solid reducing agent from 500 to 1200 °C. The SFCA phase-binder can be easily reduced to magnetite and gellenite in low-base sinter, and to magnetite, tricalcium silicate, and a small amount of srebrodolskite in high-base one. The reduction strength and softening interval of sinter with different basicity were investigated, and the best values of thermomechanical strength were found in sinter with basicity 1.8-2.0.² The temperatures at the beginning and ending of sinter softening increase as basicity increases from 1.2 to 3.0, and the softening interval narrows. This will have a favourable effect on the blast furnace smelting process.³

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MODELING OF FORMATION OF NON-METALLIC INCLUSIONS DURING THE MELTING OF DUPLEX STAINLESS STEELS IN AN OPEN INDUCTION FURNACE

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When melting duplex stainless steels (DSS) in an open induction furnace (OIF) with limited refining possibilities, it is important to predict the type and volume fraction of non-metallic inclusions (NMIs) formed depending on the furnace volume, conditions for deoxidizers adding and melt mixing. This will improve the metallurgical quality of the DSS and improve their properties. Therefore, the goal of this work was to create a kinetic model of the formation of inclusions in titanium-killed DSS.

When describing the formation of NMIs in the furnace, the entire volume of the crucible was divided into 10 zones. The transfer of elements was sequentially carried out from the first zone (ferroalloy input area) to the second adjacent one, then from the second to the third, and so on. The rate of transfer of all components, primarily titanium, between adjacent zones depended on the specific interaction area, mass transfer coefficient, and differences in element concentrations. Within each zone, the equalization of reagent concentrations occurred instantly.

At the first moment of time in the first zone, the titanium concentration was set, corresponding to the complete dissolution of the introduced ferroalloy. Then the type and mass of the forming primary NMIs were calculated, after which the time increment was set and the concentration of reagents and the mass of NMIs were determined for each zone in the next step as a result of mass transfer. To predict the type and amount of NMIs, the equilibrium constants of the corresponding reactions of the formation of titanium oxides or nitrides were calculated. In order to take into account the transformation of inclusions to an equilibrium state, the transformation coefficient α was introduced at each subsequent time step. Three cases were considered: complete transformation of NMIs ($\alpha=1$); absence of transformation ($\alpha=0$); partial transformation ($0<\alpha<1$).

As a result, the dependences of the concentrations of reagents in the liquid steel, the amount and type of NMIs for each calculation zone, as well as for the entire volume of the furnace, were obtained. It was studied how NMIs evolve depending on the melting parameters. The results of calculations are shown to be in good agreement with experimental data from studying the kinetics of NMIs formation in an induction furnace.

FORMATION OF A LOW-ALLOYED SUBLAYER WITH A BAINITE STRUCTURE FOR SURFACING OF METALLURGICAL EQUIPMENT PARTS

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Identification of the concept of the complex influence of the composition of surfacing materials and the technology of hardening surfacing on the mechanical and operational properties of pipe rolling and pipe piercing tools made from steels with a high carbon content is an urgent task of scientific research.

In order to increase the resistance of the working surfaces of the deposited product to the formation of thermal fatigue cracks, an intermediate layer (sublayer) of a low-carbon, low-alloy metal of the bainitic class of the C-Si-Mn-Cr-Ni-Mo-Cu-Al-Ti-La system was first formed on the surface of the workpiece to be strengthened using the electric arc surfacing method.

This approach to the development of metal for the sublayer makes it possible to increase the energy of crack initiation and development in it and to obtain a good combination of plastic (relative elongation up to 30%) and strength (conditional yield strength up to 620 MPa, tensile strength up to 780 MPa) properties that determine the ability of the metal in the “base metal - sublayer - wear-resistant metal” system to resist cyclic thermal and force effects.

It has been established that the use of the developed sublayer metal increases the resistance of the deposited wear-resistant alloy (25Kh5FMS and 3Kh2V8) to the occurrence of thermal fatigue cracks from the surface into the metal of the product, which is confirmed by the largest number of cycles before the appearance of the first crack during periodic thermal exposure in the temperature range (800-50 °C). The achievement of this result is due to the targeted formation of the metal structure of the sublayer during the decomposition of austenite in the bainitic region and its low sensitivity to cycles of repeated heating and cooling under conditions of multilayer surfacing. The basis of such a structure is bainitic ferrite of the granular type, in which the proportion of the martensitic-austenitic component does not exceed 2-3 vol.%, and the content of degenerate pearlite, depending on the height of the sublayer, varies in the range of 5-10 vol.%.

The study was supported by a grant from the Russian Science Foundation № 24-23-20068, <https://rscf.ru/project/24-23-20068/> and a grant from the Volgograd Region Administration under agreement No. 7 dated May 31, 2024.

PLASMOCHEMICAL PROCESSES IN A JET HIGH-FREQUENCY CAPACITIVE DISCHARGE OF LOW PRESSURE FOR THE CREATION OF NANODIFFUSION LAYERS IN METALS AND THEIR ALLOYS

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The results of a study of the process of formation of nanodiffuse coatings on the surface of structural materials using low-pressure high-frequency capacitive discharge plasma in an atmosphere of chemically active gases are presented. The processing was carried out on a high-frequency plasma installation with flat electrodes located inside a vacuum chamber.¹

The experimental studies carried out made it possible to establish that in the range of ion bombardment energy of 10-100 eV and ion current density on the surface of 0.1-5 A/m², modification of the surface layer of the studied materials with a thickness of 70-420 nm occurs, including a change in the composition of the near-surface nanolayer.

The results of experimental studies showed that when reacting gases (N₂, O₂, CO₂, CH₄, C₃H₈+C₄H₁₀) are added to the plasma-forming gas, the composition of samples from metals and alloys changes to a greater extent than during processing in a pure argon environment.

The set of results on modification of the surface of the VK6-OM alloy, steels, and titanium alloys in a low-pressure high-frequency capacitive discharge plasma flow indicates that nanodiffuse coatings are formed on the surface.

For example, when processing a tungsten-cobalt alloy in a carbon dioxide environment at a depth of less than 10 nm, the structure corresponds to graphite. Further, the C-C bond corresponds to disordered carbon, and from 30 nm onwards the intensity of the tungsten carbide peak increases.

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INVESTIGATION OF THE ELEMENTAL COMPOSITION OF FENCING BLADES MADE OF MARAGEN STEEL

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Blades used in sports fencing are divided into two large segments - for beginners and for professionals. In our work, we considered rapier blades made of maragen steel from various manufacturers.

The study of fencing blades was carried out using scanning autoemission electron microscopy on a Merlin ZEISS device.

№	Fe	Ni	Co	Mo	Ti	Al	Cr
1	63.10	17.05	8.88	4.95	0.69	0.11	-
2	64.64	17.47	9.10	4.23	0.75	0.12	-
3	59.30	16.06	8.95	4.51	0.73	0.16	0.05
4	61.33	17.30	8.95	4.60	0.72	0.20	0.12
5	63.44	17.05	8.40	4.60	0.71	0.27	0.12
6	65.20	17.90	9.20	5.03	0.69	0.13	0.15

Table 1. The content of elements in maragen steel samples, % by weight

Based on the research results, it can be argued that steel samples from different manufacturers do not differ much from each other and correspond to grade 300 according to the American standard.

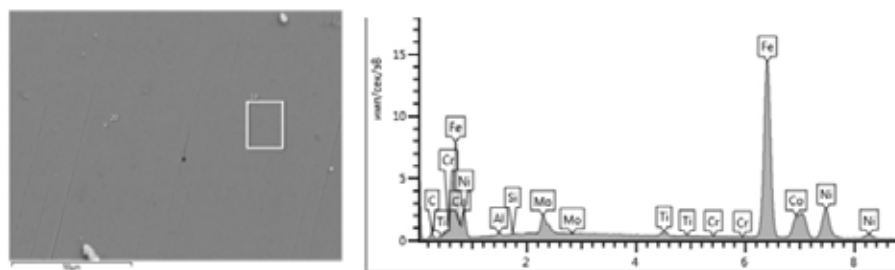


Figure 1. Cut of blade 6 and its elemental composition

Among the samples, one blade was identified, which consists of two different types of steel. The handle of this sample did not correspond to the composition of maraging steel. Some samples contained white inclusions corresponding to NaCl crystals.

The work was carried out with the support of the Sirius program. Summer: Start your project.

INFLUENCE OF THE CALCINATION OF STABILIZED ZIRCONIUM DIOXIDE POWDER ON THE DISTRIBUTION AND PORES TYPE

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Zirconium dioxide are widely used as raw materials for the formation of functional and biocompatible ceramics due to the combination of resistance, thermal stability, high biocompatibility and availability of precursors for synthesis, powders based on stabilized.

The research was devoted to the influence of the calcination on the distribution and pore type of stabilized zirconium dioxide powder for the formation of biocompatible ceramics. The powder was obtained using the controlled double-jet precipitation method at pH = 8. After precipitation, the sediment was subjected to heat treatment at 120, 1050, 1100, 1150, 1200 °C and studied the distribution of pores. Nitrogen adsorption/desorption isotherms are shown in Figures 1 and 2.

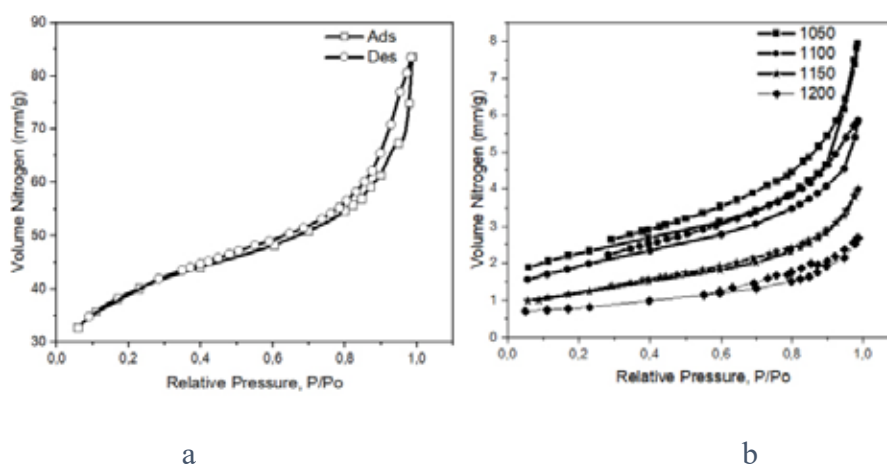


Figure 1. Pore distribution of drying (a) and after calcination at 1050, 1100, 1150, 1200 °C (b)

The powder subjected to heat treatment at 120 °C has the highest pore volume among other samples – 0.1300 mm³/g and a high specific surface area – 135 m²/g, whereas the powder calcined at 1200 °C is characterized by a low pore volume – 0.0042 mm³/g, small surface area – 2.4 m²/g. The samples are sent for milling in a planetary mill to increase pore volume and specific surface area.

INFLUENCE OF LASER PRINTING CONDITIONS ON THE STRUCTURE AND MECHANICAL PROPERTIES OF NICKEL-BASED SUPERALLOYS MADE BY LASER 3D PRINTING

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Additive technologies are a promising direction for producing metal products with complex geometric shapes and are especially relevant for the aviation and nuclear industries, in which nickel-based superalloys are widely used.

A study was carried out of the structure, phase composition, texture and anisotropy of the mechanical properties of samples of nickel heat-resistant alloys KhN58MBYu (VZh159) of the Ni–Cr–Mo–Nb–Al system, obtained using selective laser alloying.

The formation of the structure of the product in the process of selective laser melting is influenced by a number of factors, primarily the thermophysical properties of the powder material, laser radiation power, scanning speed of the laser beam, laser spot diameter, amount of distance between adjacent tracks, thickness of the powder layer and fractional composition of the powder¹. In addition, it is important to take into account the cooling rate and direction of the temperature gradient when cooling the finished product, as well as the peculiarities of structure formation during subsequent heat treatment of samples or finished parts. This issue is especially relevant for complex alloyed multicomponent nickel heat-resistant alloys, in which the formation of various intermetallic phases is possible under conditions of nonequilibrium crystallization^{2,3}.

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RESEARCH OF THE STRUCTURE AND PHYSICAL-MECHANICAL PROPERTIES OF A BILLET FROM HEAT-RESISTANT NICKEL ALLOY EP741NP FROM METAL POWDER OBTAINED BY THE METHOD OF GAS ATOMIZATION

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Based on the obtained statistical data, a histogram of the particle size distribution of the EP741NP alloy powder in microns was constructed (Fig. 1), and a graphical representation of the frequency distribution divided into intervals (histogram) was obtained, reflecting the degree of imbalance of the studied particles of the EP741NP1 alloy powder.

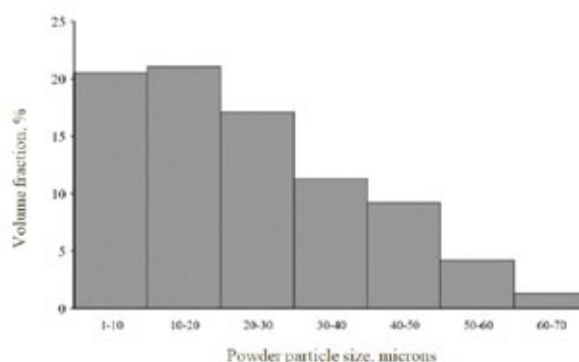


Figure 1. Histogram of particle size distribution of EP741NP powder

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The advantages of β -RuAl/ β -NiAl monoaluminides as the basis for new light, oxidation resistance, heat-resistant and thermostable alloys for critical parts of new technology samples capable of operating in hypersonic flow conditions in an atmosphere containing up to 23% oxygen at gas flow stagnation temperatures of 1500-3000 K, at flight speeds above Mach 5, have a higher melting point (2100°C/1638°C) than modern heat-resistant nickel alloys and alloys based on γ '-Ni₃Al, relatively low density (7.97/5.86 g/cm³, comparable to the density of low-alloy and lower-melting nickel alloys, or lower), Young's modulus (267/284.2 GPa), stable thermal conductivity (70–80 W/m K), 3-8 times higher than the thermal conductivity of heat-resistant nickel alloys, high resistance to oxidation in the temperature range 1100–1200°C. The coefficient of thermal expansion of RuAl is almost equal to that of α -Al₂O₃ in the temperature range from ~150 to 1500°C. Heat-loaded components of gas turbine aircraft engines, transonic and hypersonic aircraft and their engines require complex shaped parts, including miniature ones. To manufacture such parts, it is necessary to use additive technologies, including the 3D printing method, with minimal final machining (selective laser melting or selective laser sintering). Additive manufacturing is based on digital design/modeling and the use of spherical precursor micropowders, suitable for producing dense compact blanks/products of complex shape using 3D printing. Powders must meet certain requirements: strictly regulated granulometric composition, good fluidity and oxide-free surface. This work evaluates the advantages and disadvantages of known methods for producing precursors in relation to monoaluminides, and presents experimental data on precursors from alloys based on NiAl, RuAl and FeCrAl, obtained by plasma spheroidization from various initial powders using the technology developed by the authors.

SEPARATION OF Li, Co, Mn, Ni, Ti IONS FROM THEIR MIXTURE BY A HYDROPHOBIC DEEP EUTECTIC SOLVENT BASED ON TOPO AND D2EHPA

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Li-ion batteries are one of the most popular and affordable ways of storing energy today, due to their high energy density, excellent cycling and long service life¹. Batteries with LTO type ($\text{Li}_4\text{Ti}_5\text{O}_{12}$) anode can work efficiently in a wide temperature range, which makes them very demanded. The number of spent batteries is growing, and there is a need to find and research the best ways of their recycling.

Leaching followed by liquid-liquid extraction is well suited to the task of separating metals from their mixtures. By varying the extraction conditions, it is possible to influence the extraction of different metals and to achieve complete separation. However, there is a problem, that popular organic solvents for extractants (kerosene, toluene) are easily volatile and flammable liquids. Deep eutectic solvents are an excellent alternative, as they do not have the mentioned disadvantage².

This work presents results of the study of the extraction of Li, Co, Mn, Ni, Ti ions by a hydrophobic deep eutectic solvent based on two industrial extractants: trioctylphosphine oxide (TOPO) and di(2-ethylhexyl)phosphoric acid (D2EHPA). The composition of the mixture of TOPO, D2EHPA and menthol, which gives a synergistic effect in the extraction of the studied metals, was selected; the dependence of the degree of extraction of metal ions on the acidity of the aqueous phase, the volume ratio of the aqueous and organic phases and the time of extraction was established.

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INFLUENCE OF ULTRASOUND ON THE STRUCTURE OF A CRYSTALLIZING MATERIAL IN ADDITIVE TECHNOLOGIES

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Due to the large temperature gradients, the metal material grown by powder printing methods has an elongated, unequal shape. As a result, the properties of the metal deteriorate, and anisotropy of properties appears. When exposed to ultrasound during the printing process, the grain structure of the metal turns out to be more equiaxially and finely^{1,2}. One of the mechanisms explaining the effect of ultrasound on the formation of the structure is ultrasonic cavitation. Under the influence of ultrasonic waves, the bubbles in the melt pool collapse and form a shock wave, which, in turn, breaks the growing metal crystal into small parts.

There are two modes of bubble collapse: resonant and non-resonant. The most effective mode is where resonance is present, since less external pressure is required to collapse the bubble.

Calculations were performed for vapor-gas bubbles with radii in the range (10^{-4} - 10^{-6}) m in melts of 316L stainless steel and Ti-6Al-4V titanium alloy.

When moving away from the resonant frequency, the acoustic pressure required for the collapse of cavitation bubbles increases.

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THE SEARCH FOR RATIONAL WAYS TO INTRODUCE CALCIUM INTO STEEL

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Calcium has an important role in steelmaking, as it is a deep deoxidizer, and also acts as a modifier, thanks to calcium, the process of converting solid alumina particles into liquid inclusions of calcium aluminate¹ takes place¹.

A rational way to introduce calcium into steel is powder wire. The yield of calcium during calcium treatment largely depends on the wire feed rate, the ratio of the diameter and height of the bucket, as well as the technological process of the wire feeding line. Among them, the feed rate is a key factor determining whether the desired effect can be achieved. The optimal feed rate is practically independent of the temperature of the processed steel melt. There is only one optimal speed for the powder wire, and it can be roughly expressed as follows:

$$V=K \cdot H$$

V – is the optimum feed line speed, in m/min.

H – is the true depth of molten steel, in metres.

K – is a speed correction factor.

The optimal wire feed rate depends only on the depth of the liquid metal. For the best absorption of calcium by steel, the static pressure of molten steel should be greater than -1.8 atmospheres (calcium vapor pressure)². The more stable the wire feed rate, the better the calcium absorption.

The temperature of the metal directly affects the yield of calcium. The higher the temperature of processed molten steel is, the higher the vapor pressure of calcium becomes. This means that calcium leaves the molten metal faster, reducing its residence time in the steel. As a result, the modification efficiency decreases.

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CONTROL OF DUPLEX CORROSION-RESISTANT STEEL OPERATIONAL PROPERTIES BY MEANS OF REM MODIFICATION

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The expansion of the scope of use of castings made of duplex corrosion-resistant steels (DCS) and stricter requirements for their operational properties requires the search for new ways to improve their metallurgical quality and control structural heterogeneity. One of the ways to achieve this is modification with rare earth metals (REM). Therefore, in this paper, some issues of the influence of REM on the structure and properties of DCS are considered and technological recommendations for their use are developed.

The thermodynamic modeling method is used to calculate the types and number of non-metallic inclusions (NMI) formed in liquid and solidifying steel when modified with different amounts of REM. For the most complete removal of NMI due to the formation of many primary NMI, which are easily removed from liquid steel, an additive of 0.05% REM is required. At high residual concentrations, due to the greater number of oxides formed, their coagulation occurs and contamination increases. It has been shown that in the presence of REM, the solubility of nitrogen increases, which makes it possible to significantly increase its absorption during doping.

The effect of REM on the cast structure of castings and heredity was studied during further exposure at a temperature of 1100 °C with quenching in water. In the cast state, REM additives reduce the length of the columnar dendrite zone due to an increase in the number of nucleation centers during solidification. In addition, the proportion of austenite of Widmanstett morphology decreases in the cast state, which is inherited during heat treatment.

The beneficial effect of REM on the properties of DCS is shown. The 0.05% REM additive, due to an increase in the uniformity of the casting and an increase in the nitrogen content, makes it possible to increase the yield strength and strength limits by ~ 7% and ~ 4%, respectively, and the elongation by ~ 4%. Due to the formation of REM oxides with greater electrochemical potentials than chromium and silicon oxides formed in DCS without modification, resistance to local corrosion increases: the potential of pitting formation increases from 1,058 V to 1,087 V, and the basis of pitting resistance from 1,150 V to 1,230 V.

ORGANIZATION OF DIGITAL PROCESSING OF DWTT RESULTS IN METALLURGICAL LABORATORIES

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This paper presents a study dedicated to the development of a prototype mobile application for analyzing images of sample fractures using neural networks. The main goal of the project is to create a reliable and efficient tool for determining the proportion of ductile fracture, which is an important parameter in materials science and failure analysis.

The development of the mobile application prototype for Android included backend creation, setup of interaction between the server and client, integration of the neural network for image analysis, and implementation of a user-friendly interface. All components were designed and optimized to ensure high performance and reliability of the application. Stable communication between the mobile application and the server allows efficient transmission of images for analysis and obtaining results in real time. The prototype was developed using Microsoft Visual Studio. As a result, the application provides an accessible and easy-to-use tool for determining the proportion of ductile fracture in fractures, which makes it a valuable tool for materials scientists and engineers.

NEW ACHIEVEMENTS IN THE PRODUCTION TECHNOLOGY OF STRUCTURAL AND FUNCTIONAL MATERIALS BASED ON MOLYBDENUM ALLOYS OBTAINED BY VACUUM MELTING

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During the existence of the USSR, a large number of molybdenum-based alloys were developed in the country, manufactured using vacuum melting technology. The bulk of the products were smelted using the vacuum arc melting method, which made it possible to obtain ingots with an oxygen content of 30 ppm and a grain size of 3-5 mm. In the late 70s, the technology for producing ingots using electron beam vacuum melting began to develop, which made it possible to reduce the oxygen content in ingots to a level of 10 ppm. But such ingots, due to their larger grains, were not easy to process during subsequent pressure processing. At the beginning of the 21st century in Russia, it became obvious that the quality of products from molybdenum and its alloys, made from vacuum arc melting ingots, no longer meets the modern requirements of the main branches of domestic industry.

The new center for the development of vacuum fused molybdenum metallurgy was the Institute of Physics and Technology of the Russian Academy of Sciences, which, after the collapse of the USSR, retained a powerful research and experimental industrial base for the development of new alloys and new technologies for the production of products from refractory metals. All research at the institute is carried out in close contact with domestic enterprises in the aerospace, nuclear, and electronic industries, institutes of the Russian Academy of Sciences and leading universities in the country. One of the first new technologies was the “duplex process” of producing ingots. The production of ingots using this technology has made it possible to significantly improve the quality of products from the currently most popular Soviet-developed molybdenum alloys: MChVP, TSM, TsM2A. New technologies for the production of granules from molybdenum alloys have been developed, and the possibility of their use in additive technologies based on laser 3D printing or powder metallurgy technologies has been shown. New molybdenum-based alloys have been developed that significantly exceed existing ones in their strength characteristics. A new direction in materials science of molybdenum-based alloys has been created - highly alloyed alloys, which are fundamentally different in structure from lightly alloyed alloys.

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PROSPECTS FOR THE EFFICIENT PRODUCTION OF NICKEL AND COBALT FROM LATERITE ORES WITH A LOW CONTENT OF VALUABLE COMPONENTS

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Nickel is one of the necessary metals for modern industry. The production of stainless steel, electrical engineering, batteries for electric vehicles, electroplating coatings, chemical catalysts and etc. are the main areas of its uses.

More than 400 deposits of nickel ores have been explored in the world, including about 240 sulfide and more than 150 oxidized ores. The proportion of laterite nickel is approximately 70%, and sulfide nickel is 30%¹. The major producers of nickel from laterite nickel ores are China, Indonesia, the Philippines, Canada, Australia, New Caledonia and Brazil. About 60% of nickel is produced by electric melting, 14% by mine melting, 14% by Caron technology and 12% by sulfuric acid HPAL. But suitable deposits for economically efficient nickel extraction are limited. Sulfide ores are well studied and actively processed, whereas oxidized ores can be efficiently processed only with a nickel content of more than 1.3%. These ores account for only 30% of all reserves in terms of nickel. Cobalt also is an important component of nickel ores. Its consumption for industry is continuously increasing. In IMET RAS, the research to develop an effective method for processing laterite nickel ores is underway. Research object is an laterite ore with a nickel content of about 1%. Purpose is selective extraction of nickel and cobalt and separation of the main part of iron.

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MATHEMATICAL MODELING OF LADLE TREATMENT

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Modeling of the processes occurring during the melting and ladle treatment of steel is a complex task that requires the development of physicochemical models and mathematical algorithms that allow us to describe high-temperature processes occurring in open non-equilibrium metallurgical systems. Physico-chemical descriptions and mathematical models are needed, taking into account the composition of the interacting phases and thermal regimes, reflecting the dynamic characteristics of the melting, dissolving, mixing processes, the interaction speed between all components of the slag and metal phases, etc.

This work is devoted to the development of a correct physico-chemical description of the interaction between the components of the slag-metal system.

A physical and chemical description, mathematical model and a software, describing the process of interaction between the components of the slag-metal system have been created.

The input information for the software are: the geometrical dimensions of the ladle, the initial temperature, the mass and chemical composition of the slag and metal, the time and weight of the additives, the time and intensity of inert gas purge, the electrical mode, the thermodynamic database, etc.

The adequacy of the developed software was checked according to industrial data. Comparison of calculation results using the developed software showed good agreement with experimental data. The developed software can be used for dynamic modeling and optimization of metal processing technologies at the ladle-furnace unit, vacuum-oxygen decarburization units of corrosion-resistant steels.

IMPROVING THE MACHINABILITY OF LEAD-FREE AUTOMATIC STEEL GRADES

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One of the directions for increasing the technical and economic indicators of machining at machine-building plants is the use of steel with increased technological properties, including increased machinability, for the manufacture of machine parts. In Russia, the problem of increasing the machinability of steel has been solved for quite a long time by preliminary heat treatment of workpieces with the formation of a favorable microstructure in the metal. However, it is also possible to increase the machinability of steel at the stage of metallurgical processing, and sometimes this is the only possible solution.

Numerous studies have shown that to ensure optimal machinability, it is necessary to form evenly distributed inclusions of a certain shape in the metal. In automatic steel, sulfur is contained mainly in the form of manganese sulfides, the volume of which depends on the sulfur content, and their morphology - on the degree of deoxidation and the cooling rate of the metal during crystallization.

There are three morphological types of manganese sulfide inclusions: type 1 – globular or slightly deformed inclusions; 2nd type - film or eutectic inclusions, released at the last stages of crystallization in the interdendritic spaces and along the boundaries of crystals in the form of thin films and eutectic colonies; Type 3 – inclusions with a crystalline structure, formed during excessive deoxidation of steel and precipitated on any solid substrate.

To obtain optimal machinability of free-cut steel, it is necessary that the structure of the metal is dominated by large, slightly deformed inclusions of type 1 sulfides.

In the course of research carried out in the conditions of the steelmaking scientific and technical laboratory of STI NUST "MISIS", a technology for the production of automatic grades of lead-free steel was developed, ensuring the production of type 1 inclusions evenly distributed in the steel.

DEVELOPMENT OF FOUNDRY WASTE RECYCLING TECHNOLOGY

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The main element found in foundry waste is silicon. Its involvement in the production turnover is of high value, and the production of alloys containing silicon is a challenge that needs to be addressed by the industry.

A method of recycling foundry waste as a raw material for the manufacture of construction materials is proposed¹. The proposed processing method includes pelletising of the mixture, which is carried out without adding metal reducing agent, and the process itself is carried out with the help of reducing gas (H₂ or CO), and then remelting in an electric arc furnace².

The proposed method of recycling foundry waste is the production of silicon-based alloys containing other elements, which is of interest to a wide range of industries.

This method of processing includes changing the elemental composition of the reaction mixture by adding a salt composition based on alkali metal halides with simultaneous lowering the melting point of the salt mixture, based on the diagrams of state of the corresponding salt mixtures, below 1000°C.

Reduction of the obtained salt flux, with simultaneous alloy formation, it is proposed to conduct mixed reducing agent at temperatures above the melting point of the corresponding metallic component. The obtained salt flux is proposed to use repeatedly, until a certain elemental composition is achieved, and then to carry out processing at the relevant enterprises.

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DEVELOPMENT OF MATERIALS AND TECHNOLOGIES FOR THE PRODUCTION OF NPP EQUIPMENT WITH THE VVER-S-1000 REACTOR PLANT, PROVIDING AN OPERATIONAL LIFE OF UP TO 80 YEARS

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Currently, the service life of the main equipment of nuclear power plants with generation III+ reactors VVER-1200 (project AES-2006) and VVER-TOI made of steels of the 15X2HMΦA and 15X2MΦA families, respectively, is at least 60 years.

These steel grades have a fairly high range of service and technological properties, but fourth-generation reactors put forward new requirements. While maintaining the current level of strength characteristics of vessel steels at room and elevated temperatures, the maximum cross-section of the reactor vessel in the core can reach up to 300 mm, which will lead to enlargement of vessel elements, thereby requiring the solution of serious metallurgical and technological issues

Steel 15X2HMΦA-A(M) is characterized by higher short-term strength characteristics, satisfying the high strength category (yield strength = 500 MPa) at a temperature of 400 °C, compared to steels of the 15X2HMΦA and 15X2MΦA families (yield strength = 450 MPa at 350°C, service life – at least 60 years) at low DBTT, which will allow solving technological problems related to the industrial production of the vessel at the existing capacities of the domestic machine-building industry, in the case of the manufacture of reactor vessel equipment for the VVER-S-1000 project

The resulting experimental industrial billet made of 15X2HMΦA-A(M) steel has high strength and viscoplastic properties and corresponds to the declared level of properties to ensure a strength category not lower than the yield strength of 500 MPa at 400 °C, meets the requirements for metallurgical quality in terms of the content of harmful impurity elements and the score of non-metallic inclusions: no more than 1.0 points of the oxide type point and type silicates are non-deformable, brittle silicates are not detected.

EFFECT OF TITANIUM CONTENT AND THERMO-DEFORMATION PARAMETERS ON THE MECHANICAL PROPERTIES OF HIGH-STRENGTH LOW-CARBON STEELS

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New high-strength low-carbon microalloyed steels are widely used in transport, construction, mechanical engineering, and other segments of modern industry. This is due to the excellent complex of difficult-to-combine technological, mechanical and other service properties, which is achieved by the superposition of a dispersed ferrite matrix and a volumetric system of nanoscale carbide precipitates. Therewith, for steels of various strength levels, microalloying with titanium has an economic advantage. Its maximum efficiency is facilitated by the targeted formation of the most optimal structural state.

To solve this problem, the influence of titanium content and thermo- deformation parameters on the mechanical properties, microstructure, and characteristics of the formed phase precipitates of hot-rolled low-carbon steels with 0.045-0.170 wt.% Ti has been studied both on laboratory and industrial samples. Methods of optical, scanning and transmission electron microscopy were used to study the structural state. To analyse the conditions for formation of nanoscale precipitates of various types and their quantities, thermodynamic calculation of temperature dependences of equilibrium phase composition of steels was carried out.

It has been found that strength characteristics of steels under consideration improves consistently with increasing titanium concentration. The use of elevated temperatures at the end of rolling and coiling promotes the formation of a dispersed and homogeneous microstructure as well as the achievement of a high level of strength and ductility due to the formation of interphase precipitates. At higher cooling rate after hot rolling (30°C/s) nanoscale precipitates are formed in ferrite. As a result, the contribution of precipitation hardening decreases. However, in this case a significantly higher level of grain boundary and dislocation strengthening is observed.

FEATURES PRASEODIMIUM AND NEODIMIUM EXTRACTION FROM NITRIC ACID SOLUTIONS BY MIXTURES OF EXTRACTANTS

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The low Nd/Pr partition coefficient in the TBP-HNO₃ system ($\beta=1,2$) determines the multistage nature of extraction cascades. Taking into account the increasing demand for neodymium, the improvement of existing and search for new extractant systems is timely and relevant.

The limited number of studies of synergistic extraction of light-group REMs by mixtures of quaternary ammonium bases salts and TBP (Figure 1a)¹ determined the direction of research, the results of which shown in the Figure 1b.

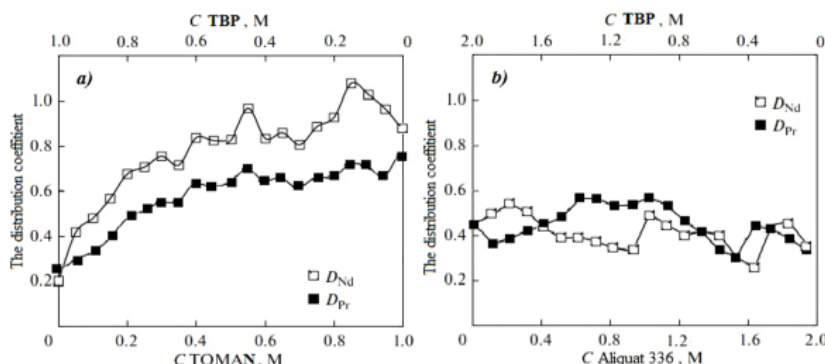


Figure 1. Dependences of the distribution coefficient D_{Nd} and D_{Pr} on the composition of isomolar mixtures TOMAN–TBP¹ in toluene (a) and Aliquat 336–TBP in TS-1 (b) from solutions containing:

a) 0,429 M Nd(NO₃)₃/0,487 M Pr(NO₃)₃; 4,0 M NH₄NO₃; 0,01 M HNO₃;

b) 1,0 M Nd(NO₃)₃/ Pr(NO₃)₃; 2,0 M NH₄NO₃; pH=2

The regions of synergistic and antisynergistic extraction by Aliquat 336-TBP mixtures from concentrated solutions of Nd and Pr (1,0 M) have established. The difference in the manifestation of these effects for Nd and Pr at varying Aliquat 336 concentrations of 0,1-0,3 M and 0,6-1,0 M is promising for their separation.

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COMPARISON OF METHODS FOR DETERMINING THE CONTENT OF NON-METALLIC INCLUSIONS IN STEEL SAMPLES

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The determination of the content of non-metallic inclusions (NMI) in steel is carried out using optical, electron microscopy and fractional gas analysis (FGA) methods, which do not allow for the rapid determination of NMI in the production process due to the duration of the analysis. Therefore, it is urgent to develop an operational method for monitoring NMI based on optical emission analysis PDA, which is capable of providing a result on the composition and amount of NMI in a metal sample within 5-8 minutes.

To develop the method, the results of the determination of NMI in samples cut from various parts of slabs of low-alloy and high-strength steel by optical metallography methods according to ASTM E1245-03 standard, fractional gas analysis of FGA and optical emission analysis of PDA were compared. Studies of samples according to the ASTM E1245-03 standard have shown that in all samples the ratio of oxides and sulfides is on average 10 % and 90 %, respectively. The content of inclusions in the samples was determined using optical emission analysis PDA. When examining samples using the PDA method, the number of flashes of intensity of spectral lines of the main elements forming non-metallic inclusions in steel was determined. The average intensity value corresponds to the content of the dissolved element in the matrix of the isolated element, and the emission corresponds to the ingress of a spark into the NMI. The results of determining the volume fraction of non-metallic inclusions by FGA and optical metallography methods were compared with the number of inclusions determined in the samples by the PDA method. A comparison of the results of the determination of HB shows the possibility of assessing the contamination of NMI using the PDA method. Based on the results of the determination of the volume fraction of inclusions by the ASTM E1245-03 method, a relationship was found between the volume fraction of non-metallic inclusions and the intensities of the spectral lines of the isolated elements. Based on the results obtained, a correlation equation was established and the volume fractions of non-metallic inclusions in metal samples were calculated.

The results of the calculation of spectral data arrays were compared with the results of determining the volume fraction of non-metallic inclusions using quantitative optical microscopy according to ASTM E1245-03 standard. The results showed good convergence.

OBTAINING A POWDER COMPOSITION FROM 42HNM ALLOY AND SYNTHESIS OF SAMPLES FOR TESTING USING LAYER-BY-LAYER LASER SURFACING TECHNOLOGY

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Alloy 42HNM is a promising material for nuclear energy. Possessing high corrosion resistance in water of high parameters, the alloy is not prone to stress corrosion cracking and steam-zirconium reaction, and is not susceptible to corrosion under irradiation. Initially, the 42HNM alloy was used as a material for the cores of VVER reactors. Currently, the scope of application of shells made of alloy 42HNM has expanded significantly¹.

42HNM is of significant interest from the point of view of producing the alloy using additive technologies. The feasibility of using additive technologies in comparison with traditional ones requires careful study, which is impossible without studying changes in the structure and properties of the deposited material.

This paper presents the results of metallographic, X-ray phase studies and electron microscopy of 42HNM alloy powder obtained by gas atomization. The influence of atomization parameters on the granulometric composition and morphology of the resulting powder is considered. Samples for mechanical testing were made from the powder using layer-by-layer laser fusion. For comparison, samples were made from the same alloy obtained by the traditional method. Comparative mechanical tensile tests showed better properties for samples made by casting. It has been shown that obtaining the required microstructure of the material, eliminating porosity and increasing the mechanical properties of products produced by the additive method can be achieved by appropriate selection of laser melting modes.

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RESEARCH NONMETALLIC INCLUSIONS AND PRECIPITATES IN ELECTRICAL NON-ORIENTED STEELS

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Nonmetallic inclusions (NMIs) reduce the technological, mechanical and magnetic properties of non-oriented steels (NGO). To control the volume fraction of inclusions, it is necessary to identify inclusions, obtain statistically significant estimates of the number and size, and determine the stage of their formation.

In this work, NMIs and precipitates in NGO containing 3%Si, 1%Al were studied. The samples differed in the intermediate thickness of cold rolling (≈ 0.6 mm and ≈ 1 mm) the amount of Mn – 0.1-0.3%, 0.5-0.7% and 0.8-1.2%.

Automated features analysis based on SEM-EDS (Tescan Mira 3) was applied to evaluate large primary, secondary and tertiary NMIs in NGO provoke the formation of surface defects and cracks in the strips during rolling. Primary and secondary inclusions are represented by calcium aluminates $n\text{Al}_2\text{O}_3 \cdot m\text{CaO}$ with a high content of CaO, as well as CaS inclusions formed at the stage of ladle treatment during modification with calcium. Another large cluster is inclusions of magnesium spinel and pure magnesium oxide, the nature of the formation of which is associated with the active interaction of the melt and the lining. The third cluster is inclusions based on aluminum nitride, formed during solidification due to nitrogen segregation.

To evaluate the precipitates, which are formed in a solid metal and which reduce the magnetic properties, they were assessed analyzed using TEM, Talos F200i. Identification was carried out using EDS analysis. There are aluminum and titanium nitrides, and copper sulfides. It has been shown that their formation is possible only at the final stage of processing during cooling after recrystallization annealing.

CALCULATION OF THE Ag–Pd–Sn SYSTEM USING AN ASSOCIATED SOLUTION MODEL FOR THE MELT

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Palladium-based alloys are widely used in various industries. To develop new materials, it is essential to rely on phase diagrams. Thermodynamic modeling combines all experimental information about phase boundaries and the thermodynamic properties of different phases, allowing us to extrapolate these data to unexplored compositions and higher-order systems. This significantly reduces the time and cost required to create state diagrams for multicomponent systems.

The previous calculations of the Ag–Pd–Sn system, using the Redlich–Kister–Muggianu¹ model to describe the melt, revealed some issues. The main one was a strong tendency towards the formation of spurious miscibility gaps in calculated melt and FCC solid solution. Therefore we have performed a new CALPHAD calculation of this system, using for melt an associated solution model that is considered to be more effective for systems with strong interatomic interactions.

Optimization of the parameters for the phase models was performed by the PARROT module of the Thermo-Calc® package. The initial data for optimization were the results of experimental studies on phase equilibria² at temperatures of 500 and 800 °C, the enthalpy of formation of the melt³, and the activity of components in the liquid phase⁴. These data was supplemented by experimental study on the temperatures and heats of melting for alloys belonged to the homogeneity range of the FCC solid solution using the DTA/DSC technique (Jupiter STA 449 F1).

The use of an associated solution model allowed for a more accurate thermodynamic description of the Ag–Pd–Sn system. Good agreement was achieved with experimental data on phase boundaries and thermodynamic properties of the liquid phase, without any spurious miscibility gaps observed over the entire composition range and a wide range of temperatures.

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KINETICS OF INTERACTION OF WOLFRAMITE CONCENTRATE WITH PO-TASSIUM CARBONATE

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The sintering method with Na_2CO_3 has become widespread in the processing of wolframite concentrates. One of the disadvantages of using sodium salts in sintering is the formation of low-melting compounds that affect the kinetics of the process. There are known [1] studies aimed at intensifying technological processes by replacing Na_2CO_3 with K_2CO_3 . However, data on the kinetics and mechanism of interaction in mixtures of wolframite ($\text{Fe}_x\text{Mn}_{1-x}\text{WO}_4$) – potassium carbonate under heating are very limited.

According to the thermal analysis, the beginning of interaction of the wolframite concentrate with K_2CO_3 was noted at 520°C (Figure) accompanied by a decrease in mass and the release of CO_2 into the gas phase. Endothermic effect with onset/maximum at $699/721^\circ\text{C}$ corresponded to the melting of the eutectic $\text{K}_2\text{WO}_4\text{--K}_2\text{CO}_3$.

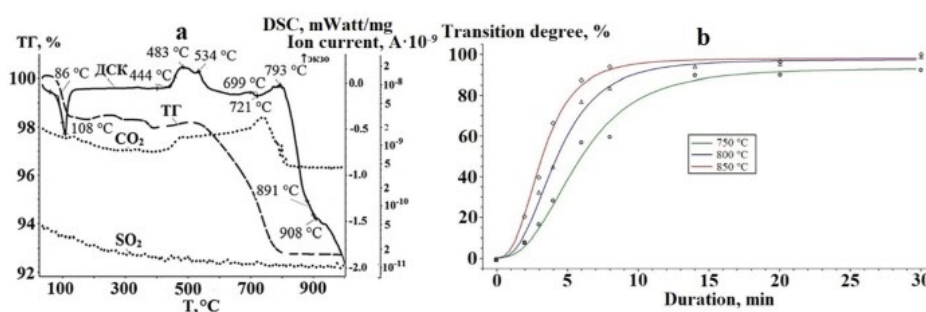


Figure Sintering of W-concentrate with K_2CO_3 : continuous (a) and isothermal heating (b)

According to the results of isothermal sintering experiments (air, $750\text{--}850^\circ\text{C}$) mixtures of concentrate with K_2CO_3 and subsequent aqueous leaching of the sinter (liquid:solid = 10, 90°C , 60 min) curves were obtained linking the degree of transition of tungsten to K_2WO_4 with the duration of roasting. The processing of experimental data in the Netzsch Thermokinetics 3.0 software package made it possible to identify the best kinetic model of the sintering process with a limiting stage of autocatalytic Prout-Tompkins nucleation with an activation energy equal to 56.8 kJ/mol .

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The work was performed on the State Assignment of the IMET of the Ural Branch of the Russian Academy of Sciences (state registration number of the topic: 122020100404-2) using the equipment of the Ural-M Central Research Center.

DEVELOPMENT OF TECHNOLOGY FOR PRODUCTION OF BISMUTH-CONTAINING GROUPS OF FREE-CUTTING STEEL GRADES

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Bismuth is one of the most promising elements that allow to largely eliminate the use of sulfur and lead added to modern free cutting steels¹. In this work the technology of obtaining bismuth-containing groups of grades of free-cutting steels is developed. Designing of the technology of obtaining steel with bismuth has been carried out. Testing of the technology in laboratory and industrial conditions has been carried out.

On the basis of experimental and calculated study, the optimal concentrations of bismuth and sulfur necessary for the formation of the required number of inclusions² were determined.

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The research is funded by the Ministry of Science and Higher Education of the Russian Federation within the framework of the World-Class Research Center: Advanced Digital Technologies program (agreement No. 075-15-2020-311 of 04/20/2022).

THE EFFECT OF B_2O_3 ON THE VISCOSITY OF HIGH-MAGNESIA BLAST FURNACE SLAGS

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Despite the acute shortage of iron ore raw materials, the carbonate iron (siderite) ores located at the Bakal deposits having its balance reserve of about 1 billion tons are not in demand among the Urals metallurgists due to its high magnesium content.

The slag transition from a solid to a liquid state occurs in a certain temperature range, therefore, the melting point (T_{melt}) is a conditional value. It is often mistaken for the temperature at which the slag begins to freely flow from the coke nozzle, which is possible when its viscosity becomes less than $2.5 \text{ Pa}\cdot\text{s}$.

For the successful course of blast furnace melting and ensuring the safety of blast furnace production, T_{melt} of slag shall be below 1400°C , and at tapping temperature (T_{tap}) (1400 to 1500°C) it shall have good mobility (viscosity less than $0.5 \text{ Pa}\cdot\text{s}$).

In slag systems $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-B}_2\text{O}_3$ containing

$$\frac{\text{CaO}}{\text{SiO}_2} = R = 0,9 \div 1,2, \text{MgO} = 15 - 36\%, \text{B}_2\text{O}_3 = 0 - 15\%, \text{Al}_2\text{O}_3 = 5 - 20\%$$

with no boric anhydride, and a basicity of less than 1.1, MgO content below 20% and $\text{Al}_2\text{O}_3/\text{MgO}$ ratio of more than 0.5, T_{melt} is within the range of $1300\text{-}1400^\circ\text{C}$, and a viscosity of $0.5 \text{ Pa}\cdot\text{s}$ is achieved at temperatures of $1320\text{-}1440^\circ\text{C}$. Blast furnace melting having slags of this composition will proceed without difficulty.

A further increase in the magnesium oxide content to 36% results in a sharp increase in the melting temperature up to 1500°C with a decrease in the crystallization interval, and the temperatures ensuring the tapping viscosity increase up to 1540°C which makes melting with such slags difficult or impossible.

B_2O_3 additives result in T_{melt} decrease, and 15% of B_2O_3 in the melt makes T_{melt} less than 1150°C , and T_{tap} less than 1500°C . The slags become long and stable, which makes it possible to conduct blast furnace melting with slags containing about 40% MgO, corresponding to the proportion of siderites in the initial charge of 40-50%.

The work was carried out according to the State assignment of the Institute of metallurgy, Ural branch of the RAS, using the equipment of the Ural-M Shared Use Center.

VISCOSITY OF SLAGS FROM MATTE SMELTING OF NICKEL AND COPPER ORES

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Viscosity of molten slags from matte smelting of oxidized nickel and sulfide copper ores is evaluated. To conduct research, model slags were synthesized that were close in composition and structure to real samples, and limiting the investigated range of slag compositions (wt.%): iron-containing primary slag (8,9 CaO, 11,8 MgO, 12,5 Al₂O₃, 47,4 SiO₂, 13,3 FeO и 5,0 Fe₂O₃) and iron-free primary slag formed by removing iron oxides from iron-containing primary slag (12,5 CaO, 16,0 MgO, 9,4 Al₂O₃ и 58,3 SiO₂). Theoretical (computations with Kalmanovitch–Frank model) viscosity (η) estimation has shown that in the interval 1400–1550 °C it is possible to continuously remove up to 80 % of iron oxides from the iron-containing primary sample with maintaining the viscosity level ($\eta = 70$ dPa·s) acceptable for industrial practice of matte smelting; to maintain this level at deeper removal of iron oxides (in order to transition to the iron-free primary model slag) it is necessary to adjust the composition by adding lime or magnesia. The experimental values of η for iron-containing (1550–1300 °C) and iron-free (1550–1400 °C) primary model slags obtained by vibrational method are in the ranges of 3.1–23.3 and 12.8–45.5 dPa·s, respectively. Regression analysis of the experimental data was carried out in the framework of the Weimann–Frenkel–Urbain formalism. The values of the viscous flow activation energy (E_η) of primary model slags were determined, and it was shown that reducing the slag basicity from 0.7 (iron-containing slag) to 0.6 (iron-free slag) increase E_η from 204 to 236 kJ/mol. Empirical models are proposed that make it possible to obtain refined values of the viscosity of the primary model slags in the considered temperature range. It was found that to maintain optimal slag viscosity, joint matte smelting of oxidized nickel and sulfide copper ores of studied compositions should be carried out at a temperature not lower than 1400 °C. The results obtained can be used in the development and improvement of pyrometallurgical technologies of mineral and technogenic raw materials of non-ferrous and ferrous metallurgy, as well as in the glass making and ceramics industries.

The work was carried out according to the State assignment for IMET UB RAS (State registration number of the topic: 122020100404-2).

GALLIUM PASTES: SINGLE-COMPONENT STARTING MATERIAL AND IN SITU SYNTHESIS OF LIQUID EUTECTIC

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One of the most important areas of use of gallium-based alloys is diffusion-hardening alloys and lead-free composite solders. Their unique property is the ability to prepare, mold and cure at room temperature.

The method presented in this report for the synthesis of a semi-liquid hardening paste-like composition exclusively from solid pre-dispersed components made it possible to provide the best parameters for the manufacture of the material, to study the structural and physical-mechanical characteristics of the initial components, gallium pastes and hardened alloys.

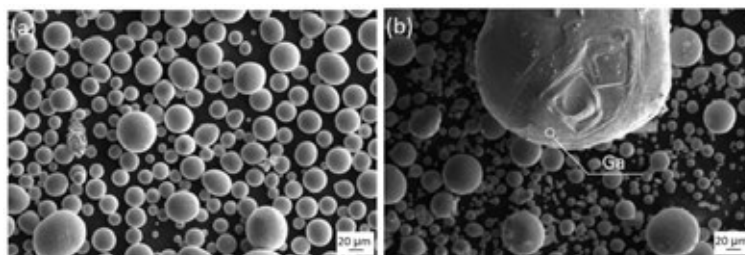


Figure 1. (a) – spherical particles of a mixture of Cu_3Sn and Sn filler powders;
(b) – a particle of solid dispersed gallium against the background of filler powders in the mass of the starting material (SEM, secondary electron detector (SE)).

The resulting initial mixture (Figure (1b)) is stable at room temperature. It shows no signs of interaction between the components until intense vibration in the amalgam mixer (in special capsules).

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The work was carried out according to the State Assignment of the Institute of Metallurgy, Ural Branch, Russian Academy of Sciences.

WEAR-RESISTANT LAMINATE IRON ALUMINIDE COATINGS ON CARBON STEEL

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One of the ways to minimize the wear of transporting, storage, mixing, pressing and other equipment in the anode production of aluminum smelters is to form coatings based on iron aluminides on the surface of parts of equipment in contact with the anode mass made of carbon or low-alloy steels FeAl and Fe₃Al. Intermetallic compounds based on aluminum and iron have fairly high hardness and wear resistance. Such coatings can implement a set of the most popular functional characteristics at the required level and reduce the amount of steel particles entering the raw material when cleaning equipment walls.

FeAl/Fe₃Al/Fe(Al) coatings are formed on a steel base using explosion welding of St3 steel with AD1 aluminum and subsequent heat treatment (Fig. 1). Heat treatment includes heating 660 °C, 3 hours + 640 °C, 3 hours, ensuring the formation of Fe₂Al₅ and separation of the unreacted aluminum layer and 1100 °C, 1.5 hours to transform the coating surface to the FeAl intermetallic compound.

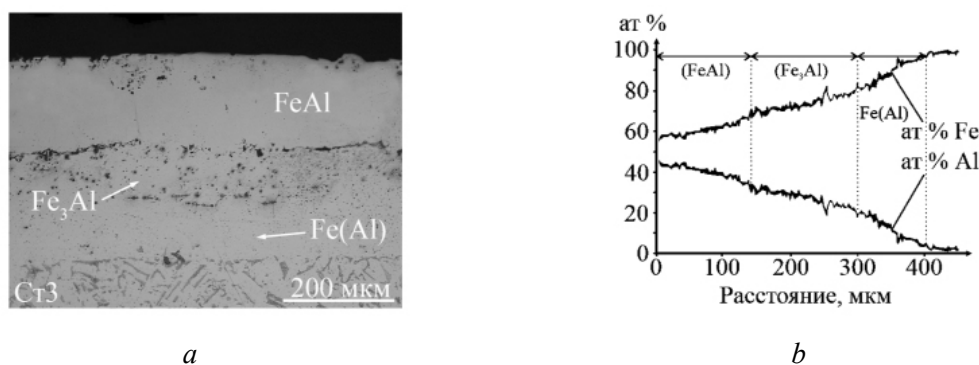


Figure 1. Coating structure (a) and distribution of chemical elements (b)

Tests of coatings under hot wear conditions showed that the main mechanism of destruction is cohesive. Peeling and chipping of coatings occurs only under heavy loads. It has been established that the hardness of a layered FeAl/Fe₃Al/Fe(Al) coating with a thickness of 440 μm increases from the substrate to the surface, reaches a maximum at a distance of 200 μm from the surface, and then decreases due to an increase in porosity. Its relative wear resistance is ~1.5 times higher than the St3 steel base.

The work was supported by Russian Science Foundation project No. 24-29-00231, <https://rscf.ru/en/project/24-29-00231/>

TECHNOLOGY OF DESULFURATION AND MICROLLOYING OF LOW CARBON STEEL WITH BORON UNDER SLAG OF THE $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3\text{-B}_2\text{O}_3$ SYSTEM

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One of the factors affecting the quality of steel is the sulfur content in the metal. To produce steels with low (0.010-0.015%) and ultra-low (0.002-0.004%) sulfur content, the desulfurization process is carried out in ladle-furnace units (LFU). The process of forming highly basic slags of the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ system is carried out with the addition of fluorspar. However, the short-term effect of fluorspar on the physical and chemical properties of slag does not provide the necessary desulfurization of steel. The environmental hazards of volatile fluorides and their aggressiveness towards equipment and the refractory lining of steel-pouring ladles necessitate the development of fluoride-free slags with low viscosity and high refining properties. One way to solve the problem is to use boron oxides instead of fluorspar.

Along with deep metal desulfurization, a promising direction for solving the problem of improving the quality and reducing the cost of metal products is steel microalloying. A special place among microalloying elements is occupied by boron, the introduction of which into the metal in an amount of 0.001–0.003% makes it possible to increase the strength of steel without reducing ductility.

Microalloying of steel with boron is carried out, as a rule, by adding boron-containing ferroalloys, the use of which increases the cost of steel. One of the ways to solve the problem of microalloying steel with boron at minimal cost may be direct microalloying through the reduction of boron from the oxide system.

The paper presents the results of fundamental studies of the physicochemical properties of the $\text{CaO-SiO}_2\text{-B}_2\text{O}_3$ oxide system containing 15% Al_2O_3 and 8% MgO .

The work was carried out according to the state assignment for IMET UB RAS

ABOUT THE USAGE OF UNDERWATER PLASMA FOR MODIFYING THE SURFACE OF BELT AMORPHOUS ALLOYS FeSiB

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Samples of amorphous electrical steel (foil) were strips about 100 μm thick and 45 mm wide, which were obtained by ultra-fast cooling by spraying the melt on a rotating copper drum. Subsequent processing of the materials was carried out in pulsed underwater low-temperature plasma [1]. The structure of the foil surface was studied using atomic force microscopy. The effect of weak magnetic field pulses [2, 3] on amorphous alloys was also studied. Unannealed amorphous alloys of the composition $\text{Fe}_{73}\text{Si}_9\text{B}_{13}$ and with the addition of 1% Cu, which affects the clustering process during subsequent thermal annealing, were studied. The influence of processing modes in low-temperature plasma and magnetic pulse processing on the surface structure and magnetic properties of materials is described.

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CRITERION OF UNIFORMITY OF MIXING OF NANO- AND MICRO-POWDERS

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Nowadays, mixing of nano- and micro-materials is used in many advanced technologies. However, the realization of this process is conditioned by a number of factors such as density, particle size and shape, etc., which can lead to different mixing uniformity of mixture components. Therefore, mixing control should be carried out to obtain quality products.

Based on this, the aim of this paper was to select criteria to quantify the quality of mixing quality.

For mathematical calculations it is assumed that at ideal mixing a layer of nanoparticles is formed on the surface of a micron particle. The scheme of micron particle powdering is presented in Figure 1a. To evaluate the uniformity of mixing, the use of the value of the total area of the areas occupied by nanoparticles on EDX maps of the elements is proposed. Figure 1b shows the distribution map of the Cu-Co₃O₄ mixture. This criterion is tested on mixtures of micron-sized Cu, Ni, W powders and nanoscale Co₃O₄ powder.

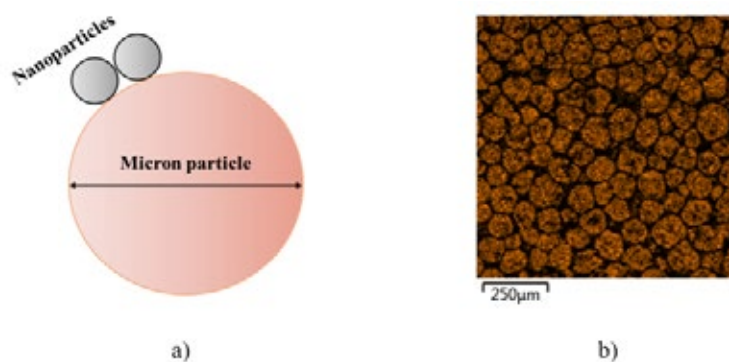


Figure 1. Schematic of micron particle nanoparticle powdering (a) and copper distribution map (b) in Cu+Co₃O₄ mixture

This work was financially supported by the Russian Science Foundation (Project No. 19-79-30025-P).

EVALUATION OF THE PROPERTIES OF TITANOMAGNETITE IRON ORE MATERIALS TO DETERMINE THEIR METALLIZATION ABILITY

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In order to utilize Ural titanomagnetite ores, innovative technologies are required¹⁻³. One such technology involves metallized titanomagnetite pellets followed by electrosmelting. Modern electric steelmaking technologies have specific material requirements, including a metallization degree of over 90%, high metallic iron content, and low FeO content, to ensure strength. The IMET UB RAS is researching the feasibility of producing titanomagnetite metallized pellets for steelmaking from upgraded concentrates of the Gusevogorsky and Pervouralsky deposits. The beneficiation schemes were determined, and the chemical, phase, and mineralogical compositions of the concentrates (with a total iron content of over 65% for both deposits) and pellets were studied. The results demonstrate that titanomagnetite iron ore is suitable for the metallization process, achieving a degree of metallization of 92% or higher when reduced by gas under specific conditions.

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THE ENTHALPY OF FORMATION OF CRYSTAL HYDRATES, DOUBLE AND BASIC SALTS CALCULATION ADJUSTED FOR THE SHARE CONTRIBUTIONS OF BINDING ENERGIES

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The chemical compounds enthalpy of formation, including natural origin, determines the thermal effects of chemical and metallurgical processes reactions and the specific energy of atomization - energy density, which is associated with the physical properties of a chemical compound.

Based on the analysis of the enthalpies of formation calculation of forty complex inorganic compounds of copper, nickel, zinc, iron, lead and rare earth metals according to the equation of V.V. Zuev¹, which was developed using the concept of electronegativity, identified the necessity for refine the calculation of the enthalpy of formation for crystalline hydrates, double and basic salts. The equation has been developed that adjusted for the share contributions of binding energies and interactions of hydrogen groups in crystalline hydrates:

$$\Delta H_{298}^0((M_1)_{k_1} \dots (M_n)_{k_n} A_m) = \sum_{n=1}^N \left[x_n \cdot \Delta H_{298}^0((M_n)_{y_n} A_{x_n}) \right] - \sum_{i=1}^{N-1} \sum_{j=i+1}^N \left[\frac{1}{Kq_i} \cdot \left(\sum_{z=1}^{Kq_i} \frac{q_z}{\sqrt{Q_z - 1}} \right) \cdot \left(\frac{\partial \sigma_i - \partial \sigma_j}{0,102} \right)^2 \right],$$

где CN – atomic coordination number;

z – bond counter of the coordination number of the less electronegative atom;

q – number of the same name atoms $Mi(A_i)$, that are connected to a more electronegative atom/s $Mj(A_j)$ through a common atom

$(Mi-A-Mj/ Ai-M-Aj)$;

Q – total number of a common atom bonds;

r – the indicator adjusted for options for the interaction of hydrogen-containing groups (H_2O) in crystalline hydrates

The calculation error compared to the reference data decreased for antlerite from 25.7 to 1.1%, langite from 20.1 to 0.6%, devilline from 25.9 to less than 0.1%, lanthanite from 8.7-9.1 to less than 0.3%.

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ANALYSIS AND ADJUSTMENT OF LADLE TREATMENT TECHNOLOGY OF ISOTROPIC STEEL

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The work included an analysis of heat certificates and a study of metal samples taken at the main stages of ladle treatment (RH degasser, ladle treatment stand), from a tundish, from slabs and hot-rolled isotropic steel sheets. The samples were studied using fractional gas analysis (FGA) and scanning electron microscopy with X-ray microanalysis. To assess the size, morphology and chemical composition of inclusions was used the method of X-ray spectral microanalysis. The purpose of this work was to analyze the reasons for the formation of various types of non-metallic inclusions (NMI) and adjust the technology to reduce the overall contamination of the metal.

The FGA results showed the dynamics of changes in the content of various types of non-metallic inclusions at all stages of the technology. It has been established that oxygen is contained in four main types of NMI: silicates, oxynitrides, aluminates and spinels. In metal samples taken from the stages of ladle treatment were detected large oxide non-metallic inclusions and films type by electron microscopy. In samples taken from the tundish was noted the presence of oxysulfide inclusions, which are oxysulfides based on CaS with various oxides based on the Al-Mg-Ca-O system. In samples taken from slabs and hot-rolled sheets were found CaS-based sulfide and oxysulfide inclusions, aluminum nitrides (AlN), complex Al-Mg-N-based nitrides, oxynitrides and various Al-Mg-O-based oxides.

Based on the analysis of heat certificates and laboratory studies, were given recommendations for adjustment of ladle treatment technology.

Test melts were carried out taking into account these recommendations and metal analysis was carried out on similarly selected samples. By method electron microscopy in the selected samples were found non-metallic inclusions similar to those that were in the samples of melts before the introduction of corrective actions. At the same time, the FGA method showed a decrease of contamination of oxide non-metallic inclusions in samples taken from the slabs.

PRODUCTION OF 'GREEN STEEL' AND HYDROGEN METALLURGY: A BRIEF REVIEW OF RESEARCH

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Currently, the metallurgical industry is faced with demands to reduce carbon dioxide emissions. It is known that the steelmaking sector accounts for 7-8% of CO₂ emissions, prompting metallurgical companies to strive for technologies that allow to produce metallurgical products with minimal use of fossil fuels. According to preliminary assessments by Bloomberg, to achieve zero emissions in the industry by 2050, an investment ranging from \$215 billion to \$278 billion will be required.

Given the magnitude of the necessary investments, governments and producers in various countries are seeking to collaborate towards a common goal. For instance, the Ultra-Low CO₂ Steelmakers Consortium (ULCOS) in Europe has set a target to reduce CO₂ emissions by more than 50% within the next 5-15 years. Companies such as Vale (Brazil), Jindal Shadeed Group (India), H₂ Green Steel (Canada), Thyssenkrupp (Germany), Blastr (Finland), Hydnum Steel (Spain), Oshivela (Namibia), Nippon Steel (Japan), Baowu and HBIS Group (China), Metalloinvest (Russia), among others, are investing significant funds in the construction of new facilities based on low-carbon technologies.

Significant attention is being given to the technology of direct iron reduction using "green hydrogen" as a reducing agent, which is produced through electrolysis using renewable energy. The process involves stages of ore preparation, hydrogen reduction in a shaft furnace (Midrex), and melting of the metallized intermediate product in an electric arc furnace. The world's first pilot steel plant utilizing this technology (HYBRIT) was opened in Sweden in 2020.

Given the relevance of the issue, there has been a growth in the number of scientific studies worldwide dedicated to various aspects of metal production without the use of fossil fuels. From 2010 to 2024, the number of such studies has increased by more than 6 times, with over 50 review articles published alone.

This paper presents an analysis of publications on this topic.

The work was carried out under government contract No. 075-00320-24-00



SECTION 4

**RESOURCE SAVING,
ENVIRONMENTAL SAFETY
AND CHEMICAL-TECHNOLOGICAL
PROCESSES IN CIRCULAR
ECONOMY**

THE METHODOLOGY OF CHROMATO – MASS-SPECTROMETRIC DIAGNOSTICS OF ENERGY EFFICIENCY AND ENVIRONMENTAL SAFETY OF CHEMICAL TECHNOLOGIES

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The creation of new chemical technologies is a fundamental task of modern science. Taking into account the complexity, duration and resource intensity of such developments, methods that allow them to be evaluated quickly and reliably are of particular importance. Chromatography – mass-spectrometry can be one of such methods.

Traditionally, chromatography – mass-spectrometry (CMS) is considered only as an analytical method, but modern advances have made it possible to expand the scope of application and use it to determine the physico-chemical characteristics of substances and materials. Numerous variants of CMS: gas, supercritical and liquid chromatography with mass spectrometry, two-dimensional variants of methods (GC/GC, MS/MS) in the on-line version, thin-layer chromatography in the off-line version, allow you to cover all the tasks that arise when evaluating technologies.

A feature of modern CMS is an integrated approach to obtaining a result based on the joint use of mass spectrometric, chromatographic and computational data obtained by classical and new methods, including using neural networks¹.

Using the example of technologies used in the rocket and space industry, the report shows the advantages and limitations of the CMS method in technology assessment. The technologies of production of rocket and jet fuels, catalytic processes used in the application of fuels, technologies for neutralizing surfaces of structural materials and optimization of environmental technologies aimed at reducing the negative consequences of rocket and space activities are considered².

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PHYSICO-CHEMICAL FUNDAMENTALS, RESEARCH DEVELOPMENT AND IMPLEMENTATION OF RESOURCE-ENERGY-EFFICIENT ENVIRONMENTALLY SAFE WASTE PROCESSING WITH NON-FERROUS AND RARE METALS EXTRACTION

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The enterprises of ferrous and non-ferrous metallurgy and chemical industry have accumulated the hundreds of millions of tons of zinc-containing waste of hazard classes 2-4, the processing of which makes it possible to replenish the raw material base of factories, improve the environmental situation and solve the main task of circular economy to create the waste-free production.

The basic scientific and technical idea based on Waelz technology is distillation separation of elements of multicomponent oxide systems under reducing conditions.

The series of studies, development and implementation of Waelz process were carried out for zinc, lead, tin, indium, cadmium extraction as well as the combined technology for products production in the form of compact metals, powders, concentrates, salts and oxides.

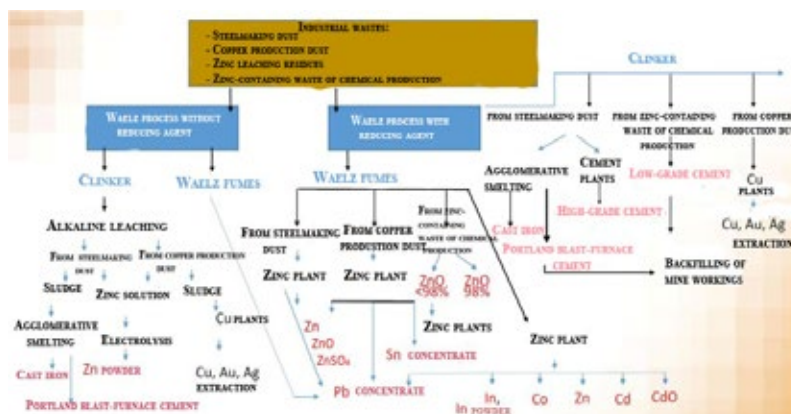


Fig.1. Industrial waste processing flow diagram

The process flow diagram was made based on research work, patented technical solutions performed at UMMC Technical University and implemented at Chelyabinsk Zinc Plant. The ZINCUM, LLC Company is working to start-up the complex for processing more than 100 thousand tons of steelmaking dust per year.

TASKS OF SCIENTIFIC AND TECHNOLOGICAL SUPPORT OF ENVIRONMENTALLY SAFE INTEGRATED PROCESSING OF MINERAL AND MAN-MADE RAW MATERIALS IN ORDER TO DEVELOP A CLOSED-CYCLE ECONOMY

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The subject of discussion is the objective need to move from the development of economic activity according to a linear model to the construction of a closed-loop economy. The analysis of the reasons for the insufficient provision of the Russian metallurgical industry with domestic material resources is carried out. There is a given brief overview of currently unused mineral raw deposits and huge volumes of unprocessed man-made waste containing a significant amount of scarce elements. The demand of the Russian economy for a number of strategic metals is fully met by imports, which, in an unstable international situation, poses a threat to the country's economic sovereignty. The possibility of expanded using of the complex raw material base of the North-Western region of Russia is discussed. As one of the aspects of solving the problem of shortage of raw materials, as well as eliminating environmentally hazardous effects on the environment and human health, technological solutions aimed at the reuse of current waste and previously accumulated technogenic formations are being sought. Particular importance is attached to the development of technologies for the extraction of rare and rare earth elements used in high-tech, including military, industries. The activities of the TECHNOGEN congresses, which have been held in Yekaterinburg every two years since 2012, are devoted to the collection, discussion and systematization of information about current developments. Based on the results of the presented researches, a range of developers of specific problems is determined; an ongoing register of resource-saving technological solutions is being maintained. Some of them have already been introduced into production and contribute to the creation of a closed-cycle economy. A number of examples of promising waste recycling technologies in ferrous and non-ferrous metallurgy, recovery of solid domestic and organic anthropogenic products are considered. It is noted that there is a need to accelerate the pace of innovation in practice.

INNOVATIVE TECHNOLOGIES FOR DESIGNING DIGITAL INFORMATION MODELS OF CHEMICAL-TECHNOLOGICAL OBJECTS WITH WASTE RECYCLING

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Research of multi-product industrial production (oil refining, steelmaking, polymer) with waste recycling showed the relevance of solving the complex problem of resource-saving design and production control, taking into account energy and material resources. The industries under consideration are characterized by large amounts of information, are distinguished by multi-factor interactions and connections, the presence of a changing composition of raw materials, strict requirements for waste capacity and environmental characteristics¹. Innovative technologies for the development of digital information models are proposed to solve the problem of designing chemical-technological facilities with waste recycling. Digital information models include customizable databases of characteristics of chemical-technological objects, bases of design rules, virtual models of equipment, libraries of mathematical models for calibration calculations of equipment and research of cause-and-effect relationships in the control object². Digital information models allow you to generate a variety of design solutions that display the characteristics of chemical-technological objects in the form of a set of information-rich elements in accordance with the requirements of the technical specifications, as well as generate multilingual design documentation. Digital information models are developed using technologies of artificial intelligence, machine learning, industrial data mining, virtual and augmented reality. The use of innovative technologies for designing digital information models of chemical-technological objects with waste recycling makes it possible to reduce the time and number of errors in the design and generation of documentation, and increase the level of automation of design stages.

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SCIENTIFIC BASIS OF METAL FORMING PROCESSES INTENSIFICATION BY CREATING AN OPTIMAL CRYSTALLOGRAPHIC STRUCTURE OF BLANKS DURING ROLLING

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During rolling it is necessary to create such structure crystallography in sheet materials, which ensures an increase in the material utilization coefficient, the formability of blanks and the operational characteristics of parts.

To solve the problems of calculating and controlling the formation of the optimal crystallographic orientation of the structure in sheet materials, a variant of the plasticity theory is developed, in the main equations of which such parameters of the materials structure as elastic constants of the crystal lattice and orientation factors of the texture are introduced. These equations allow to solve the direct problem, that is, to determine the stress-strain state, the force and the formability of textured blanks in specific sheet metal forming processes (deep drawing, wrap-stretching, expansion, flanging, etc.). Also these equations allow to solve the inverse problem - to determine by calculation the necessary crystallographic orientation of the material structure and the corresponding values of anisotropy indicators that meet the requirements for the intensification of sheet metal forming processes. Finally, it is necessary to create a computational model of the composition of the texture components of an ideal structural material and develop technological recommendations for its implementation during rolling and heat treatment of sheet metal.

In general, as a result of the conducted research, the direction of intensifying the metal forming processes and improving the operational characteristics of parts is formulated. Its essence lies in the fact that with the help of texture design, it is possible to change the predominant development of strains during sheet metal forming in favor of those directions that will lead to an increase in the blanks formability, the accuracy of geometric parameters and fatigue characteristics of products.

THE ROLE OF BEST AVAILABLE TECHNIQUES IN ENHANCING INDUSTRIAL RESOURCE EFFICIENCY AND ENVIRONMENTAL PERFORMANCE

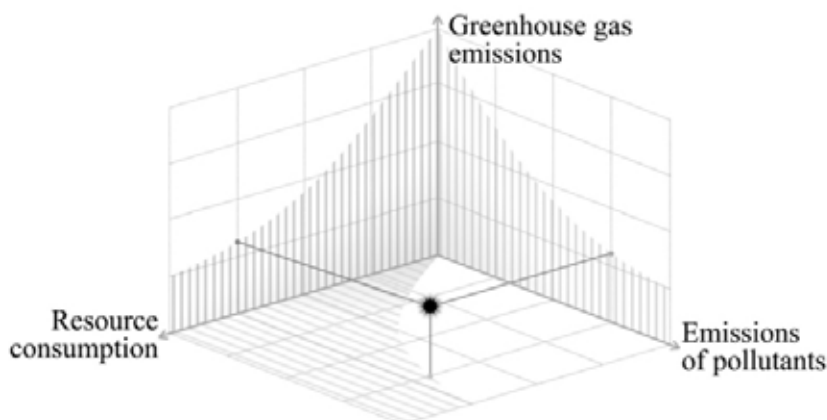
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The Best Available Techniques (BAT) are a set of technological, technical and managerial solutions, the practical application of which allows industrial enterprises to achieve high resource efficiency and good environmental performance (including reduction of greenhouse gas emissions)¹.

In Russia, BAT implementation requirements are set for more than 40 sectors of industry; BAT Reference Documents (BREFs) have been developed for all sectors. As a result of the sectoral benchmarking, three groups of BAT indicators are established, namely BAT-associated emission levels (for emissions of key pollutants), BAT-associated resource efficiency levels, and indicative carbon intensity levels.

In Russia, BREFs are applied for the purposes of the environmental regulations as well as for developing stimuli for industrial enterprises to enhance production resource efficiency and to implement green projects².



Investments in BAT-associated environmental and technological modernization projects, planned (and already being implemented) for the period from 2019 to 2028, already exceed 3 trillion Rubles.

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MANAGEMENT OF SECONDARY RESOURCES IN THE OPERATIONS OF A METALLURGICAL ENTERPRISE

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Management of secondary resources makes it possible to minimize waste generation and contribute to the environmental sustainability of the territory where an industrial enterprise operates. It will allow industrial enterprises to reduce the anthropogenic load on the environment and contribute to the circular economy¹.

Table 1. Formation of by-products in the main workshops of JSC TAGMET

Workshop name	Name of waste/by-product
TPC	Scale (rolled production)
ESPC	Slag intermediate product
	Ferrous metal scrap
	Ferrous metal gas purification dust

Effects when processing mill scale: allow you to reduce the amount of waste generated and reduce disposal costs.

Effects when processing the semiproduct of slag metallurgical melt of electric furnace production: will make it possible to obtain fractional slag crushed stone, reduce the use of auxiliary materials due to recycling of the share of recovered scrap, increase revenue by selling the slag product to third-party consumers.

Effects on recycled ferrous metallurgical scrap: will allow you to reinvolve (recycling) it into production by means of remelting in the form of charge materials and reduce the cost of products.

Effects on dust from gas purification of ferrous metals: will allow you to receive revenue by changing the utilization to the sale of intermediate products.

It is very important for the metallurgical industry to adopt and implement global best practices to ensure longterm sustainability and improve the environment and standard of living in the regions where it operates.

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INNOVATIVE ENVIRONMENTALLY SAFE TITANIUM ALLOYS

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Currently, the development of materials that are safe for humans and the environment at all stages of their service is very relevant.

Titanium alloys are one of the main structural materials used in high-tech industries. In particular, VT6, Ti-6Al-4V, VT22, VT23, PT-3V, PT-3M, PT-1M, PT-7M, TL-3, TL-5, T-A4D3V and 4Al-3Mo-1V have depending on the composition and processing, the following mechanical properties: $E = 104...115$ GPa, $\sigma_{0.2} = 800...1100$ MPa, $\sigma_b = 850...1200$ MPa, $\delta = 4...16\%$. Alloys such as VT6 and nitinol Ti-Ni are used for the production of implants for various purposes. Most alloys contain V, Cr and Al as alloying elements, and Ti-Ni shape memory alloy contains Ni. All these elements, during production, operation and waste processing, enter liquid media or enter the environment in the form of particles and dust, from where they enter living organisms, incl. into the human body, causing very unpleasant diseases (including oncology, genetic damage and severe allergic reactions).

In the field of medical devices, where the requirements for safety and biocompatibility are initially very high, the development of new titanium alloys that do not contain Cr, Ni, V and Al has begun relatively recently. In new alloys, instead of dangerous alloying elements, it is proposed to use Nb, Ta, Mo, Zr, and sometimes Hf and Fe, which are not toxic to humans and do not release into the environment. In this regard, the IMET RAS has developed a number of alloys of the Ti-Nb-Ta, Ti-Nb-Ta-Zr and Ti-Nb-Mo systems, which have a set of mechanical properties quite comparable with traditional alloys: $\sigma_{0.2} = 220...810$ MPa, $\sigma_b = 620...1100$ MPa, $\delta = 5...14\%$. In particular, Ta and Zr improve the quality of passivation oxide layers and increase corrosion resistance compared to existing titanium alloys and pure Ti. These alloys, to a certain extent, are also nature-like materials, since their elastic modulus $E = 47...59$ GPa is close to the elastic modulus of natural materials, including human bones, and their high environmental safety allows the use of products made from them even inside the human body.

The research was supported by the Russian Science Foundation grant No. 24-13-00186.

THEORETICAL AND EXPERIMENTAL JUSTIFICATION OF ENERGY AND RESOURCE EFFICIENT TECHNOLOGY FOR ENHANCING OIL RECOVERY FROM HIGH WATER CUT WELLS WITH ORGANOHYBRID SEALING COMPOSITIONS

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In recent years various gelling compositions with adjustable properties have been widely used to conformance control in heterogeneous reservoirs. The main objective of this work was to justify and develop thermally stable organohybrid compositions (OHC) based on cross-linked polymer systems with viscoelastic and viscoplastic properties¹.

A three-component cross-linked polymer system (organohybrid composition) based on sodium silicate, chromium acetate and PAM is substantiated in this work. The composition was experimentally investigated for further application as a base composition in the development of a new organic-hybrid complex with the addition of organic dispersed phase. For qualitative analysis of the rheological and structural properties of OHC, a relaxometer was used to determine the lifetime of the polymer filament. Observation of the liquid filament formation macrotopology (formed only in cross-linked systems) allowed us to substantiate the methodology of cross-linking of polymer compositions according to the liquid filament lifetime. The results of OHC oscillation studies allowed quantifying viscoelastic and viscoplastic properties using Burgers' mechanical models.

Experimental research has justified an organo-hybrid system with predetermined properties and adjustable characteristics, capable of forming a strong barrier in the porous medium to hinder water filtration and prevent potential premature well water flooding processes.

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The research was carried out within the framework of the strategic project «New Technological Solutions in Fuel and Energy Complex» (Priority 2030). Research project «Development of acid stimulation technology with in-situ deviation to control the inflow profile in carbonate reservoirs».

RESOURCE SAVING, ENVIRONMENTAL SAFETY, TECHNOLOGICAL PROCESSES AND CLOSED CYCLE ECONOMICS OF METALLURGICAL WASTE PROCESSING IN ACID INDUCTION FURNACES IN THE SIBERIAN REGION

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Nowadays the processing of metallurgical wastes (magnetized iron and steel corollaries, slag dumps, processing of scrap of pallets and casters, etc.) is actual. These types of waste are characterized by high content of sulfur, phosphorus, unstable structure of products made of pig iron obtained from heterogeneous charge, increased gas content of metal. In the Siberian region at a number of enterprises the technology of pig iron smelting, including the processing of both pig iron and steel scrap and waste has been worked out. The developed technology was characterized by obtaining products with stable, predictable microstructure and specified mechanical properties of cast iron, and the required performance of products made of this cast iron. High performance of products is mainly due to the application in this technology modes of thermal-time processing and out-of-furnace melt by resonance-pulsing refining method. The methods of determining the optimal temperature modes of melt processing were based on the approaches developed in the school of Samarin A.M., as well as implemented jointly with the schools of Baum B.A., Kudrin V.A. and Elansky G.N.,. The peculiarity of this technology lies in the fact that at its realization the processes of oxidation of alloying elements are minimized, thus the problem of resource-saving is largely solved, the consumption of ferroalloys is significantly reduced and, as a consequence, the environmental safety of this technology is significantly increased. In fact, the economy of the closed cycle in the production of replaceable heat-resistant metallurgical equipment has been realized. This technology is realized at large metallurgical and tested at a number of machine-building enterprises in the Siberian region. A mathematical model was built to study the effect of phosphorus on iron properties.

SUPERCRITICAL TECHNOLOGIES FOR OBTAINING INNOVATIVE MATERIALS

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Supercritical technologies (SCT) are «green», resource- and energy-saving technologies that make it possible to obtain materials that are new in structure and properties. Around the world, SCT is used to produce aerogels, to produce printed circuit boards (PCB), to isolate valuable components from plant materials, to carry out unique chemical reactions, to produce nanocoatings, etc.¹. The most commonly used supercritical fluid is carbon dioxide, which is characterized by its non-flammability, non-toxicity, availability and low cost. The supercritical state of carbon dioxide corresponds to a pressure of 73.8 bar and a temperature of 31.1 °C. A polar co-solvent can be added to the carbon dioxide if necessary.

The following processes occurring in a supercritical fluid environment are distinguished: chemical reactions; adsorption or impregnation processes that make it possible to obtain composite or functional materials; foaming of polymers; drying processes to obtain porous materials; extraction processes; dispersion processes using the supercritical fluid antisolvent method, which make it possible to obtain substances in an amorphous nanosized state, as well as to apply thin films.

Using sol-gel technology and supercritical drying, aerogels can be produced. The production of aerogels is an extremely important task for the economy of the Russian Federation. Aerogels are materials with high porosity, low thermal conductivity, non-flammable, which are used for thermal insulation of pipes, containers with liquefied gas, automobile and rocket production, as a building material in the Arctic and Northern regions. The addition of nanotubes to aerogels leads to the appearance of electrically conductive properties, which can be used as sensors, sorbents, filters, and artificial controlled prostheses. Aerogels based on polysaccharides and proteins have found application in pharmaceuticals as a means of drug delivery, and in medicine, as medical devices and implants.

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ENGINEERING OF NATIONAL SOFTWARE FOR ENVIRONMENTAL SAFETY MANAGEMENT OF CHEMICAL INDUSTRIES

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In the conditions of existing chemical production, the technical condition of the equipment in use is the most important factor influencing the industrial and environmental safety of enterprises and adjacent areas. In practice, maintaining equipment in a reliable and operational condition is ensured by a maintenance and repair system¹, which involves the implementation of a large number of engineering and technical measures that make it possible to identify and prevent emergency scenarios in advance.

A large amount of equipment on the balance sheet of large enterprises makes the maintenance process very expensive both in cost and time. This is due to the presence of a large number of routine procedures associated with performing engineering and technical calculations, generating diagrams and reporting document. As a result, the possibility of timely and accurate identification of critical defects is reduced, and consequently, conditions for dangerous operation of enterprises arise.

Based on the above, with the participation of the authors, special software «ELPAS Enterprise» was developed². The use of this software allows not only to significantly reduce labor costs for equipment maintenance, but also to significantly improve its quality. This significantly reduces the risk of accidents occurring in production and, as a result, increases the industrial and environmental safety of chemical technology enterprises in general.

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WORKING-OFF OF THE ANAMMOX BIOTECHNOLOGY FOR NITROGEN REMOVAL FROM WASTEWATER USING LABORATORY BIOREACTORS

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Anammox, the process of microbial oxidation of nitrite with ammonium, resulting in dinitrogen production, is one of the newest and most efficient technologies for nitrogen removal from wastewater. Compared to traditional technologies, Anammox is characterized by lower capital and maintenance costs, higher rates of nitrogen removal, and low release of CO₂ and N₂O.

Bioreactors for investigation of the Anammox technology developed in the Federal Research Center of Biotechnology, Russian Academy of Sciences implemented two coupled processes in a single reactor in accordance with the simplified reaction: ammonium oxidation to nitrite ($NH_4^+ + O_2 \rightarrow NO_2^-$) and Anammox as such ($NH_4^+ + NO_2^- \rightarrow N_2$). The work was carried out using a set of laboratory bioreactors (Figure).



The process was shown to be stable in the presence of fluoride and sulfide; folate and formate were found to act as stress protectors. The effect of pH and oxygen concentration on the efficiency of the Anammox process was investigated. The technologically optimal loading materials, which are used for biomass attachment and higher efficiency of the process, were revealed.

Figure 1. Automated laboratory bioreactors using the Anammox technology.

The work was supported by the Russian Science Foundation (grant no. 21-64-00019) and by the Russian Federation Ministry of Science and Higher Education.

EVOLUTION OF TECHNOLOGIES: INCREASING INDUSTRIAL RESOURCE EFFICIENCY

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The implementation of the Strategy for Scientific and Technological Development of the Russian Federation includes development of a Directory (classifier) of Technologies and its subsequent integration into the information systems of the Ministry of Industry and Trade and the Ministry of Science and Higher Education. According to the authors' intention, the Directory should become an effective tool in determining priority areas of the Technological Development Policy and assessing the results of its exertion.

In the proposed Directory, an attempt is made to build a classification by forming a list (catalogue) of currently known technological solutions. Thus, an extensional approach is used and it is based exclusively on the external characteristics of the classification object, which is at the same time ambiguously defined.

The descriptive classification being formed, already at the initial stage of development, demonstrates the artificial unification into common taxa (section, class, etc.) of objects that are very different in nature, which leads to contradictions with the further widening of the Directory.

Such a classification – a list or catalogue – does not allow us to consider the essence of classification objects and expand the classification field. Thus, it will not have predictive power. The planned integration of the Directory other information systems will lead to migrating its contradictions into other systems and to forming problems in determining the priorities and directions of the national scientific and technological development. There are many examples describing various sectors, in particular – chemical and petrochemical industry.

We suggest adjusting approaches used for the Directory development as follows. It is necessary to take into account that technology is, first of all, an idea that cannot live and progress without its developers; the most important element of any technology is the human factor, knowledge, abilities, skills and cultural characteristics of people.

The hierarchical structure can be maintained, but the number of dimensions must be increased. At minimum three axes are needed: an axis of purpose (result) of technology (*why*), an axis of a technical solution (*what*) and an axis of the natural effect on the basis of which this solution can be obtained (*how*).

To determine the priorities and key directions of the country's scientific and technological development, the expertise of the Russian Academy of Sciences is crucial.

METHODOLOGICAL FEATURES OF DEVELOPING REGIONAL PROGRAMS ON CIRCULAR ECONOMY IN THE CONTEXT OF SUSTAINABLE DEVELOPMENT

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In 2021, the strategic initiative «Circular Economy» (ECC) was launched, and in 2022. A federal program of the same name was approved. In 2023 The Ministry of Natural Resources has sent out to the regions a working concept of the new federal law on EPC. The regions are invited, without waiting for the law, to begin developing regional programs for EPC. The process is complicated by the fact that the expert community has not yet agreed even on the basic concepts and principles of ELC in general, and the approach to drawing up regional ELC programs in particular. The researcher proposes a version of the basic terms, principles, mechanisms and tools for integration and transition to EZC in Russia, as well as what needs to be done to make it work in the regions, taking into account socio-ecological and economic risks, adjusting the territorial development strategy and verifying this development.

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SYSTEMATIZATION OF ECOLOGICAL CHARACTERISTICS OF POLYMER-INORGANIC COMPOSITES

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Presently due to the rapid increasing of application of polymer composite materials a need to diagnose of it characteristics as well as of engineering of new ecologically safe composites with improved exploitation characteristics is arising.

Systematization of ecological characteristics of polymer-inorganic composites, including chemical components, toxicological characteristics, radiation characteristics, noise and vibration measurements, possibilities of utilization etc. have been carried out by the author.

Methodology of point-rating ranging of ecological characteristics of polymer-inorganic composites has been suggested based on the estimation of the degree of potential ecological safety, impact to the humans etc. Summary rating point is determined by summing up the components of the point-rating score. It is proposed to evaluate it according to the following scale: 12-15 points – maximally high ecological hazard; 8-11 points – high ecological hazard; 4-7 points – average ecological hazard; 1-3 points – low ecological hazard. Thus, summary rating point has 4 gradations, taking into consideration the degree of negative ecological impact of polymer-inorganic composites to the humans and to environment.

Estimation of potential components of polymer-inorganic composites providing chemical pollution of air environment has been carried out by using of the suggested approaches to systematization and of the methodology of point-rating ranging.

Results of research are allowing us to determine exploitation possibilities of existing and new composites from the point of view of ecological safety, to develop new methods and technical solutions of reduction of negative impact of polymer-inorganic composites to the humans and to environment etc.

This research was done under financial support of the Ministry of higher education and science of Russian Federation within the framework of the state assignment to universities, topic FSSE-2023-0003.

THE ROLE OF MEMBRANE SEPARATION TECHNOLOGIES IN THE OIL AND GAS INDUSTRY IN THE CLOSED-CYCLE ECONOMY OF THE RUSSIAN FEDERATION

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Modern demands of industry and society require intensive development of separation and purification technologies, including gas media, especially in the context of modern industrial trends aimed at decarbonisation and formation of a closed cycle economy. Since the environment is under constant influence of various anthropogenic factors, the problem of intensification of productions in terms of their energy intensity and emission reduction is one of the key ones today.

Membrane and membrane-gas hydrate technologies for carbon dioxide capture from flue gases of CHPPs were proposed in the framework of the present study. In order to evaluate the performance of the separation process implemented in such apparatuses, modelling of technological schemes was performed using Aspen Custom Modeler blocks in the Aspen Plus technological environment. The results of mathematical modelling were verified using laboratory experimental setup. This integrated approach was aimed at identifying the key performance parameters of the separation processes realised in different variants of process flow diagrams, as well as at estimating the product cost by performing a technical and economic analysis of the calculated industrial scale process flow diagram.

As a result of such research, the prospectivity of application of membrane and membrane-gas hydrate technological schemes in the problems of carbon dioxide capture from flue gases of CHPPs has been demonstrated. The possibility of achieving the stated parameters of the process was experimentally established, namely, the content of carbon dioxide in the output stream ≥ 95 mol.%, in the purified gas stream < 2 mol.% with the degree of its recovery ≥ 90 %. The achieved results fully meet the modern requirements for such a process, and the technical and economic analysis demonstrates the economic efficiency of such technical solutions at the facilities of the energy infrastructure of the Russian Federation.

APPLICATIONS OF RADIATION TECHNOLOGY IN WASTEWATER TREATMENT FOR ENVIRONMENTAL POLLUTION

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Water availability is a major problem facing many regions around the world. To meet growing residential and agricultural needs, effective technologies have to be adopted to address microbial and chemical contaminants as part of water reuse programs. Unfortunately, conventional primary and secondary water treatments have been demonstrated to be insufficient for quantitative large-scale treatment. Therefore, to prevent increasing levels of organic pollutants entering environmental waters, additional treatment using radical-based, advanced oxidation processes (AOPs), are being considered to augment our traditional water treatments. In real-world waters most AOPs utilize the hydroxyl radical (HO \cdot), which can be created using a variety of techniques. Other AOPs that produce a mixture of reducing and oxidizing radicals include the irradiation of water via electron beams or γ -rays. Once these radicals are generated, the dissolved oxygen present will predominately react with the reducing hydrated electron or hydrogen atoms produced. The underlying hypothesis was that electron beam (EB) technology can breakdown the emerging contaminants of concern in water reclamation and reuse projects. In this study, EB irradiation was investigated as a method for removing persistent organic pollutants (dyes, pesticides...) from a synthetic water designed to simulate a treated wastewater. The degradation efficiency of these organic pollutants by electron beam or γ -ray radiolytic degradation is discussed in various conditions, such as different initial concentrations, irradiation doses and pH values. Besides, by-products of certain pollutants are identified using liquid chromatography mass spectrometry and radiolytic degradation mechanisms of these organic pollutants are illustrated. These results demonstrate that radiation technology is an effective method to degrade the organic contaminants, especially the persistent organic pollutants, hydroxyl radicals and hydrate electrons play significant roles in the radiolysis of organic pollutants. In addition, the limitations and the future trends of radiation technology applied in the environmental protection are also discussed.

ALGORITHMIZATION OF THE PROCESS OF MANAGING THE DEVELOPMENT AND USE OF RESOURCE AND ENERGY-SAVING TECHNOLOGIES IN INDUSTRIAL PRODUCTION

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One of the most important areas of the closed-loop economy is to reduce the material intensity of production (MI), including its energy intensity, through the use of resource-saving technologies, but this process must be managed during the development and use of technologies. At the same time, on the one hand, algorithmization of the management process itself is necessary to optimize the reduction of the ME level, and on the other hand, the developed technologies should ensure an increase in the level of stability of industrial production. Accordingly, the maximum possible, but economically justified increase in material output is required, that is, a decrease in the level of MI.

Criteria and indicators-indicators for identifying sustainability levels for energy-intensive industrial enterprises are defined in work¹, but with the appropriate transformation, they can also be used to develop individual technologies, including for processing industrial waste².

The procedure for selecting the best technology option that provides the maximum level of sustainability is justified during its development at the stages of research and development, including changes in the composition and prices of material and energy resources and equipment used, as well as when several types of products are released from waste.

An algorithm is proposed for reaching the economic limit of improving the existing production technology and, if necessary, replacing it with a new one.

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FRAGMENTARY MODEL OF THE ATOMIC STRUCTURE OF AMORPHOUS MATERIALS

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The fragmentary model developed by the authors is based on the assumption that the structure of amorphous substance is a mosaic of fragments of the crystalline phases structures. Their formation is possible for certain elemental composition. The experimental atomic radial distribution function (ARDF) with an ordering region of ~ 1 nm is analyzed using model ARDFs. The model ARDF is calculated by pair functions technique, using interatomic distances of the crystalline phase, according to the formulas proposed by the authors^{1,2}. The coincidence in the positions of the maxima of the model and experimental ARDFs in the entire ordering region indicates the presence in the amorphous substance of crystal nuclei of the phase, whose interatomic distances were used to construct the model. One should note that the model and experimental ARDFs never coincide in full, because the model is constructed from the interatomic distances of an ideal crystal, while the experimental one is obtained for a real sample including defects and interfaces of nanocrystallites. The study of various amorphous materials made it possible to establish the following.

Al-based amorphous metal alloys (AMAs) are heterogeneous. They consist of Al and intermetallic phases crystalline nuclei.

Spectrally pure heat-resistant SiO_2 contains crystalline nuclei of β -quartz and β -tridymite.

The structure of vitreous As_2S_3 and AgGeAsSe_3 is formed from structure fragments of layered and molecular crystalline phases. Vitreous As_2S_3 consists of the fragments of orpiment layers bound together by As_4S_5 molecules. AgGeAsSe_3 glass is formed by the fragments of layers attributed to GeAsSe and GeSe_2 compounds. Silver atoms replace germanium in selenium tetrahedra.

The resistance of zinc and cadmium phosphides and arsenides to high-power laser pulses was determined by analyzing the structure of thin amorphous condensates based on these substances.

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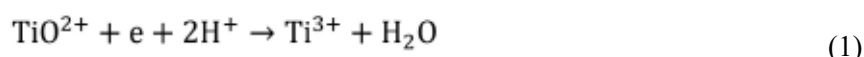
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CATALYTIC REDUCTION OF SELENATE IONS FROM WASTE SOLUTIONS AND WASTEWATER IN THE PRESENCE OF TITANIUM (IV) IONS

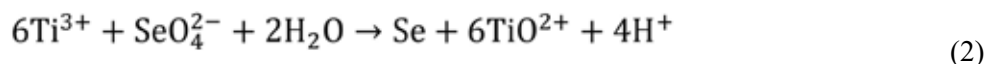
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During copper electrorefining, copper electrolyte sludges are formed, which are one of the primary sources for obtaining selenium. However, extracting selenium from such sludges presents a significant challenge, as during processing, selenium converts into a difficult-to-reduce form-selenate ion¹⁻³. To date, there are no satisfactory methods for the reduction of selenate ions⁴. Electrochemical reduction of selenate ions is also difficult because selenium oxoanions are negatively charged and repel from the cathode surface. In light of this, we explored the possibility of reducing the difficult-to-reduce selenate ions using the redox system Ti (IV)-Ti (III). In this process, titanium (IV) ions are reduced at the cathode (reaction 1):



The titanium (III) ions formed in the near-cathode space reduce the selenate ions, leading to the reaction (2), during which titanium (IV) ions are regenerated and subsequently discharged again at the cathode.



Thus, it has been demonstrated that the process is catalytic, and the selenate ion, often referred to as a «difficult-to-reduce» anion, can be reduced in the presence of titanium ions with variable valence, which act as charge carriers.

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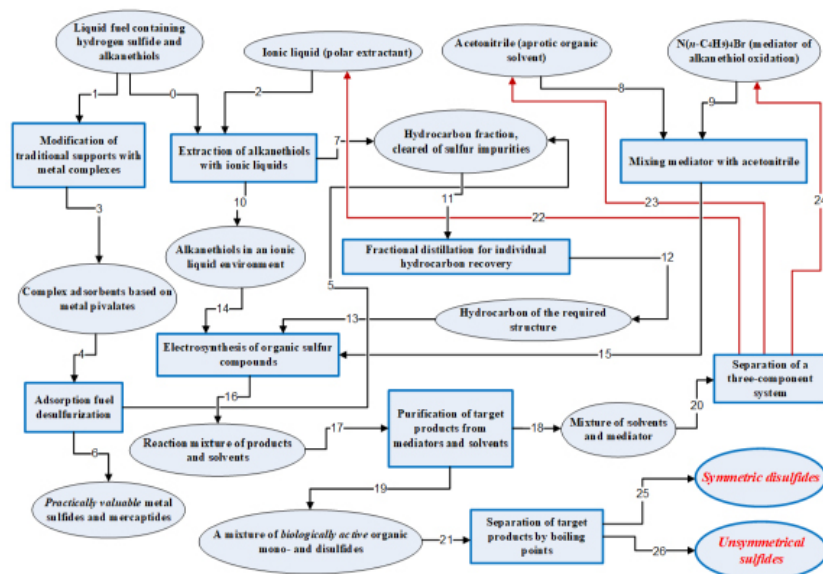
This work was supported by the Ministry of Education and Science of the Republic of Kazakhstan, project No. AP 23486503.

THE COMBINATION OF TECHNOLOGICAL PROCESSES FOR REMOVING SULFUR-CONTAINING WASTE FROM LIQUID FUELS AND THEIR CONVERSION INTO PRACTICALLY USEFUL ORGANIC SULFUR DERIVATIVES

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Global industrialization and over-dependence on non-renewable energy sources have led to an increase of waste and negative climate change. This problem requires a solution in the form of developing strategies for introducing a circular economy in the industrial sector, namely in the oil and gas processing industries. Sulfurous industrial waste has a significant impact on the environment and adversely affects human health. The combined technology proposed in the work, including the utilization of alkanethiols (hydrogen sulfide) with their subsequent processing into biologically active organic sulfur compounds, will ensure economic and environmental safety of the sale of toxic waste and effective desulfurization of liquid fuel. The logical-information model of the developed technology is presented below.



Adsorption purification of liquid fuel using pivalate zinc complexes promotes the conversion of sulfur-containing components into sulfides that have antibacterial and antifungal properties. The combination of two areas of using new generation solvents (ionic liquids) as extractants and electrically conductive media for electrosynthesis will significantly increase the efficiency of this technology.

This work was supported by the Russian Science Foundation, grant 23-13-00201.

ENGINEERING OF ENERGY AND RESOURCE EFFICIENT ENVIRONMENTALLY SAFE CHEMICAL-TECHNOLOGICAL SYSTEM FOR PROCESSING WASTE ORE RAW MATERIALS

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The problem of rational resource use, particularly in the context of energy-intensive industrial production, is of paramount importance. This encompasses conveyor roasting and sintering machines for processing ore raw materials, which are complex chemical-energy technological systems¹. The operational modes in the chemical-energy-technological system and the completeness of the interrelated chemical-energy-technological processes, namely drying, calcination, and sintering, which provide the final strength of the dense multilayer pellets moving mass on the conveyor, determine the quality of their thermal processing².

The digital engineering of energy and resource efficiency in chemical-energy technological systems entails the utilisation of contemporary intellectual methodologies and multiscale mathematical models of intricate chemical-energy technological processes. Additionally, it encompasses the adaptation of efficacious technical solutions and technological modes of functioning in chemical-energy technological systems to the periodically evolving properties of extracted ore raw materials. This represents a complex scientific and technical challenge³. The digitalization and engineering of firing modes of a moving dense multilayer mass of ore raw materials in a complex chemical-energy-technological system will permit an increase in the energy and resource efficiency of the chemical-energy-technological system, as a consequence of the intensification and optimization of heat and mass transfer processes.

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THE USE OF EXTRACTION-ELECTROLYSIS TECHNOLOGY TO ISOLATE VALUABLE COMPONENTS FROM INDUSTRIAL WASTE AND POOR ORES

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The extraction - electrolysis (SX-EW) method is used to extract copper from solutions¹. The purpose of the work is to isolate valuable components from industrial waste using the example of furnace dust from bronze production (RFA: Si - 48.742, Zn – 33.405 wt.%). After leaching in H₂SO₄, the copper cake was subjected to copper-ammonia MA and copper-chloride MX leaching. Copper extraction was carried out using extractants of various nature and concentration (Fig.1). The highest efficiency of the process was achieved in a solution of LIX 54 (50%).

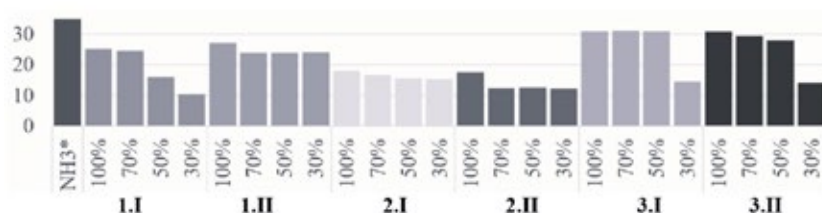


Figure 1. Efficiency of Cu²⁺ extraction from the etching solution into the extractant (I) and H₂SO₄ (II) in the range of extractant concentrations (%) 1. D2EGFC, 2. DX510A, 3. Lix54

The efficiency of extracting copper from the MX solution into the extractant and HCl is 35 and 32 g/l, respectively. The efficiency of extraction from the MA solution into the extractant and H₂SO₄ is 32 and 30 g/l. The results of copper electroextraction and the main process parameters are presented in the table:

Table 1. The main parameters of the copper extraction process from industrial waste

Leaching of sediment MA/MX (copper after sulfuric acid leaching)	T = 25 °C (MA solution); T = 50 °C (MX solution); τ = 40 min/
Extraction extraction of copper from MA/MX solutions	Extractant LIX54-1 50% in kerosene; τ = 20 min; Phase ratio 1:1
Electroextraction of copper from a sulfuric acid reextracting solution	i _k = 0,6 A/dm ² ; τ = 90 min.; CE= 65%

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RESOURCE SAVING IN PETROLEUM FUEL TECHNOLOGY DUE TO THE INVOLVATION OF OIL REFINING AND PETROCHEMICAL BY-PRODUCTS

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Oil-containing wastes and by-products are a source of valuable hydrocarbons, so finding new ways to extract them and use them as fuel components is an urgent resource-saving and environmental task.

The possibility of involving in low-viscosity marine fuel (LMF) by-product weighted fractions of primary and secondary oil refining has been established: 10-12% heavy diesel fraction, 47-60% straight-run middle distillate fraction, 2-10% heavy diesel fraction, 35% vacuum distillate, 10% bottoms of hydrogenation blocks, 4-10% light catalytic cracking gas oil¹.

A new way has been found to use a petrochemical by-product - LMPE (low molecular weight polyethylene) as a component of LMF. Its effectiveness on the pour point of LMF containing up to 75% heavy fractions has been proven. It is shown that the use of low-molecular-weight polyethylene will eliminate the use of expensive depressant additives from the preparation of LMF².

A new component of motor gasoline has been obtained based on by-products - low-boiling distillates of the fractionation of the reaction mixture of the synthesis of butyl alcohols and the hexane fraction of the isomerization process of light straight-run gasoline³.

Fractions have been isolated from commercial waste after dehydration, making it possible to obtain new components of motor gasoline and low-viscosity marine fuel. A simple, accessible technological scheme has been developed, based on the processes of mixing and separation⁴.

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THE MAIN DIRECTIONS OF THE RECYCLING OF THE WAE LZ SLAG FROM THE ELECTRIC ARC FURNACE DUST PROCESSING

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The Waelz process is the main technology for the electric arc furnace dust processing to recover zinc in the Waelz oxide, which is used as a raw material for zinc plants. A by-product of the dust processing is the Waelz slag, which contains up to 45% iron, up to 20% carbon and residual zinc. Currently, the main part of the generated Waelz slag is not utilized and dumped in landfills. The Waelz slag production is about 600–800 kg per ton of the processed dust that leads to a significant waste accumulation.

In the world there are studies of the possibility of the Waelz slag application in road construction, in the production of concrete, cement, bricks and other building materials. For ferrous metallurgy, the extraction of iron from it is of interest. The Waelz slag utilization in ferrous metallurgy is limited mainly due to a high residual content of zinc, which is a harmful impurity for the blast-furnace smelting process. Zinc content in the Waelz slag can vary in the range of 0.2–6%.

In IMET RAS studies is carried out to develop various technological flowsheets for the processing of the Waelz slag with the extraction of valuable elements into separate products using the stages of magnetic separation, flotation, reduction smelting, reduction roasting, and leaching. The flowsheets being developed have shown the possibility of obtaining products from the Waelz slag suitable for further use.

The utilization of the Waelz slag will increase the technical and cost efficiency of the electric arc furnace dust processing and will contribute for reducing the environmental impact in the places of its formation and storage.

This research was funded by Russian Science Foundation, grant number 24-23-00507.

THE CONCEPT OF WASTE-FREE PROCESSING OF MINERAL AND MAN-MADE RAW MATERIALS OF THE NORTHERN URALS IN AN INTEGRATED MANNER

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The closed-cycle economy provides for the creation of waste-free technological processes in all areas of material production. At the same time, additional natural material resources should be used in the minimum necessary amount. Since the late 70s of the last century, domestic scientists have been interested in the possibility of complex processing of red sludge from alumina plants of the USSR in order to solve the problem of cost-effective extraction of scandium. Based on the results of numerous research works, a waste-free and environmentally friendly concept for the disposal of red sludge from the Bogoslovsky Aluminum Plant was developed.

This concept is based on the following principles:

1. The main and auxiliary raw materials of production processes are exclusively industrial waste (except for specific reagents);
2. Complete exclusion of storage for long-term storage on the earth's surface of any solid or liquid waste materials;
3. The location of the enterprise must have the necessary energy and water resources;
4. It is necessary to have a stable market for commercial products.

Taking into account the peculiarities of the chemical and mineral composition of red slurries, the presence of nearby calcium-containing rock dumps of the Severouralsky bauxite mine and quartz-glaucanite sand of overburden rocks of the Tyninsky deposit of carbonate manganese ores, it becomes possible to create a mining and metallurgical complex for the disposal of these wastes to obtain a wide range of highly liquid products (including up to 120 tons of scandium oxide annually).

CLOSED CYCLE TOLUENE PROCESSING IN CHEMICAL-TECHNOLOGICAL PROCESSES OF LOW-TONNAGE CHEMISTRY

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The data are presented on the processes of deep processing of toluene - one of the least demanded as a raw material source of hydrocarbon contained in the light fraction of aromatic hydrocarbons (benzene, toluene, xylenes, etc.). Toluene processing is based on combining physical, chemical and technological processes into a single complex, which was named «Production Complex for Deep Processing of Toluene» (Figure 1)¹.

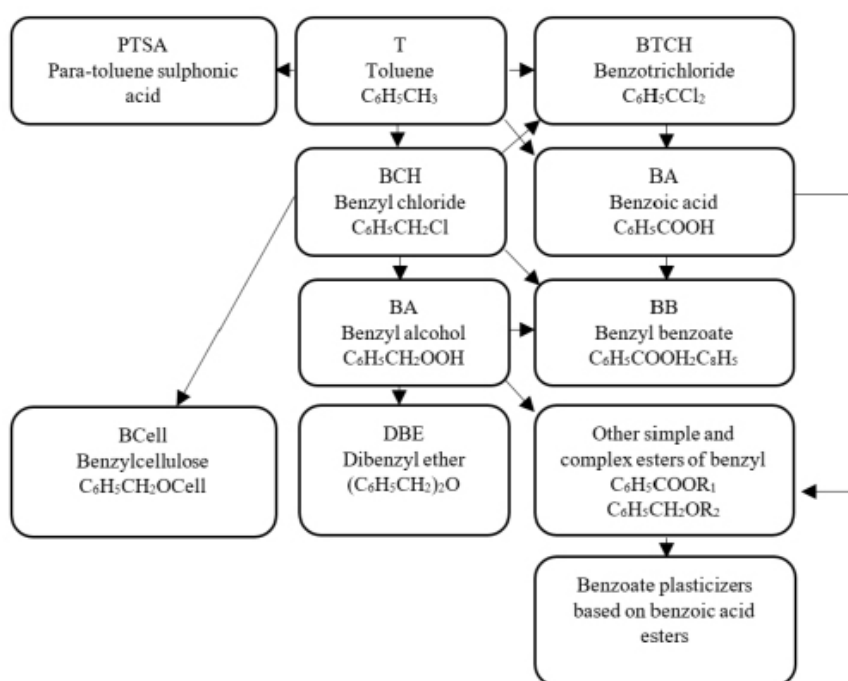


Figure 1. Production complex of deep toluene processing

The data on the specifics of chemical-technological processes included in the «Production complex of deep processing of toluene» are given.

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POTENTIAL OF THE PRODUCTION OF L-LACTIC ACID AND ENVIRONMENTALLY SAFE CHEMICAL PRODUCTS BASED ON IT

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The results of complex fundamental and applied research in the field of environmentally, economically and technologically efficient processing of renewable sugar-containing raw materials into in demand chemical products through the stage of high-capacity fermentation of raw materials into optically pure L-lactic acid (L-LA)¹⁻³, using a natural strain of lactic acid bacteria, are presented. Fermentation is carried out without removing biomass from the biosynthesis zone using the fed batch-withdrawal method in a membrane bioreactor, which provides specific capacity an order of magnitude higher than traditional methods of batch-mode fermentation. This makes it possible to increase the yield of L-LA up to 90–95% with an optical purity of at least 99%, reducing the content of biosynthesis by-products in the resulting post-fermentation medium to minimal level and reducing the cost of growth factors by 3 times, which makes L-LA acceptable at cost as an intermediate product for further chemical processing.

The subsequent isolation of L-LA from the culture liquid is carried out throw synthesis of L-butyl lactate (L-BL) without the formation of waste in the form of calcium sulfate (in traditional production, about 1 ton of gypsum is formed per 1 ton of L-LA). The resulting L-BL is not only an environmentally friendly solvent, but also an intermediate product for the production of other alkyl esters of lactic acid, high-purity L-LA, 1,2-propylene glycol, and biodegradable plastic – polylactide. Polylactide is not only biodegradable polymer, but it can also be thermocatalytically processed into the initial monomer – lactide, that can serve as the basis for the closed life cycle of such a polymer.

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The research was carried out within the state assignment of Ministry of Science and Higher Education of the Russian Federation, project FSSM-2023-0004.

EXPERIMENTAL AND THEORETICAL ANALYSIS OF THE PROPERTIES OF AN ENVIRONMENTALLY FRIENDLY SORBENT BASED ON THE PROCESSING OF AGRICULTURAL WASTE

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The composition and characteristics of waste of plant origin vary widely and depend on the plant variety, growing conditions, climatic conditions and other factors. The purpose of this study was to develop a program for predictive analysis of sorption properties of materials based on plant waste from agriculture for the purification of heavy metal pollution.

The use of machine learning methods makes it possible to find patterns between heat treatment modes (time, temperature) and sorption properties of materials, as well as to analyze the use of sorption materials under various factors for further predictive assessment of the influence of factors such as the concentration of pollutants, interfering impurities, and the type of pollutant on the sorption properties of materials. The obtained results of the influence of factors are shown in Figure 1.

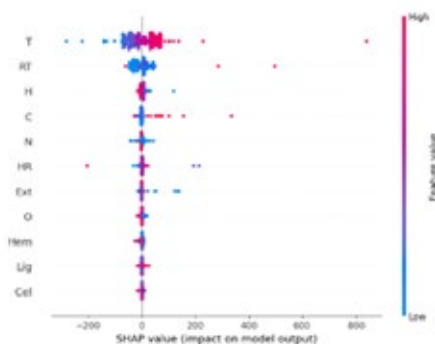


Figure 1. The influence of factors on the specific surface of the sorbent

The XGBoost regression allowed us to build a predictive model of sorption capacity with a determination coefficient of 84%. An example of a forward-looking estimate is shown in Table 1.

Observation	Actual value	Predictive value
0	3.8	3.5
1	6.2	6.5

Table 1. Predictive assessment of sorption capacity

The work was carried out with the financial support of Blue Sky Research, the BSR-2023-9 project.

PERSISTENT ORGANIC POLLUTANTS: PROBLEMS OF ENVIRONMENTAL POLLUTION AND TECHNOLOGICAL METHODS OF NEUTRALIZATION

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The problem of destruction of persistent organic pollutants (POPs) is very relevant today. The issue of environmental pollution by POPs has been identified by the United Nations as a priority environmental problem. In 2001, the international Stockholm Convention on POPs was signed. In 2011, Russia ratified this convention by federal law dated June 27, 2011 No. 164-FZ. The Convention aims not only to prohibit the production and use of CO₃, but also to destroy existing stocks of these substances by 2028. The neutralization of POPs is an important international problem, since there is still no generally accepted, safe technology.

The work carried out a comprehensive systematic study (calculation, experiment) of a two-stage method for the destruction of POPs by example of polychlorinated biphenyls - PCBs, using chemical functionalization and subsequent safe thermal destruction of new PCB derivatives:



Scheme. Stages of an integrated method for processing chlorine-containing industrial waste.

The work obtained fundamental scientific knowledge about the thermodynamic properties of chlorine-containing POPs, developed a method for predicting their behavior in thermal and chemical processes, and substantiated possible approaches to solving the problem of safe destruction of chlorine-containing industrial waste.

ZERO LIQUID DISCHARGE AT THE NON-FERROUS METALLURGY ENTERPRISE

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One of the main concepts being introduced in the field of water use in industrial enterprises and being an integral part of the transition to a circular economy is so-called ZLD – zero liquid discharge, which involves the complete reuse of wastewater after the necessary treatment without its discharge into water bodies.

At Uralelektromed JSC the transition to ZLD was completed in 2023 through the introduction of technology based on baromembrane methods of water purification: ultrafiltration and reverse osmosis. It combined two different areas of water use at the enterprise: wastewater treatment and the preparation of deionized water by involving the former in the production of the latter. The combined wastewater processing scheme can be presented as follows (the stages of the implemented technology are highlighted in italics): *wastewater* → alkali treatment → coagulation and flocculation → clarification → filtration → *pH adjustment* → *ultrafiltration* → *1st stage of reverse osmosis* → *2nd stage of reverse osmosis* (processing of concentrates from the 1st stage of reverse osmosis) → *decarbonization of permeate* → carbon adsorption → ion exchange on a cation exchanger → ion exchange on a weakly basic anion exchanger → ion exchange on a strong base anion exchanger → pH adjustment → *deionized water*. Taking into account the total dissolved solids content (TDS) in the initial wastewater of about 1 g/L, an overall permeate yield of 94% from reverse osmosis plants was achieved. Moreover, the TDS of permeate does not exceed 25 mg/L, which is from 5 to 6 times less than in raw water used for production of deionized water. The final concentrate is processed in the existing evaporation plant to salts consisting mainly of sodium sulfate and sodium chloride.

As a result of the transition to ZLD an environmental and economic effect of 175 rubles (1.7 € or 1.9 \$) per 1 m³ of recycled wastewater was achieved at Uralelektromed JSC. The implementation of the project led to the following positive effects: 1) elimination of wastewater discharges and damage to water bodies; 2) reduction of raw water consumption; 3) improvement of the technical and economic indicators of the technology for producing deionized water due to the use of cleaner raw material and, as a result, reduction of costs for regenerating reagents, energy resources and processing of eluates; 4) elimination of the need for regular elaboration and updating of standards for permissible discharges, constant monitoring of water quality at the water outlet and in water bodies.

MATERIALS WITH MOSAIC SURFACE FOR OIL SPILL RESPONSE

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Environmental pollution during oil production and transportation is a significant problem, especially in the Far North, where the main production facilities are now moving.

This project is focused on obtaining coatings with a hydrophilic-hydrophobic surface that switches when changing pH. Its practical significance lies in the production of regenerated materials for the elimination of emergency oil spills. In the activated state, when the surface of the material is hydrophobic, it can be used as a sorbent to collect oil from the surface of water. Then, when the pH changes, the surface of the material becomes hydrophilic and the oil is easily removed from its surface and can be collected and used as a commercial product. Moreover, the thickness of the mosaic coating is so small that it will not negatively affect the cost. Material with a mosaic surface can be used many times without the need for disposal or disposal.

Mosaic coatings have been prepared by depositing cellulose microgels with an average diameter of 150–450 nm on glass fiber or fabric¹. For all microgels obtained in this work, a complex of structural studies was carried out, including ¹H NMR, IR, ESR, dynamic light scattering (DLS) and scanning probe microscopy. These studies revealed the domain structure of the resulting microgels. Domains measuring 20–30 nm, consisting of denser material, are connected by a softer gel into a single structure measuring 200–300 nm². Chemical modification of individual domains made it possible to obtain a mosaic structure, which was preserved during the deposition of microgel particles on the fiber and formation of the coating. Currently, a comprehensive study of the properties of the resulting materials is being carried out.

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PROCESSING ZINC-CONTAINING DUSTS OF FERROUS METALLURGY WITH INDUSTRIAL PRODUCTS OF THE NON-FERROUS INDUSTRY – A PATH TO RESOURCE SAVING

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Increase of zinc production with the use of substandard raw materials in the processing is an important task. Zinc content in the dusts of blast furnaces reaches 5-7%, and it is at the level of 10-25% in the dusts of electric steelmaking furnaces. The problem of processing of zinc-containing dusts is actual both for ferrous and non-ferrous metallurgy¹. Pyrometallurgical methods of zinc extraction are different in the technological process parameters, types of reducing agent to be used, fuel and structural design of equipment². Effect of a binder at briquetting of charge, type and consumption of reducing agent, grinding fineness of charge components on the zinc recovery process from oxidized zinc ore with the addition of laid gas purification dust of blast furnace smelting has been studied³. Bentonite, burnt lime and molasses were tested as a binder for briquetting the charge. It was found that the optimal binder is molasses in the amount of 4.5-5.0 wt. % of the charge mass. It is shown that the residual zinc content in the roasting product with the use of special coke is 1.9 times less than with the use of anthracite and 3.3 times less than with the use of metallurgical coke. Carbon consumption at zinc recovery from oxidized ore with dust addition is reduced by 22-24%. It is established that charge grinding to the class +0.071-0.04 microns reduces the zinc sublimation degree. High recovery efficiency is achieved at the following composition of charge, wt. %: zinc ore 53,8; gas purification dust of blast furnace smelting of 26,9; special coke 21,0; molasses 5,3.

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SOLID MUNICIPAL WASTE IN A CYCLE ECONOMY

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Municipal solid waste (MSW) is a complex fraction in waste management.

The purpose of the legislation of the Russian Federation is to involve waste in economic turnover. There are several ways to handle MSW: preparation of RDF fuel for the cement industry and metallurgy, recycling of glass, tires, plastics. The object of research: solid municipal waste management, waste sorting complexes, extraction of glass, paper, metals and plastics at waste sorting complexes, preparation of RDF fuel. Initially, the morphological composition of municipal solid waste is being studied, the composition and calorie content of RDF is being determined, the market for the use of secondary components, RDF, the calculation of economic indicators of waste involvement at potential enterprises, including the cost of equipment at waste sorting complexes, the use of RDF fuels in the cement industry and metallurgy.

The legislation of the Russian Federation defines the need for the preparation and use of solid fuels obtained from waste, the possibility of using which is fixed in the Information and Technical Handbook on the best available technologies ITS 6-2015 «Cement Production», approved by Order of the Federal Agency for Technical Regulation and Metrology dated 12/15/2015 No. 1576. This task was successfully implemented by the HeidelbergCement group (Heidelberg Cement), in the village of Novogurovsky, Tula region, and Sterlitamak, the LafargeHolcim group (Lafarge Cement), the Shchurovsk sites of the Moscow region and the village. Ferzikovo, Kaluga region. Currently, TSEMROS (Eurocement Holding) is working on this issue at the Slantsy site in the Leningrad region and in the village of Podgorensky in the Voronezh region. Currently, work is underway to add cord textile waste generated during tire recycling to the RDF.

Thus, the disposal of MSW is minimized, only 20-30% of the tailings will go to landfills, this is one of the goals of the Cycle Economy.

CONTRIBUTION OF HIGH MOLECULAR NAPHTHENIC ACIDS TO THE FORMATION OF DEPOSITS IN OILFIELD EQUIPMENT

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High molecular weight naphthenic acids (ARN-acids) are a class of tetrabasic organic acids with Mw 1200-1400. It was suggested that their source in oil is microorganisms of the *Archaea* domain found in oil-bearing formations¹. Despite the low content of ARN-acids in crude oil (~ 20 ppm)², they form a significant amount of insoluble naphthenates, mainly with alkaline earth metal cations. A characteristic feature of such deposits is thermal instability: with increasing of temperature decarboxylation occurs with a sharp increase in volume, with the formation of a hard plastic-like mass upon solidification, which significantly complicates the operation of commercial oil treatment systems. The purpose of the work is to develop a method for isolating, identifying and quantifying ARN-acids in crude oil and industrial deposits.

To extract ARN-acids mixtures of organic solvents were used to convert the analyte into acid or salt form. To stabilize ARN-acids esterification was applied. Identification was carried out using IR-spectroscopy, py-GC/MS, and HPLC/MS. Pyrograms of purified ARN-acids are dominated by signals from branched aliphatic and alicyclic hydrocarbons. ESI-MS in negative ion detection mode shows the presence of singly and doubly charged ions with m/z 1230 and 615, which are attributed to an acid with the empirical formula $C_{80}H_{142}O_8$. Methyl esters of ARN-acids give a mass spectrometric signal in the positive ion detection mode (m/z region 1306-1314). To analyze them by HPLC-MS, tetramethylsilyl phase was used; a mixture of methanol and isopropanol was used as an eluent. Quantity of ARN-acid in industrial equipment deposit ranged from 1.6 to 21% depending on the sampling unit.

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This work was carried out according to state order No FWFN-2022-0002 with use of equipment of Far Eastern Center of Structural Research

STUDIES OF THE PROCESS OF SMELTING CAST IRON IN A ROTARY-INCLINED FURNACE FROM IRON-CONTAINING WASTE FROM METALLURGICAL PRODUCTION

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During the development of the technology for processing iron-containing waste from metallurgical production, laboratory studies and industrial experiments were carried out in a rotary-inclined furnace to extract iron from briquetted iron-containing waste and carbon.

In the course of laboratory studies, optimal compositions of briquettes with the addition of mineral components that ensure the fusibility of the resulting slag were developed, temperature parameters of the process and factors affecting the process of iron reduction and carburization were determined.

In the course of industrial experiments in a rotary-inclined furnace, modes of solid-phase reduction of iron, modes of transition from a solid-phase process to a liquid-phase process and modes of a liquid-phase process were worked out, allowing for sufficiently efficient separation of slag and metal.

The work carried out made it possible to determine the main technological parameters of reducing smelting and the technical characteristics of a rotary-inclined furnace for smelting cast iron from iron-containing waste from metallurgical production.

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DEVELOPMENT OF EFFECTIVE ENVIRONMENTALLY FRIENDLY ANTIFOULING COATINGS

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The problem of preventing marine biofouling has concerned humanity for hundreds of years, since the birth of navigation. One of the most common methods of protection is antifouling coatings. However, to date there are no ideal ways to protect against this natural phenomenon. The report will discuss current global trends in the development of antifouling coatings. In recent years, special attention has been paid to protecting the environment, in particular, to the selection of environmentally friendly biocidal additives that give coatings antifouling properties¹. The results of many years of full-scale bench tests carried out at the White Sea biological station «Kartesh», located in Karelia, in Chupa Bay of the Kandalaksha Gulf of the White Sea, 30 km from the Arctic Circle¹⁻³ will be presented. The influence of the following factors on the degree of fouling will be discussed: the nature of the binders (vinyl oligomers, epoxy resin, acrylates and their adducts); surface conditions, leaching and self-polishing effects; the nature of biocidal additives: micro- and nanoparticles of oxides, atranes, complex metal compounds, incl. on a porous carrier a natural antifoulant (extract of the sea sponge *Halichondria panicea*); fouling species. Prospective directions of research will be substantiated.

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RECYCLING OF SPENT LITHIUM-ION BATTERIES FOR HOUSEHOLD APPLIANCES AND ELECTRONIC DEVICES

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The problem of recycling spent lithium-ion batteries (LIB), which belong to the waste of hazard class 2, is becoming more acute. In Russia, currently spent LIBs are mainly represented by batteries from household appliances and electronic devices. According to the concept of electric transport development in the Russian Federation, by 2030 about 220 thousand electric cars per year will be produced and the total number of electric vehicles will exceed 1,4 million¹. By this time, the mass of spent LIA in the world will be more than 600 kt/year with processing of about 45%². In 2022, about 44 companies in Canada and the United States and 47 companies in Europe were recycling lithium batteries or were planning to recycle them; LIB recycling in Russia is now carried out in small quantities.

Among the used methods of processing LIB, the most effective is hydrometallurgical, which allows to obtain fractions of stainless steel, plastic, graphite, solvents, individual compounds of elements in demand by the market as well as precursors of cathode materials.

As a result of mechanical processing of a mixture from various spent household appliances and electronic devices, a concentrate (black mass) and the cathode mass was obtained. The physicochemical characteristics were studied, options for opening and further processing of the black mass and cathode material were considered.

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The financial support for this study was obtained from JSC «Science and innovations», project EOTII- MT- 492.

CELLS INCUBATION IN A HYDROGEL VOLUME WITH AN ARTIFICIAL MICROCHANNELS NETWORK FOR OXYGEN SUPPLY AND CARBON DIOXIDE REMOVAL

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In 3D bioprinting technology used for organs and tissues artificial cultivation in regenerative medicine, hydrogels are promising materials for cells incubation. The main their practical use problem is insufficient mass conductivity which makes difficult to supply nutrition and oxygen to cells as well as to remove metabolic products from the hydrogel matrix volume. One of the solving ways is to create artificial microchannels networks in the gel similar to the circulatory system, i.e. the nature-like technology implementation for organizing cells respiration¹.

The mass transfer processes description in structured media with microorganisms is a difficult problem². There are several process stages: the initially oxygen diffusion and absorption to ensure the seeded cells vital activity, the their active division period with an increase in oxygen consumption, a further sharp increase in the metabolic products release with the possible carbon dioxide bubbles formation, cells population stabilization with the stationary state establishment.

A mathematical model describing the growth of a cells population encapsulated in a hydrogel under oxygen supply conditions to the hydrogel matrix volume and carbon dioxide removal from it through artificial microchannels networks is proposed. The presence of a stationary states plurality in such systems is shown, and their stability is analyzed. Estimates of the cells concentration in the population achieved as a their incubation result in a hydrogel depending on the relative microchannels specific surface area for oxygen supply and carbon dioxide removal were obtained.

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CHEMICAL, TECHNOLOGICAL AND ENVIRONMENTAL ASPECTS OF THE PRODUCTION OF ULTRAFINE POLYTETRAFLUOROETHYLENE FROM FLUOROPLAST-4 WASTE AND ENERGY- RESOURCE SAVING MATERIALS BASED ON IT

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The unique technology for the production of ultrafine low molecular weight polytetrafluoroethylene¹ (UNPTFE) from the waste of fluoroplast-4 (Fig.1) is created on the basis of a thermogradient method for the synthesis of high-er fluorides of elements². The manufacturing of PTFE-based products is es-tablished and the in-depth study of the new polymer and its derivatives is underway.³

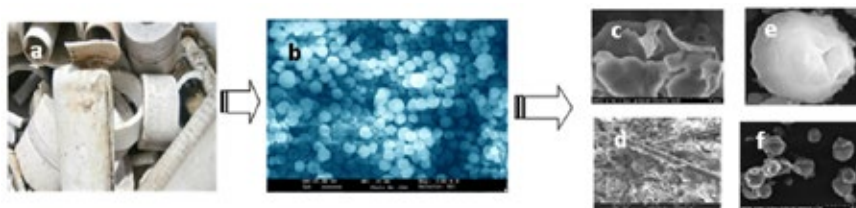


Figure 1. Fluoroplast-4 waste (a). SEM: UNPTFE (b), UNPTFE nanofilms (c), microtubules from UNPTFE nanofilms (d), UNPTFE particle ~ 1 μm (e), particles of high-temperature frac-tions of UNPTFE (f).

Uncontrolled waste disposal of fluoroplast-4 can lead to the formation of highly toxic perfluoroisobutene and fluorophosgene, that's why the pro-posed technology not only solves an environmental problem, but also allows to create a new material that forms an antifriction, anti-wear, chemically re-sistant, biologically inert, hydrophobic and high-temperature coating on any surface. Energy- resource saving is achieved by significantly reducing the wear of parts and improving the performance of engines, transmissions and other mechanisms, significantly reducing the consumption of fuel and lubri-cating oils. PTFE frictionally applied to the surface or added into the com-position of marine paints significantly prolongs the service life of antifouling coatings of the underwater part of ships and protects the surface part from icing⁴.

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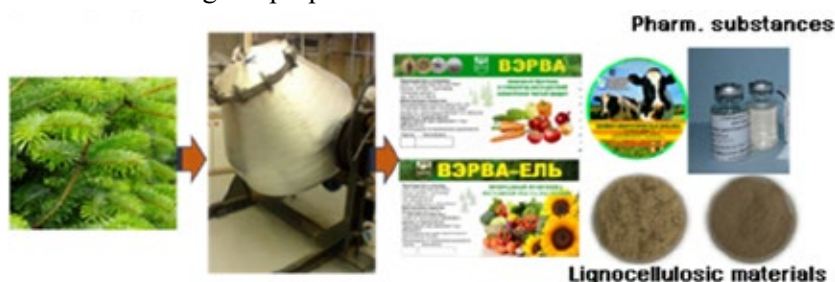
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INNOVATIVE TECHNOLOGY OF COMPLEX PROCESSING OF CONIFEROUS WOOD GREENS INTO BIOLOGICALLY ACTIVE SUBSTANCES AND MATERIALS APPLICABLE IN PLANT CULTIVATION, ANIMAL HUSBANDRY AND MEDICINE

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The use of natural compounds from plant sources as raw materials for producing medicinal preparation, plant and animal protection products is a global trend in recent years. We have developed and implemented a technology for complex processing of coniferous woody greens using emulsion extraction with aqueous solutions of alkalis. There are no toxic or environmentally hazardous reagents in the technological cycle. At the same time, lipids, terpenoids and a lignocarbhydrate complex are isolated in one stream, and preparations for crop production, animal husbandry and medicine can be obtained on their basis. As a result, highly effective protective agents and plant growth stimulants Verva and Verva-spruse as well as a feed additive Verva were introduced into production. Low molecular extractive components of plant raw material were used as drug platforms for the treatment and prevention of socially significant diseases. Schroth is a by-product of wood green extraction was used as a raw material for producing lignocelluloses materials and cellulose esters of a wide range of purposes.



The completed work is aimed at solving a fundamental scientific problem in the field of complex study of advanced technically valuable plant species that are part of Russia's natural resources with the aim of creating bio-preparations in demand for the development of highly productive and environmentally friendly agro-forestry and medicine

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MEMBRANES FOR OIL REFINING AND PETROCHEMISTRY

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Around 10-15% of global energy consumption is devoted to various separation processes, underscoring the importance of developing more advanced and energy-efficient separation and purification approaches. Presently, approximately 100 million barrels of crude oil are processed daily at refineries worldwide, with atmospheric and vacuum distillation being the predominant separation methods employed. Concurrently, membrane separation processes are widely regarded as a promising alternative to distillation, owing to the absence of phase transitions. Notably, reverse osmosis has emerged as the primary industrial technique for seawater desalination globally. In TIPS RAS, we have developed polyacrylonitrile-based filtration membranes enable the direct oil fractionation, facilitating the separation of compounds with molecular weights below 600 g/mol while achieving near-complete asphaltene removal (99.9%).

Mitigating the carbon footprint in petrochemical and refining processes can be realized through carbon dioxide capture, implementation of hydrogen production methods without carbon dioxide emissions (e.g., thermal decomposition of methane), and the recovery and recycling of valuable components from various gas streams. This presentation will showcase the results of several gas separation membranes developed for diverse applications, including carbon dioxide, hydrogen, and C_{2+} hydrocarbon separation. In the fabrication of these membranes, commercially available polymeric materials, including those produced within the Russian Federation, were employed as membrane materials.

This work was funded by the State Program of TIPS RAS.

DEVELOPMENT AND IMPLEMENTATION OF «GREEN» INHIBITORS OF GAS HYDRATE FORMATION AT OIL FIELDS

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During the development and operation of oil and gas fields the formation of gas hydrates in oil production becomes a serious problem both for flow and pressure control in the wellbore and for the transport of hydrocarbon raw materials. To prevent the formation of gas hydrates, thermodynamic inhibitors of gas hydrate formation (IH) such as methanol, glycols, salt solutions that change the conditions of hydrate formation and kinetic IG - poly(N-vinylpyrrolidone) (PVP), poly(N-vinylcaprolactam) (PVCap) that increase the induction period and reduce the rate of hydrate formation are used. The main purpose of this work is to develop promising «green» oilfield reagents based on polysaccharides.

It was found that polysaccharides change the conditions of gas hydrate formation, exhibiting the properties of thermodynamic and kinetic IH with an efficiency superior to methanol by tens of times depending on the selected polysaccharide¹. It is assumed that the inhibition is due to electrostatic interaction of ionised carboxyl groups with water molecules, and as a result of hydrogen bond formation with oxygen atoms and OH-groups of D-glucose fragments. The conducted studies are the basis for the developed line of new inhibitors of gas hydrate formation to prevent the formation of gas hydrate deposits in oilfield systems². The preparative forms of IHs have undergone pilot field tests at wells of oil fields of Western Siberia, effective dosages of IHs have been determined, recommendations for their industrial application have been developed.

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FORECASTING THE CHEMICAL-TECHNOLOGICAL PROCESS OF IN-SITU WATER SHUT-OFF IN OIL-PRODUCING WELLS THROUGH HYDRODYNAMIC MODELING

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Technological processes of water shut-off operations in producing wells are a complicated but extremely urgent task. Emulsion systems, hydrogel compositions, foam systems, resins, etc. can be used for water shut-off and inflow profile control. To improve the efficiency of such operations, the application of mathematical modelling is widespread.

Earlier the author developed a model of technological process of water shut-off operations¹. The model differs from the previously considered ones by predicting not only the placement of water-sealing compositions in the bottom-hole zone and changes in the filtration characteristics of the rock, but also the dynamics of oil and water flow rates during subsequent well operation, due to changes in the direction of filtration flows in the near-wellbore zone of the formation and degradation of the sealing screen. Further improvement of the model is connected with modelling of rheological, structural and mechanical properties of water-sealing compositions and their behaviour in the porous medium and fractures of the productive reservoir, which allows not only to increase the success of predicting the starting parameters of the well for a given operation mode, but also the dynamics of its subsequent watering. The model takes into account the results of rheological and filtration studies of the compositions, namely, the dependence of the residual resistance factor on the pressure gradient in the bottomhole formation zone, shear stress on the shear rate, as well as the value of the elastic modulus of the composition.

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The research was carried out within the framework of the strategic project «New Technological Solutions in Fuel and Energy Complex». Research project «Development of acid stimulation technology with in-situ deviation to control the inflow profile in carbonate reservoirs»

PHYSICO-CHEMICAL ENGINEERING OF DURABLE ENVIRONMENTALLY FRIENDLY CERAMIC BUILDING MATERIALS

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It was proved for the first time that the process of destruction of building ceramics proceeds according to the type of chemomechanical process¹. Consideration of the process of destruction of the material of ceramic products by the type of chemomechanical process allowed us to obtain a quantitative assessment of the durability of the material of building ceramics.

For the first time, the authors developed and proved theoretically, on the basis of thermodynamic calculations, and experimentally, on the basis of laboratory studies, the chemistry of the destruction process of building ceramics material. It is proved that the destruction of the building ceramics material is determined by an internal two-stage chemical process that takes place in the material constantly when it is moistened². Based on experimental studies, the main kinetic characteristics of each stage of the destruction process have been determined. A phenomenological equation has been obtained to determine the durability of the building ceramics material.

The study of the durability of building ceramics is primarily aimed at creating scientifically sound proposals for the modernization and technological improvement of the production process of building ceramics with a given period of preservation of physical properties, including strength. The production of products with a given durability will reduce energy consumption not only at the material production stage, but also at the disposal stage, which ultimately will help reduce the technological burden on the environment.

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ASSESSMENT OF TECHNOLOGICAL EFFICIENCY OF JOINT CARBOTHERMAL REDUCTION OF ILMENITE AND PEROVSKITE CONCENTRATES

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The advantages of the proposed joint processing of ilmenite and perovskite raw materials by pyrometallurgical method give the possibility to obtain rich titanium slags from concentrates of different types of ores and selectively convert rare earth metals into slag, and rare metals into the metallic phase, separating them from titanium within one technique.

According to the thermodynamic models obtained, the process of joint reduction of ilmenite (IC) and perovskite (PC) concentrates proceeds with the formation of an iron alloy and high-titanium slag (more than 70% TiO_2). The composition of the melting products depends on the temperature and carbon consumption. When stoichiometric amount of carbon is as required there is a possibility of complete transition of niobium into the metallic phase (cast iron) in the form of carbide. With a PC/IR ratio <0.5 , the formation of slags with a TiO_2 content of more than 80% can be expected, but the concentration of Nb extracted into the alloy will be low, and the content of REE in the slag will decrease several times compared to the initial one in PC. With a PC/IR ratio equal to 1.0, a temperature of 1500°C and carbon consumption value is higher than required by stoichiometry for complete reduction of iron, it is possible to obtain a titanium-rich slag (62-74% TiO_2) and completely convert all niobium and tantalum into an alloy in the form of carbides (3.4% NbC and 0.06% TaC).

As a result of experimental verification of thermodynamic models of carbothermic reduction of titanium concentrates, the possibility of their use for predicting the composition of interaction products and optimizing the operating parameters of the process – temperature, reagent consumption, the ratio of IR and PC during joint processing is shown.

Joint processing of titanium-containing ores will improve the environmental and economic efficiency of new and existing methods for processing mineral and technogenic raw materials of heavy non-ferrous metals and titanium in Russia.

The work was performed on the State assignment of the Institute of the Ural Branch of the Russian Academy of Sciences (state registration number of the topic: 122020100404-2).

IMPROVING THE EFFICIENCY OF USING BAKAL SIDERITES IN THE CAST IRON METALLURGY

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The share of the local iron ore raw materials of metallurgical enterprises in the Ural region is 50-60 %. The rest of the raw materials is imported from Central Russia, the Kola Peninsula and Kazakhstan. The replacing of the imported raw materials with local, cheaper ones is very relevant.

However, less than half of 50 iron ore deposits in the Urals are exploited, and the intensity of their production often does not coincide with the possibilities. In particular, the extraction of siderite iron ore from the Bakal deposit (Southern Urals), whose reserves amount to about 1 billion tons, is many times less than it is allowed by mining and geological conditions due to low demand for this raw material because of its poor quality. The high content of magnesium oxide in the ore makes blast furnace melting using more than 20 % of siderites in the charge difficult or impossible. Slags containing more than 20 % MgO are short and refractory.

The share of siderites in the charge can be increased up to 40-50 % on condition of further additive of 3 % boric anhydride. The slags formed during the melting process will be long and stable. At the tapping temperature (1450-1500 °C), their viscosity does not exceed 0.5 Pa·s, gradually increasing to 2.5 Pa·s at temperatures around 1150 °C. Boron-alloyed cast iron will be obtained as a result of such a melt.

The work was carried out according to the state assignment for IMET UB RAS using equipment of the Collaborative usage centre «Ural-M».

RESOURCE-SAVING TECHNOLOGY FOR PRODUCTION AND APPLICATION OF GRAIN MOLASSES IN THE RATIONS OF PRODUCTIVE ANIMALS

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At the moment, agronomic farms of the Russian Federation are faced with the problem of a surplus of grain crops associated with record harvests in recent years, as well as sanctions on export. On the other hand, in livestock enterprises there is a chronic deficit of carbohydrates in the diets of productive animals - from 40% to 70%, which is one of the reasons for the decrease in the profitability of this business¹.

One way to solve this problem is to process grain into feed energy ingredients containing 25–32% easily digestible carbohydrates. Feed energy ingredients are suitable for use in the diets of all types of animals: «liquid» - for cows, horses, goats, sheep; «wet» for pigs, calves, geese, ducks; «dry» for broilers, layers, turkeys. The technology has been implemented in a number of farms in the Russian Federation and the EU. It has a wide type and size range of production lines (from 60 kg to 70 tons or more of products per day) as well as it has technical and technological sovereignty and it serves as import advance.

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OBTAINING MICRO- AND MESOPOROUS ALUMINOSILICATES IN THE PRESENCE OF POLYETHYLENGLYCOL

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Template synthesis is a promising method for obtaining mesoporous materials that allow controlling the pore diameter and surface area¹. The porous structure is defined by selecting the appropriate template, which serves as the center around which the main structural units of the matrix are organized and the framework is formed. One of the most common nonionic polymers is the hydrophilic polymer polyethyleneglycol (PEG), which, depending on molecular weight and concentration, is able to form three-dimensional structures in aqueous solutions.

The purpose of this study is to obtain samples of potassium aluminosilicate from rice husks by template synthesis using PEG–6000.

In the work, samples of potassium aluminosilicate Al(K)-x were obtained (where x= 5; 10; 15; 20 mmol·L⁻¹ PEG). Rice straw (*Oryza sativa*) was used as a silicon source.

It was found that with an increase in the concentration of PEG, the structure of the material becomes more pronounced and clear. The sample with a PEG concentration of 20 mmol·L⁻¹ was the most structured. Visually defined boundaries between the particles indicate the organized nature of the surface. The surface of the sample has a spongy structure consisting of irregularly shaped particles.

The study of the structural characteristics of potassium aluminosilicate samples by low-temperature nitrogen adsorption showed that the introduction of the structure-controlling agent PEG-6000 leads to a decrease in the specific surface area and an increase in the proportion of pores with a large size.

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The work was performed within the framework of the state assignment of the Institute of Chemistry of the Far Eastern Branch of the Russian Academy of Sciences (FWFN(0205)-2022-0002).

HYBRID ENERGY-EFFICIENT METHOD - MEMBRANE-ASSISTED GAS ABSORPTION FOR THE REMOVAL AND CAPTURE OF ACID GASES

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Currently, chemical absorption using alkanolamine aqueous solutions is a prevalently used technology for the removal and capture of acid gases. Despite the fact that this process is widely used, it has a number of disadvantages such as loss of absorbent due to its degradation and formation of chemically active substances. In addition, this approach is characterised by high energy intensity, mainly due to the absorbent regeneration step, and high capital costs due to complex apparatus design and large size.

The developed method of membrane-assisted gas absorption is intended to increase the efficiency of the process of capture and removal of acid gases through the use of an energy-efficient and highly selective hybrid method. This process of gas separation, realised in a continuous stationary mode under the pressure differential, involves absorption of gas by a liquid absorbent located on the surface of a gas separation membrane. Conceptually, the process can be divided into several stages: injection of the gas mixture directly into the absorbent layer, absorption of acid gases, gas diffusion through the absorbent mass, desorption on the reverse side and gas transfer through the highly permeable polymer membrane. In such a process, the selectivity is mainly provided by the liquid absorbent and the productivity is provided by the highly permeable polymer membrane.

As a result of separation of the eight-component mixture simulating natural gas, it was possible to reduce the concentration of carbon dioxide from 5.40 to 0.07 mol.%. The concentration of hydrogen sulphide was reduced six times (from 1.39 to 0.23 mol.%). Thus, the achieved composition of the retentate stream meets the requirements of GOST 5542-2022 standart in terms of carbon dioxide content in commercial natural gas.

The study was funded by a grant from the Russian Science Foundation № 23-79-10288

OPTIMISATION OF THE MEMBRANE-ASSISTED GAS ABSORPTION METHOD OF CARBON DIOXIDE CAPTURE IN TERMS OF APPARATUS CONFIGURATION AND ABSORBENT COMPOSITION

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Currently, chemical absorption using aqueous solutions of amino alcohols is a universally used technology for the removal and capture of acid gases. Despite the fact that this process is extensively used, it has a number of disadvantages such as loss of absorbent due to its degradation and formation of chemically active substances. In addition, this approach is characterised by high energy intensity, mainly due to the absorbent regeneration step, and high capital costs due to complex apparatus design and large size.

The present study is devoted to the continued development, improvement and optimisation of a new hybrid separation method, membrane-assisted gas absorption, which is designed for the removal of acid gases. As part of the work, a new configuration of membrane-absorption gas separation module based on hollow fibres was proposed.

The main feature of the membrane-assisted gas absorption module used in this project is the use of a combined system of two types of hollow fibres. In this module configuration, the gas separation hollow fibre is placed inside the ultrafiltration fibre and is used to remove continuously desorbed gas from the liquid. The use of a gas separation membrane with a non-porous selective layer avoids leakage of the liquid used and further contributes to the overall selectivity of the process. Thus, the liquid absorbent is located in a closed volume between two hollow polymer fibres, through one of which the feed gas mixture is in contact with the liquid absorbent and through the other the absorbed component is removed.

The study was funded by a grant from the Russian Science Foundation № 23-79-10288

SYNTHESIS OF CHLORINATED COBALT PHTHALOCYANINE

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Today in the oil refining industry there is a need for development of domestic catalysts for regeneration of alkaline solution containing mercaptides, which is formed as a result of treatment of oil products. Cobalt phthalocyanine and its derivatives show the highest activity in regeneration of sulfur-alkali effluents^{1,2}.

The aim of the work is to synthesize low chlorinated cobalt sulfophthalocyanine for regeneration of alkaline solution. After synthesis, the product was analyzed for its catalytic properties in the oxidation reaction of sodium isopropylmercaptide in comparison with cobalt tetrasulfophthalocyanine (TSFC), American Merox catalyst and commercial cobalt dichlorodisulfophthalocyanine (DCDSPC). The obtained product in the ratio of 1:4 (MN SCPA:SPM) showed catalytic activity at the level of commercial dichlorodisulfophthalocyanine cobalt.

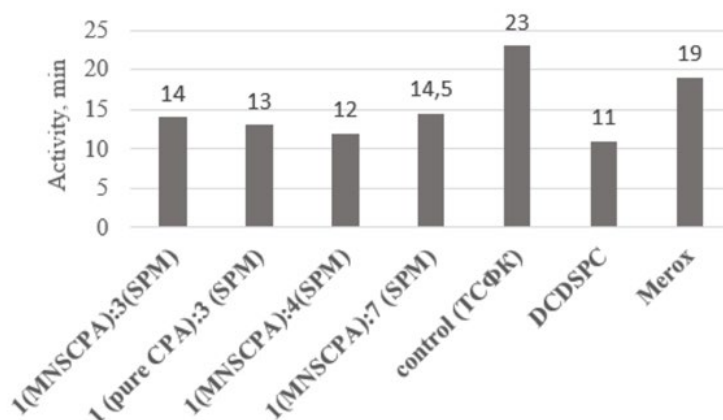


Figure 1. Dependence of FDC activity on the concentration of MN SHPC

Judging by the analysis of NMR spectra obtained from samples of monosodium salt of 4-chlorophthalic acid, chemical shifts of doublets 7.03-7.07; 7.12-7.14 and 7.29-7.31 belong to the chloro derivative of sodium phthalate.

The work was carried out with the financial support of Akhmadullin Scientific and Technical Center LLC

INFLUENCE OF ANTIOXIDANTS ON OXIDATION RESISTANCE OF RAPESEED OIL OF HIGH-VOLTAGE ELECTRICAL EQUIPMENT

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The possibility of replacing mineral transformer oil (dangerous for the environment) by vegetable oil with the addition of stabilizers against oxidation has been studied.

Table1.

Type of oil	Amount of AO, %	Content of volatile low molecular weight acids, mg KOH/1 g of oil	Acid number, mg KOH/1 g of oil	Sludge content, %
Refined rapeseed oil without antioxidants	-	5,990	5,105	0,2770
Transformer oil MIDELE N 1204 Fluid, CAS 68956-68-3	*	3,630	3,840	0,0054
Rapeseed oil with AO: OM AG-2 BF-5	0,083 0,083 0,083	4,635	4,200	0,0306
Rapeseed oil with AO: OM AG-2 BF-5	0,167 0,167 0,167	2,870	3,665	0,0008
Rapeseed oil with AO: OM AG-2 BF-5	0,333 0,333 0,333	3,150	3,750	0
Rapeseed with AG-2	0,5	3,790	4,190	0
Rapeseed with OM	0,5	2,740	3,660	0,0072
Rapeseed with BF-5	0,5	2,220	3,290	0,0009
Rapeseed with TTBBF	0,5	3,060	3,570	0,0424

* Content and type of the used antioxidant are unavailable

The results obtained during analyzing oxidation of rapeseed oil can be used for development of preparation of transformer fluids based on vegetable raw material. Therefore, it becomes possible to substitute MIDELE N 1204 transformer fluid for a domestic transformer fluid addition of BF-5 antioxidant.

NEW POLYIMIDES FOR MEMBRANE GAS SEPARATION IN AMMONIA INDUSTRY

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Ammonia production is a large-scale industrial process. To enhance its overall efficiency, it is common practice to recover hydrogen from purge gases and recycle it back into the synthetic cycle. On the other hand, the transition to a low-carbon energy and economy primarily involves the development of a hydrogen fuel infrastructure, and one of its components is the development of safe methods for H₂ storage and transportation. Ammonia is considered as a promising H₂ carrier due to its easy liquefaction and relatively high H₂ capacity. The catalytic decomposition of NH₃ leads to the formation of hydrogen/nitrogen and hydrogen/ammonia mixtures, from which a high-purity H₂ should be obtained for use in energy applications. For the H₂ separation from gas mixtures, membrane gas separation can be successfully applied. This approach offers several advantages: modularity, compactness, ease of implementation, maintenance, and scalability. Polyimide-based membranes have significant potential due to their advanced properties (high mechanical strength, high chemical stability, relatively high H₂ permeabilities and selectivities over other gases). One approach to the directed synthesis of polyimides with desired transport and separation properties involves the incorporation of bulky substituents into the polymer structure. However, this method often leads to an increase in the permeabilities with a dramatic decrease in selectivity, which eliminates the application of such materials for membrane gas separation. Therefore, the selection of substituents that lead to an improvement in the properties of a polyimide material is significant and relevant, both for addressing fundamental questions in materials science and for solving practical problems. The incorporation of phosphorus-containing groups into polyimides will improve their solubility, which is relevant to produce polyimide-based membranes. Additionally, the high fire resistance of these materials makes a significant contribution to ensuring the safety of processes when they are used. Thus, the report will present the first results of the novel project on new polyimides and gas separation membranes based on them for separation of H₂-containing mixtures in ammonia industry.

The study was carried in TIPS RAS and was supported by a grant from the Russian Science Foundation, No. 24-49-02058, <https://rscf.ru/en/project/24-49-02058/>.

PREVENTION OF AMINE SOLVENT DEGRADATION WITHIN POST-COMBUSTION CO₂ CAPTURE OR NATURAL GAS SWEETENING: AN OPPORTUNITY FOR MEMBRANES

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CO₂ capture using amine-based absorption is the mature technology that is widely used for natural gas sweetening and can easily be implemented in various industrial facilities to prevent CO₂ emissions. A drawback of this technique is the degradation of the solvent in the presence of oxygen, which accelerates at elevated temperatures. Oxidative amine degradation produces a mixture of different products, mainly carboxylic acids, amides, aldehydes, ammonia, etc. The interaction between the residual amine and the generated acids results in the formation of heat stable salts (HSS), which intensifies the corrosion process. The first way to expand the solvent lifetime is the removal of degradation products, or solvent reclaiming. A cycle of fundamental studies has been done by the TIPS RAS team on the development of the amine membrane filtration and electrodialysis purification from HSS. As a result, TIPS RAS and LLC "Reocat" developed a mobile containerized demonstration unit for commercial filtration and purification of alkanolamine solvents, with a capacity of 2.5 m³/h for degraded solutions. The second way to expand the solvent lifetime is the removal of dissolved oxygen from the amine solvents before thermal regeneration, or solvent deoxygenation. This process can be carried out in a compact, modular, and mobile form with hollow fiber gas-liquid membrane contactors. TIPS RAS team carried out research on the development of membranes and demonstrated the performance of contactors in the deoxygenation of the O₂-rich monoethanolamine solvent, which simulates the real one. The membrane scale up to size the developed composite membrane for a hypothetical post-combustion CO₂ capture plant with a solvent flow rate of 120 m³/h was estimated. The results of both membrane ways will be presented in the report.

Part of this work (first fundamental and pilot results of amine electrodialysis reclaiming) was funded from the EU FP7 under grant agreement n° 295645 (OCTAVIUS). Part of this work (industrial solvent filtration and electrodialysis reclaiming; scientific support and author's supervision for demo unit) was carried out within the state assignment of TIPS RAS (FFZN-2022-0004 Carbon dioxide capture and utilization, No. 123012300040-4). Membrane deoxygenation was carried out in TIPS RAS and was funded by Russian Science Foundation (RSF), grant number 21-79-10400. SDB, DOK and SAS acknowledge the support of RSF.

BROWN ALGAE BIOMASS FRACTIONATION USING ALKYLIMIDAZOLIUM IONIC LIQUIDS

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Arctic brown algae are considered a promising source of biologically active compounds in the form of polysaccharides, polyphenols, and low molecular weight secondary metabolites on an industrial scale. Existing technologies for their processing are mainly focused on the extraction of polysaccharides and are associated with the use of hazardous solvents.

In the present work, a green approach is proposed for the fractionation of brown algal biomass based on dissolution in ionic liquids (ILs) with 1-butyl-3-methylimidazolium (bmim) cation followed by sequential precipitation of polysaccharides and polyphenols with acetone and water, respectively.

The effects of the type of IL cation, temperature, and duration of treatment on the dissolution of algal (*Fucus vesiculosus*) biomass, the yield of fractions and their chemical composition were studied using FTIR and NMR spectroscopy as well as exclusion chromatography and monosaccharide analysis. It is shown that the use of bmim acetate allows almost complete dissolution of plant raw materials after 24 h treatment at 150 °C and separate separation of a mixture of polysaccharides (alginates, cellulose and fucoidan) and polyphenols with yields of ~40 and ~10%, respectively. Practically quantitative isolation of the polyphenolic fraction with a weighted average molecular weight of 10–20 kDa is achieved even under mild conditions (80–100°C). Efficient isolation of polysaccharides requires harsh conditions. Increasing temperature favors the increase of fucoidan content in the polysaccharide fraction.

This conceptual study opens perspectives for the development of new approaches for the valorization of algal biomass for the environmentally sustainable production of various value-added products.

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THE ELECTROCHEMICAL ETCHING METHOD APPLICATION FOR DETERMINING RESIDUAL STRESSES IN PIPES

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The method of layer-by-layer etching of metal layers with subsequent determination of elastic body reactions and indirect assessment of residual stresses is known in classical scientific literature^{1,2}. In this work, the method was used to calculate residual stresses in the wall of pipes 16x1.5 made of AISI 304 steel. The electrolyte composition and the current strength are selected for uniform bleaching and obtaining a smooth surface of the pipe.



Figure 1. Assembly of the electrochemical etching stand: 1 – clamp; 2 – glass cup with steel tape; 3 – rectifier, 4 – prepared sample

An acid solution containing H_3PO_4 – 85%, Cr_2O_3 – 13% (in grams) и H_2SO_4 – 2% was used as the electrolyte. It was found that to achieve a high-quality surface, a current of no more than 1-2 A should be used, since at a high etching rate, it is possible to etch grain boundaries with uneven removal of the metal layer.

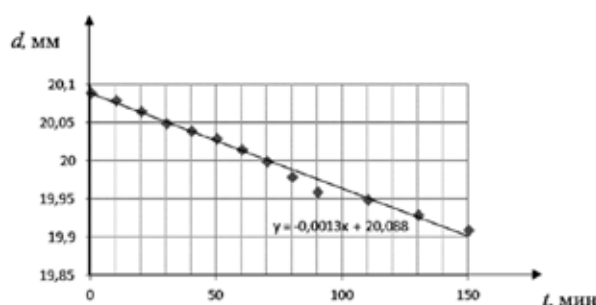


Figure 2. Changing the diameter of the sample during the etching process

As a result, according to the selected mode, experiments were carried out on a batch of pipes with the calculation of the residual stresses distribution along the wall³.

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EXTRACTION OF Pr^{3+} , Nd^{3+} , Dy^{3+} AND Tb^{3+} WITH DEEP EUTECTIC SOLVENTS BASED ON DIALKYLPHOSPHINE ACIDS

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Lanthanides and their compounds are used in various fluorescent materials, magnets, superconductors, lasers and many other materials. At the same time, the constant expansion of the areas of their application leads to an increase in demand for high-purity lanthanides¹. One of the stages of their production is extraction from acidic solutions, and to optimize this process, the active development of new extractants continues. One of the new generations of extractants are deep eutectic solvents, which are attracting increasing attention due to their commercial availability, ease of synthesis and lower risk².

This paper presents the results of studies of the extraction of Pr^{3+} , Nd^{3+} , Dy^{3+} and Tb^{3+} with deep eutectic solvents based on di(2,4,4-trimethylpentyl)dithiophosphinic and di(2,4,4-trimethylpentyl)phosphinic acids³. Menthol and phenol were used as hydrogen bond acceptors.

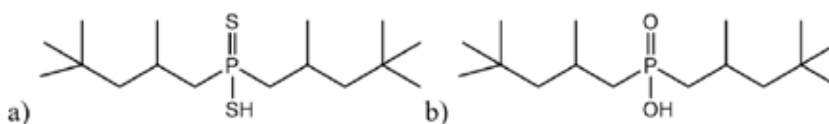


Figure 1. Structural formulas of a) di(2,4,4-trimethylpentyl)dithiophosphinic acid and b) di(2,4,4-trimethylpentyl)phosphinic acid.

The phase diagrams of two-component systems were studied and their physical properties were investigated. The quantitative characteristics of the extraction of Pr^{3+} , Nd^{3+} , Dy^{3+} and Tb^{3+} with the proposed deep eutectic solvents have been established. It has been shown that these eutectic solvents are promising extractants for the joint extraction of lanthanides from aqueous solutions.

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STUDY OF LANDFILL WATER FILTRATE

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The purpose of the work is to identify the causes of the unpleasant odor at the Barsukovka solid waste landfill in the Kaliningrad region and prepare recommendations for eliminating its causes.

Solid waste landfills are considered as complex natural-technogenic systems. In their massifs, waste decomposition processes occur, generating emissions of pollutants in the form of biogas and filtrate¹. Each period of the life cycle of a landfill corresponds to its own phase of biochemical destruction of waste. As oxygen reserves are used up, the activity of aerobic processes decreases, and anaerobic processes, caused by the activity of anaerobic microorganisms, among which sulfate-reducing bacteria predominate, begin to predominate in buried waste².

In areas where industrial and municipal wastewater enters, the sulfate reduction process is activated, which causes the appearance of sulfides in water and bottom sediments. Only sulfate-reducing bacteria can use sulfate as the final hydrogen acceptor during anaerobic respiration.

The samples taken in March 2024 are being examined. During the study, the degree of water contamination (oxidizability), the hydrogen index of samples, and the chemical composition using IR spectroscopy were determined. This made it possible to identify the presence of sulfate-reducing bacteria in the samples.

A number of recommendations to eliminate unpleasant odors have been developed: packing sludge deposits into tanks, mixing settled sediments with adsorption material and disinfecting the remaining water fraction with chlorination or hydrogen peroxide, which can be obtained by recycling sodium peroxide accumulated on military ships as a source of oxygen in emergency situations and also in need of disposal at the end of the shelf life.

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SYNTHESIS OF AEROGEL AND CRYOGEL FORMS OF NANOCOMPOSITE SORBENTS BASED ON CARBON NANOTUBES AND GRAPHENE OXIDE

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The report presents methods for producing multifunctional nanocomposite materials for effective water sorption from pollutants of various chemical natures. The sorbents under development are nanocomposites based on reduced graphene oxide (r-GO), modified with a functional organic component - polyaniline, also including oxidized carbon nanotubes (CNTs) as a structure former. The use of polyaniline can significantly increase the sorption activity and capacity of the synthesized materials. The authors were developed a number of nanocomposites that differ in the final stage of the technology - processing of a pre-prepared hydrogel: convective (sample No. 1), freeze drying (sample No. 2), as well as drying in a supercritical fluid - isopropyl alcohol (sample No. 3). In addition, by authors were studied the effect of an additional stage of samples carbonization in an inert media at 800 °C. For nitrogen adsorption, the specific surface area was: sample No. 1 – 80 m²/g, sample No. 2 – 180 m²/g, sample No. 3 – 290 m²/g. The carbonization stage allowed to increase the specific surface area to 350 m²/g. This fact can be explained by the partial thermal destruction of polyaniline and amorphous carbon, as well as the internal restructuring of graphene agglomerates containing CNTs. Also, the authors studied the static sorption capacity of nanocomposites under batch experiments in relation to organic dyes (methylene blue (MB) and solar yellow (SY)), as well as to heavy metals (zinc ions). It is noted that more than 90% of pollutants were removed within 30 minutes; the time for adsorption equilibrium to occur was 40 minutes in all cases. It has been established that the sorption value is up 1380 to 1800 mg/g for MB, up 159 to 300 mg/g for SY and up 31 to 230 mg/g for zinc ions. At the same time, the best characteristics were shown by the sample processed under supercritical conditions followed by carbonization.

This research was funded by the Russian Science Foundation, Grant No 22-13-20074, <https://rscf.ru/project/22-13-20074>

PRODUCTION OF CARBON MATERIALS BY PLASMA PROCESSING OF OIL INDUSTRY WASTES IN ARC DISCHARGE PLASMA UNDER DIFFERENT CONDITIONS

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In the context of environmental development and the great attention to the problems of rational use of natural resources, the search for new alternative methods of processing and using oil residues becomes relevant.

The aim of the research is to develop a technology to produce carbon materials by processing waste from the oil industry.

The object of study was asphalt-resin-paraffin formations (ARPF), heavy oil deposits on the inner surface of oilfield equipment, which complicate their production. The ARPF deposits studied belong to the paraffin type of deposits ($\beta=0.418$), the content of mechanical impurities is 0.92 wt. %. The main element in the ARPF molecule is carbon (99.5 wt.%). Destruction of ARPF occurs in the temperature range of 250-500 °C with the destruction of carbon-carbon and carbon-heteroatomic bonds.

Thermal processing of ARPF was carried out in a vacuum-free electric arc reactor under different conditions (current strength, exposure time): 50 – 125 A, 30 s; 100 A, 5 – 30 s. It was found that 5 s at 100 A and 30 s at 50 A are not sufficient to completely convert ARPF into carbon materials, since the product of plasma processing contains an amorphous fraction. X-ray diffraction analysis showed that the optimum conditions for processing of ARPF are 100 A and 30 s.

Thus, the work confirmed the possibility of obtaining carbon materials from oil waste and selected optimum conditions for plasma processing of oil waste with low energy costs (100 A, 30 s).

The work was carried out with the financial support of a grant from the Russian Science Foundation (project No. 22-13-20016) at SurSU and TPU.

CONVERSION METHOD OF PRODUCTION OF POTASSIUM FORMATE

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Potassium formate is widely used as a component of coolants, deicing materials and drilling fluids, due to low crystallization temperatures of aqueous solutions and low corrosive activity. Potassium formate is also used as a component of liquid fertilizers and feed additives in livestock farming.

The synthesis from formic acid, its esters, formaldehyde or carbon monoxide, as well as potassium hydroxide, carbonate or bicarbonate is multi-stage and requires the use of high temperatures and pressure. Alternative way to obtain potassium formate is an exchange reaction between sodium formate and water-soluble potassium salts ($\text{NaHCOO} + \text{KAn} \rightleftharpoons \text{KHCOO} + \text{NaAn}$).

It has been experimentally proven that the production of potassium formate is possible by using only potassium bicarbonate, carbonate or sulfate as the starting salts. When chloride, nitrate, dihydrogen phosphate or potassium hydrogen phosphate are used as raw materials, conversion is not possible.

As a result of the study of phase equilibria in quaternary reciprocal systems $\text{K}^+, \text{Na}^+ // \text{HCO}_3^-$, $\text{HCOO}^- - \text{H}_2\text{O}$ и $\text{K}^+, \text{Na}^+ // \text{CO}_3^{2-}$, $\text{HCOO}^- - \text{H}_2\text{O}$ at 25°C the conversion process for producing potassium formate has been established. The reaction of the starting salts is accompanied by the formation of crystalline sodium carbonate or bicarbonate, as well as solution containing potassium formate. Under optimal conditions, it is possible to obtain solutions of potassium formate with density of 1.45–1.49 g/cm³ and carbonate ion content of less than 5.0 wt.%, as well as crystalline anhydrous sodium carbonate. Synthesis using potassium bicarbonate is less preferred since, due to its lower solubility in water, more dilute solutions of potassium formate are formed.

Thus, the research carried out made it possible to propose a new low-waste method for the production of potassium formate from available raw materials.

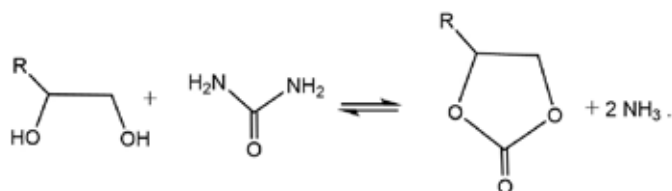
BIFUNCTIONAL CATALYSTS FOR PROPYLENE CARBONATE OBTAINING FROM UREA AND PROPYLENE GLYCOL

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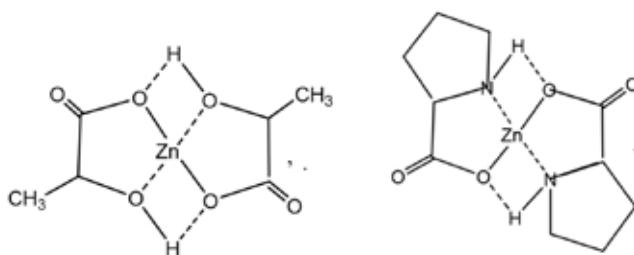
Cyclic carbonates have great application as solvents in different chemical transformations due to its low toxicity and good biodegradability. They are used as electrolytes in lithium-ion batteries¹. Polyethylene- and polypropylene carbonate are raw material for biodegradable packaging materials. Ethylene carbonate and its homologues are used for other dialkylcarbonates and glycerol carbonate obtaining².

Interaction of urea and 1,2-glycols is one of the most environmentally friendly method of cyclic carbonates producing:



Ammonia is the only by-product in this process. However, it can be used as raw material for urea obtaining. This fact makes this process non-waste.

We have shown, that effective bifunctional catalysts of reaction of urea and 1,2-propylene glycol with propylene carbonate formation are zinc salts of lactic acid and L-proline.



Possible reaction mechanism studied. Also, temperature, reaction time, and molar ratio influence on propylene carbonate yield was investigated.

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THE DEVELOPMENT OF PYROMETALLURGICAL TECHNOLOGIES BASED ON THE CHARACTERISTICS OF THE DISPERSED STATE OF MATTER

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The main way of obtaining metals from ore since the advent of metallurgy has been air purging of fuel pieces and ore components. In the second half of the 19th century, examples appeared that, it is more expedient to burn not pieces of coal, but coal dust or liquid fuel sprayed in nozzles from the point of view of energy saving. That is, go to dispersed systems. It took another 100 years for dispersed systems to be used in pyrometallurgical processes: pulverized coal fuel, sponge iron for steelmaking and others. Currently, another problem is coming to the fore. Rich deposits of metals have been practically depleted for example gold. And about 60% of the world's reserves of this metal are concentrated in refractory ores and technogenic formations. That is, it is in an ultra-dispersed state. Thus, the dispersed state of the substance becomes the basis for the development of pyrometallurgical technologies.

A method for selecting pulverized coal fuel components for blast furnaces has been developed and the possibility of using milled peat as part of the charge has been established on the basis of experimental laboratory studies (including the study of thermal destruction and combustion processes) and industrial experiments.

The role of dispersed carbon black is especially important in coke-free metallurgy. The carbon content of metallized products must correspond to the grade of steel being smelted. It was found that the particles of carbon black obtained by the Boudoir reaction have a complex openwork structure, including globular amorphous and graphitized crystalline elements connected by bridges, the sizes of which are in the nanometer range. The consequence of the highly dispersed and amorphous structure of carbon black is an increase in Gibbs energy and high kinetic activity.

Based on experimental data, a mechanism for the transition of gold and platinum into slag in non-ferrous metallurgy furnaces has been established. Matte droplets together with associated noble metal particles are carried into the slag by gas bubbles by flotation. A size of particle is no more than 5-7 μm . The stages and the general mechanism of flotation of dispersed sulfide and metal droplets in non-metallic melts are considered.

The research was funded in accordance with the state assignment on science for Ural State Mining University №075-03-2024-132 dated 17.01.2024 and the state assignment of the Institute of Metallurgy of the Ural Branch of the Russian Academy of Sciences using the equipment of the Ural-M Central Research and Development Center.

DEVELOPMENT OF THREE-DIMENSIONAL MODELS OF TECHNOLOGICAL LINES OF CHEMICAL ENTERPRISES

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Industry 4.0 technologies offer important perspectives for future innovations in manufacturing and industry. One of the promising areas developing within Industry 4.0 is the creation of digital twins¹.

At the first stage of creating the virtual space of the digital twin, a three-dimensional model is designed, which is an analog of chemical production with regard to the regulations and specifications². To create a three-dimensional model of technological lines of an industrial enterprise, a set of modeling tools from Autodesk was used. Currently, the transition to domestic products of information modeling, in particular NanoCAD, is underway.

On the basis of the data of the developed design documentation and the description of the chemical-technological process a three-dimensional model of the plant line is designed, this task is performed in several stages:

- development of three-dimensional models of technological devices according to the equipment specification;
- design of structural grid and steel structures;
- placement of designed apparatus models in accordance with the design documentation;
- creation of pipelines, fittings, pumping equipment and instrumentation.

Three-dimensional models provide an opportunity for qualitative calculation of geometric parameters for subsequent interpretation in real production, and can also be used as simulators for training students and staff of enterprises.

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TECHNOGENIC WASTES AND MATERIALS BASED ON THEM AS RAW MATERIALS FOR OBTAINING CERAMIC MATRIXES FOR IMMOBILIZATION OF COBALT-60 AND STRONTIUM-90

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In this study, the effectiveness of ceramic matrices for the immobilization of ⁶⁰Co, ⁹⁰Sr radionuclides from boron production waste (borogypsum) obtained by spark plasma sintering (SPS) and cold uniaxial pressing and sintering (CPS) was studied. SPS and CPS matrices were obtained directly from borogypsum and CaSiO₃ obtained by alkaline treatment of borogypsum^{1,2} (Table).

Table. Comparative analysis of quality indicators of ceramic matrices

Sample	Consolidation temperature (T), °C	Sintering method	Leaching rate (R _{Co} /R _{Sr}), g·cm ⁻² ·day ⁻¹
Borogypsum	800	SPS(R _{Sr})	3.67·10 ⁻⁵
	900		3.74·10 ⁻⁵
	1000		3.69·10 ⁻⁵
CoCaSiO ₃ from borogypsum	800	SPS(R _{Co})	1.08·10 ⁻⁶
	900		8.19·10 ⁻⁷
	1000		2.04·10 ⁻⁷
	1000	CPS (R _{Co})	2.81·10 ⁻⁶

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The research was carried out within the framework of the State Assignment No. FZNS 2024 0013, topic: "X-ray absorption spectroscopy on synchrotron radiation sources for the directed chemical synthesis of functional materials in demand in the nuclear industry"

SYNTHETIC COMPOUND AM-4 AS AN ANALOGUE OF A RARE MINERAL AND A BASIS FOR OBTAINING A NUMBER OF NEW USEFUL COMPOUNDS IN THE FIELD OF CATALYSIS, RADIO- AND ELECTROCHEMISTRY WHEN PROCESSING MINING WASTE

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The synthetic compound AM-4 is the only analogue of rare minerals of the lintisite group (Khibiny alkaline massif, Kola Peninsula, Russia). New areas of promising application of these minerals have been confirmed experimentally, both as a result of many years of studying natural samples and their synthetic analogues. This work presents data on laboratory and large-scale laboratory synthesis of AM-4 based on accessible, inexpensive titanium-containing raw materials from the Kola Peninsula that require additional processing. It is shown that the material under study can fairly complement the list of already known heterogeneous catalysts for the pharmaceutical and paint and varnish industries, sorbents for the selective extraction of iodine ions and 137-cesium radioisotopes, and also act as an interesting basis for the production of composite materials of complex chemical composition for problems of electrochemistry and catalysis. The work also provides data on methods of granulation of AM-4 and its modifications using different types of granulations: centrifugal, rotary screw and granulation of samples in a fluidized bed.

The work was carried out within the framework of the Russian Science Foundation project 24-23-00374 and the topic of the state assignment FMEZ 2022–0019.

RICE HUSK CARBONS MODIFICATION FOR ADSORPTION PROPERTIES IMPROVEMENT

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The problem of wastewater treatment from various human-generated pollutants, including medications, is a pressing concern. One of the most used medications is diclofenac, which negatively affects the environment.¹ Developing of effective adsorbents for removing this substance is an important goal. Another urgent issue is utilization of agricultural waste, such as rice husk (RH), which is a by-product of rice production and is produced in enormous amounts.² The aim of the study was to investigate the potential use of rice husk carbon for the removal of diclofenac from water, which can solve both problems of RH utilization and adsorbent producing.

In this work, diclofenac adsorption on carbons produced by rice husk pyrolysis was studied. The possibility of chemical modification of carbon surface and changing its physicochemical properties by doping it with nitrogen using various methods was investigated. N-doped carbon with 6.5 at.% of nitrogen in the surface and surface area 630 m²/g was synthesized.

Adsorption equilibrium studies showed high adsorption capacity of obtained rice husk carbons towards diclofenac. Adsorption kinetics on carbons with different nitrogen content was also investigated. Doping carbon surface with nitrogen-containing functional groups showed to improve adsorption efficiency. Based on this, it has been concluded that it is possible to improve the adsorption characteristics of carbon through amination.

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The work was carried out as part of the state-funded project «Surface physicochemistry, adsorption and catalysis» № AAAA-A21-121011990019-4.

IRREVERSIBLE DISCOLORATION AND DETOXIFICATION OF FOOD DYES

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Electron beam treatment (radiolysis) of food dyes solutions demonstrates the possibility of effective irreversible degradation of conjugated bond systems. For 10 representatives of quinophthalone, indigoid, triphenylmethane and azo dyes, the same type of correlation is observed between the absorbed dose and the degree of discoloration. At a dye concentration of 20 mg/dm³, complete discoloration is observed at ≈ 1 kGy (1 kJ/kg) dose. The effect is achieved under conditions of deficiency of dissolved oxygen. It is these conditions that are most attractive for the neutralization of colored wastewater using powerful electron accelerators¹⁻³.

The main transformations are caused by the dissociative addition of OH radicals to conjugated bonds. For example, at 1-1.5 kGy dose, each dissolved molecule of the E124 dye can interact with several OH radicals and, thus, lose all side groups without the formation of mutagenic products. Degradation of the chromophore system occurs with the elimination of the connecting bridge and side substituents due to the formation of a new functional group OH, the binding energy of which is higher than that of sulfo groups and diazo bridges. Dye E124 in a 0.02% aqueous solution is initially a nonspecific mutagen. Without metabolic activation, it induces G→A transitions as well as +1 and -1 frameshift mutations. In turn, in the presence of liver enzymes, it causes mutations of the first two types. The decrease in color and mutagenicity occurs in parallel and is caused by the same radical processes, and mutagenic activity decreases to the level of spontaneous mutagenesis.

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RESEARCH OF EMULSION EXTRACTS OF PINUS AND ABIES BY MICROSCOPIC METHOD

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Coniferous wood greenery are a rich source of compounds that have surface-active properties (salts of resin and fatty acids, phospholipids, glycerides, etc.) that affect the formation of the emulsion during the extraction of raw materials. The task of this work is to study the technological conditions affecting the formation of an emulsion from *Abies* and *Pinus* wood greenery.

We have studied the effect of the concentration of an aqueous alkali solution, the hydromodulus and the duration of extraction on the formation of the emulsion and its quality (the size of particles of the dispersion phase and their distribution). To assess the influence of technological parameters on the formation of the emulsion, an analysis of the extracts was carried out using a microscopic method to determine the average diameter of microdroplets of the dispersed phase and their distribution in the dispersion medium.

It has been established that under optimal conditions an «oil in water» emulsion is formed with an average particle size of the dispersed phase of *Pinus* extract of 2.5 microns and *Abies* extract of 2.7 microns (Fig.). The particles of the dispersed phase represent the lipophilic fraction of the components of woody greenery, the dispersion medium is an aqueous-alkaline solution containing water-soluble compounds of the raw materials.

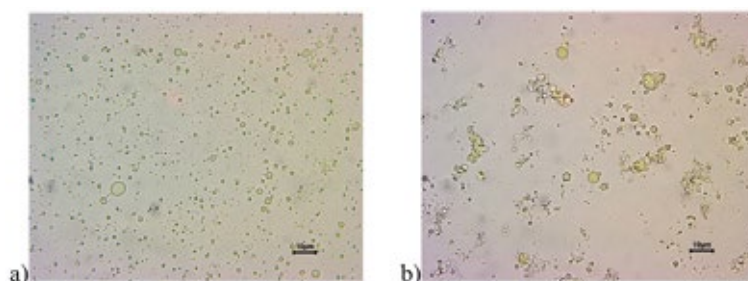


Figure 1. Microphotographs of emulsion extracts of wood greenery of *Pinus* (a) and *Abies* (b)

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DEVELOPMENT OF AN COMPLEX TECHNOLOGY FOR PROCESSING FROM PINUS WOOD GREENERY

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Complex rational processing of renewable plant raw materials, including logging waste, using the principles of green chemistry to obtain practically significant biological products is an urgent task in forest chemistry. At the Institute of Chemistry Komi Science Center, the scientific basis for the creation of new drugs based on extractive compounds of wood greenery coniferous that have biological activity is being developed. The novelty of the approach lies in the use of an environmentally friendly emulsion method for extracting valuable extractive components.

Research has been carried out to develop a method for complex processing of wood greenery pine by extracting polysaccharides and subsequent emulsion extraction of low molecular weight components. The yield of polysaccharides pine amounted to 3.56% by weight of dry raw materials. Subsequent emulsion extraction of raw materials isolates the extractive components of pine with a yield of up to 3.25% by weight of dry raw materials¹.

Natural polysaccharides have anticoagulant, antiviral, antioxidant, antitumor, and anti-inflammatory activity. Low molecular weight pine compounds are distinguished by a high content of diterpene acids with proven antioxidant and immunostimulating properties². The isolated components can be used in the pharmaceutical industry and agriculture.

Coniferous meal, formed after the extraction of polysaccharides and extractives, is washed with water, dried at a temperature of 50-60 °C and used as a feed additive for animals at a dose of 2-6% of the main diet.

Involvement of wood waste in economic circulation contributes to the rational and economical use of forest resources.

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SEPARATION OF AN INDUSTRIAL MIXTURE OF DECALIN OR NAPHTHALENE FLUORINATION PRODUCTS. PERFLUORODECALIN – PERFLUOROBUTYLCYCLOHEXANE

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Perfluorodecalin (PFD) is widely used in medicine and various fields of chemistry. The main industrial method for producing PFD is the fluorination of decalin or naphthalene. In this case, the carbon chain of part of the molecules is destroyed and impurities are formed, including perfluoro(butylcyclohexane)¹ (BCH), which has its own commercial value. In industry, the PFD–BCH mixture is separated by crystallization, which is aimed only at obtaining PFD.

Distillation has a number of advantages over crystallization, but the separation of PFD and BCH is complicated by thermodynamic limitations². Our own experimental data showed the absence of separation during the distillation of a cis-PFD–BCH mixture on a column with an efficiency of 85 theoretical plates.

In this study, to intensify the distillation separation of the cis-PFD–BCH mixture, we proposed to use the method of heteroazeotropic distillation in the presence of water. In this process, BCH is concentrated in the bottoms, and PFD is concentrated in the distillate. This method makes it possible to obtain BCH and cis-PFD with a purity of up to 99.7 and 99.8% wt. respectively. It should be noted that water, due to its low mutual solubility with PFD and BCH (about 15 ppm), can be separated by decanting. In addition, the water is safe and non-toxic, which is especially important due to the use of PFD in medicine.

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SORPTION OF DISSOLVED OIL PRODUCTS WITH CARBON-CONTAINING MATERIALS FROM PLANT RAW MATERIALS

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Today, oil remains the main energy resource in the world. In the process of extraction, processing and transportation, due to accidents or spills, a significant part of the oil enters natural and wastewater. Currently, new and well-known methods of wastewater treatment from oil products are being actively developed and improved, among which adsorption occupies an important place. Plant raw materials, including agricultural waste, which are generated annually in large quantities at grain processing enterprises, attract great attention as sorbents.

The purpose of the work: to investigate the sorption of dissolved oil products by materials obtained from waste products of rice and sunflower production.

Carbonaceous materials obtained from rice (RH) and sunflower (PH) husks were used as the object of the study. Sorbent samples were obtained by roasting rye and PS under conditions of oxygen deficiency¹.

Extracts of oily waters before and after the sorption process were studied by gas chromatography-mass spectrometry. It is shown that the composition of dissolved oil products includes branched and unbranched alkanes with different carbon chain lengths. The extraction efficiency of decane is almost 100%, of hydrocarbons with a chain length of C_{16} - C_{20} – 30%.

The processing of kinetic data using pseudo-first, pseudo-second order, Elovich and Maurice-Weber models showed that the sorption process is limited by interaction with an inhomogeneous surface of carbon-containing materials.

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DEEP EUTECTIC SOLVENT BASED ON D2EHPA AND TBP FOR RECYCLING LFP BATTERIES

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Today, lithium iron phosphate (LFP) batteries are promising current sources for various types of electronic equipment. However, due to such high demand, an excessive number of end-of-life batteries may be generated in the coming decades, creating an urgent need to develop a recycling strategy for LFP batteries. Hydrometallurgical methods for processing current sources, including leaching followed by extraction separation of metals, are the most promising.

This work proposes a new hydrophobic deep eutectic solvent based on di(2-ethylhexyl)phosphoric acid (D2EHPA), tributyl phosphate (TBP) and menthol as an extractant for metal ions included in lithium iron phosphate batteries. Previously, a mixture of D2EHPA and TBP was dissolved in volatile organic solvents and used for the synergistic extraction of metals from their aqueous solutions¹. This work uses a safer and more environmentally friendly approach - replacing the flammable solvent with a substance of natural origin - menthol. The formation of the deep eutectic solvent D2EHPA/TBP/menthol was confirmed by IR spectroscopy. Both the physical properties of this extractant (density, viscosity, refractive index) and extraction properties in relation to a number of metal ions (Li, Fe, Cu, Al) were studied, depending on the process conditions. A search for synergism of the extractant under study was also carried out by comparing the extraction properties of D2EHPA/TBP/menthol with D2EHPA/menthol and TBP/menthol.

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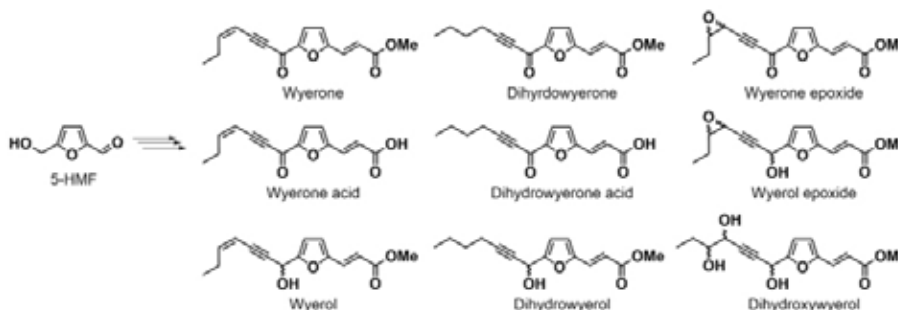
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Biomass conversion is one of the most promising areas of modern green chemistry. The main product of hexoses conversion is 5-(hydroxymethyl)furfural (5-HMF), which has been included in the list of platform compounds for the chemical technology of the future.

In this work, natural fungicides from the class of furanoacetylenes, released by legumes when infected by mold fungi,¹ were synthesized from 5-HMF. The furanoacetylenes include compounds of the wyerone series, which are phytoalexins, low molecular weight substances rapidly produced by plants in response to contact with a phytopathogen. Phytoalexins are part of the plant immune system and are considered as natural fungicides, insecticides and herbicides.²



Laboratory synthesis of these natural plant protection agents is a daunting challenge, as it will allow to significantly reduce the costs of their production, compared to the isolation of phytoalexins from plants. The efficiency and toxicity of the obtained compounds of wyerone series are now being studied.

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PROSPECTS FOR THE APPLICATION OF CONCRETE MIXTURES BASED ON ALUMINA AND ORDINARY PORTLAND CEMENT FOR GEOLOGICAL ISOLATION OF RADIOACTIVE WASTE

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In many concepts of geological disposal facilities for radioactive waste (GDF), the use of cementitious materials to create engineered safety barriers (ESB) is considered¹. An important criterion that determines the method of application ESB based on cementitious binders is the mobility of the concrete mixes used, which is the basis for choosing the most effective method of their application to the surfaces of underground drift. Also, a significant parameter of the concrete ESB in GDF is the hydraulic permeability to groundwater, the values of which for the considered materials should not exceed the values obtained for the rocks in which the isolation of radioactive waste will be carried out¹.

The purpose of this work is to determine the mobility of mixes based on ordinary Portland cement (Portland cement concrete (PCC) and cement buffer NRVB) and calcium aluminate cement (calcium aluminate concrete (CAC)), as well as the hydraulic permeability to a synthetic groundwater of the 'Yeniseysky' site for the studied concretes.

According to the methodology of GOST 5802-86 and the criteria of GOST 28013-98, it has been established that NRVB and CAC can be applied using mechanized means, while PCC can be used for manual application. It has been shown that the hydraulic permeability of PCC, NRVB, and CAC does not exceed the order of magnitude of 10^{-10} m/s¹, characteristic of granite rocks similar to the 'Yeniseysky' site of the Nizhnekansky massif, selected for the construction of GDF in Russia. Thus, it can be said that the studied compositions are promising for creating ESB in GDF due to their low permeability to groundwater and the identified effective methods of their application.

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STUDY OF GAS TRANSPORT CHARACTERISTICS OF INDIVIDUAL GASES AND COMPONENTS OF GAS MIXTURE OF POLYMER HOLLOW FIBER MEMBRANES FOR AIR SEPARATION

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Air separation is one of the main processes of chemical technology. Membrane technology makes it possible to obtain nitrogen with a concentration of more than 99 vol.% and is characterized by high energy efficiency in comparison with competitors – cryogenic and sorption methods.

Traditionally, the study of one of the main gas transmission characteristics of membrane elements – permeance – is carried out for individual pure gases. The permeance of each of the components of the gas mixture is measured separately and compared with each other to assess selectivity. This approach does not allow to objectively measure permeance due to the inability to take into account the phenomena of competitive sorption and plasticization of the polymer.

An alternative method for studying permeance is a method using a mass spectrometric complex, which allows real-time recording of the permeate flow simultaneously for each component of the gas mixture. This approach allows us to obtain permeance of the components of the gas mixture during the gas separation process.

In this paper, two described approaches to the study of permeance were compared using the example of air separation. Membrane elements from five promising commercially available polymer hollow fiber gas separation membranes were used: polysulfone (PSP), polyphenylene oxide (PPO), polyetherimide (PEI), polyimide (PI) and polyetherimide with polyimide (PEI+PI). The results of the experiment showed significant deviations. The permeance of the polyetherimide membrane for nitrogen in the composition of the air mixture was 4.33 GPU, and for pure nitrogen – 3.45 GPU, which is 25.5% lower, and on average the discrepancy between the permeance of the membranes for nitrogen was 17.3%.

The work was carried out with financial support from the Ministry of Science and Higher Education of the Russian Federation within the framework of a scientific project under state assignment No. FSSM-2023-0004

OPTIMISATION OF THE MEMBRANE-ASSISTED GAS ABSORPTION METHOD OF CARBON DIOXIDE CAPTURE IN TERMS OF APPARATUS CONFIGURATION AND ABSORBENT COMPOSITION

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Currently, chemical absorption using aqueous solutions of amino alcohols is a universally used technology for the removal and capture of acid gases. Despite the fact that this process is extensively used, it has a number of disadvantages such as loss of absorbent due to its degradation and formation of chemically active substances. In addition, this approach is characterised by high energy intensity, mainly due to the absorbent regeneration step, and high capital costs due to complex apparatus design and large size.

The present study is devoted to the continued development, improvement and optimisation of a new hybrid separation method, membrane-assisted gas absorption, which is designed for the removal of acid gases. As part of the work, a new configuration of membrane-absorption gas separation module based on hollow fibres was proposed.

The main feature of the membrane-assisted gas absorption module used in this project is the use of a combined system of two types of hollow fibres. In this module configuration, the gas separation hollow fibre is placed inside the ultrafiltration fibre and is used to remove continuously desorbed gas from the liquid. The use of a gas separation membrane with a non-porous selective layer avoids leakage of the liquid used and further contributes to the overall selectivity of the process. Thus, the liquid absorbent is located in a closed volume between two hollow polymer fibres, through one of which the feed gas mixture is in contact with the liquid absorbent and through the other the absorbed component is removed.

The study was funded by a grant from the Russian Science Foundation № 23-79-10288

SORPTION OF TBP BY POLIMER SORBENT POLYSORB-1

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In this work, using small-angle X-ray scattering methods, we obtained data on the porous structure and mechanism of adsorption of TBP on Polysorb-1, which is a copolymer of styrene with 40% divinylbenzene in the presence of 100% pore-forming agent - BR-1 gasoline.

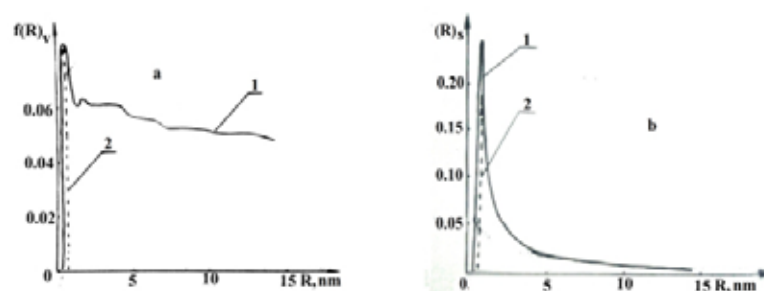


Figure 1. Differential distribution curves of pore volumes (a) and pore surfaces (b) by inertia radii: 1 - dry sample; 2 - sample, saturated with TBP.

The pore size distribution includes micro-, super-micro and meso-pores. The sorbent has one maximum at $R=0.5$ - 0.6 nm corresponding to micro-pores. From a comparison of curves 1 and 2 (curve 2 at $R \geq 0.8$ nm coincides with curve 1), we can conclude that the meso-pores and part of the super-micro-pores are filled with TBP. The minimum radius of gyration of pores filled with TBP is 0.5 - 0.6 nm and the position of the distribution maximum shifts to 0.7 - 0.8 nm, and its height decreases. When TBP is adsorbed from an aqueous solution, the distribution of the pore surface becomes of paramount importance. This is due to the fact that the binding energy of the dipole-dipole interaction between TBP molecules is less than the energy of the hydrogen bond of phosphoryl oxygen in the TBP molecule with a water proton¹ and adsorption in this case should be monomolecular in nature. TBP sorption occurs mainly in mesopores at $R \geq 1$ nm, which accounts for $\sim 80\%$ of the total pore surface.

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A NEW APPROACH TO RECYCLING PLASTIC BOTTLE CAPS AND RINGS

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Intensive consumption of plastic products has created a problem of accumulation of plastic waste. An increase in the volume of consumption of polymer materials leads to the accumulation of non-degradable waste from used plastic products, which ultimately leads to pollution of the air, soil, and groundwater with products of incomplete decomposition, including nano- and microparticles of polymers. In the total mass of polymer waste, the main share is occupied by high- and low-density polyethylene (LDPE, HDPE), propylene (PP) and polyethylene terephthalate (PET), the problem of recycling waste from these plastics is currently relevant.

Previously, a new physicochemical method was proposed for recycling PET bottle waste, based on their gas-phase processing in a nitrating atmosphere with subsequent disposal of conversion products. This method allows to recycle PET bottle waste regardless of its color. At the same time, bottle waste contains rings and caps made from other types of plastic (for example, LDPE, PP). Currently, production processes for recycling bottle waste, with the exception of mechanical and thermal recycling, require the separation of caps with rings from bottles. In this regard, it was of interest to find out the possibility of recycling rings and caps from plastic bottles by gas-phase processing in a nitrating atmosphere with subsequent disposal of conversion products.

The paper discusses a new method for processing plastic waste (fixing rings and caps from plastic bottles of mineral and carbonated water), based on their gas-phase processing in a nitrating atmosphere with subsequent disposal of conversion products. It has been established that keeping samples of caps (LDPE, PP, PET) in a nitrating atmosphere obtained as a result of evaporation of 12 mol/l HNO_3 at temperatures of 403-423 K for 24 hours, followed by dissolution of the conversion products in 1.0 mol/l NaOH and ozonation the resulting solutions allows for effective disposal of bottle waste, regardless of the type and color of plastic.

EXTRACTION OF URANYL TRICARBONATE COMPLEX BY CLAY MATERIALS FROM AQUEOUS SOLUTIONS

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An important component of the system of engineering safety barriers (ESB) at disposal or storage sites for radioactive waste (RAW) is clay buffer material, the main purpose of which is to limit and delay the release of radionuclides into the environment. During long-term operation of radioactive waste storage facilities, the possibility of water penetration into the insulation circuit due to changes in the hydrological regime, geological, technogenic or other processes cannot be excluded. To predict the state of ESB after water penetration, studies of the properties of barrier mixtures and their behavior under various watering scenarios are necessary. Clays have a high adsorption capacity for cations, with the highest values being characteristic of minerals of the montmorillonite group, and the lowest values being characteristic of kaolinites. The adsorption of anions is weaker than the adsorption of cations and occurs on the side chips of clay particles. The chipped area is an insignificant fraction of the entire surface of the particles as a whole. At the same time, as a result of both activation of the clay surface and the inclusion of various anions capable of exchange into the clay structure, it is possible to increase the adsorption capacity of clays for anions. A promising method for activating clays is alkaline activation. Alkaline activation of natural aluminosilicates, compared to acidic activation, has a number of fundamental differences associated both with the mechanism of dissolution of clay minerals and with the composition of the products formed in the solution.

The work investigated the processes of extraction of the uranyl tricarbonat complex $[\text{UO}_2(\text{CO}_3)_3]^{4-}$ from aqueous solutions on clay powders from kaolin clays of the Kampanovskoye deposit and from bentonite clays of the 10th Khutor and Dinozavrovoye deposits, as well as their mixtures. The studies were carried out with clay powders, both untreated and treated with water, solutions of 0.5 mol/l Na_2CO_3 and NaNO_3 , and 2 mol/l solutions of NaOH . The $[\text{UO}_2(\text{CO}_3)_3]^{4-}$ complex is not sorbed on clay materials from aqueous solutions under static conditions. Filtration of an aqueous solution of $[\text{UO}_2(\text{CO}_3)_3]^{4-}$ through columns with clay mixtures makes it possible to extract up to 87% of uranium from the amount passed through the column.

DEVELOPMENT OF A COST-EFFECTIVE SCHEME FOR PROCESSING ASH AND SLAG WASTE

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In Russia, according to various estimates, over 1.7-1.9 billion tons of ash and slag waste (ASW) from coal combustion have been accumulated. The relevance of the research is due to the low degree of their recycling (on average 10-15%) and the need to achieve by 2035 a 50% recycling level of the annual volume of ash and slag waste generation planned in the Energy Strategy of Russia¹. Solving the environmental problem of eliminating ash and slag waste dumps will simultaneously expand the country's raw material base by involving this aluminosilicate mineral raw material in the processing. Currently, the main direction of recycling is the use of ASW in the construction industry, road construction and agriculture. However, their direct use is often limited due to the need to comply with increased requirements for the composition and physical and chemical properties of the replaced components (sand, etc.), which is one of the main reasons for low demand. A promising way to solve environmental problems associated with the disposal of ash and slag is its comprehensive processing for the purpose of producing marketable products. An analysis of the results of a technical and economic assessment of the scheme developed at ICEMR RAS showed the possibility of almost complete processing of ash generated from the combustion of ash and slag waste, with a productivity of 100 tons/hour, producing marketable products: coal concentrate with a carbon content of 58%, iron-containing concentrate (58% Fe₂O₃) and aluminosilicate product for construction, road-building and other industries, with a discounted payback period of 1.84 years.

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AGRONOMY EFFICIENCY OF STRUVITE PRODUCED USING MODIFIED TECHNOLOGY

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Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is used as a key component to obtain long-acting fertilizer.¹ However, the rate of release of nutrients by struvite obtained using traditional technology (St_{trad}) turns out to be lower than the rate of N and P consumption by agricultural crops, and there is a need to additionally solve the problem of maintaining a balance between the release and consumption of essential elements. The development of a modified technology for the chemical deposition of struvite (St_{mod}), based on the conduct of nucleation and phase formation processes using a low-barrier phase formation mechanism with the participation of prenucleation and ion-associated clusters², has opened up the prospect of significantly increasing the efficiency of using St_{mod} -based fertilizers when growing crops.

Using the example of growing radish variety Saksa RS (Gardenstar) using St_{mod} and St_{trad} as fertilizers, as well as commercial fertilizer - ammoniated superphosphate «Fasco», agronomic efficiency was determined. In Fig. 1 shows the results of an experiment conducted on sandy soil.

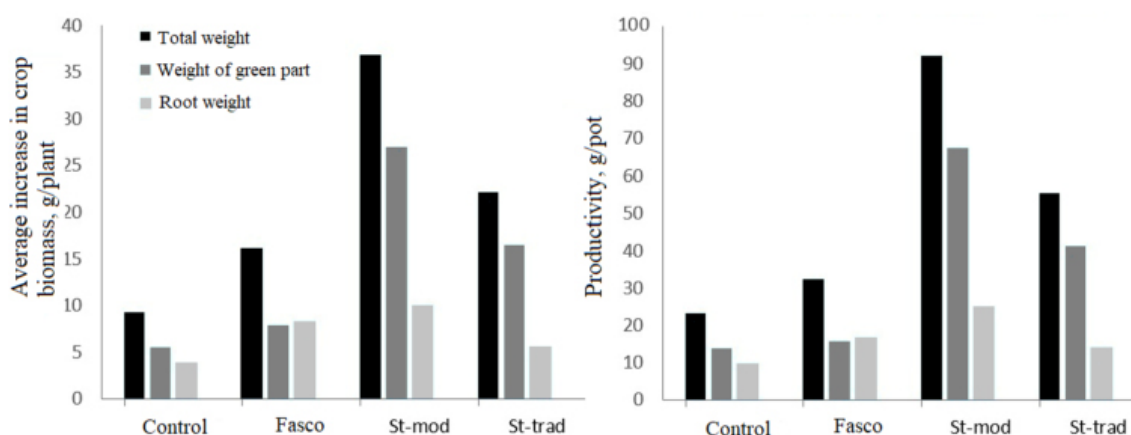


Figure 1. Dependence of average biomass growth and yield depending on the type of fertilizer applied.

The results of vegetation experiments confirmed the high agronomic efficiency of St_{mod} compared to St_{trad} and commercial mineral phosphate-containing fertilizer.

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ANALYSIS OF REALIZATION OF LIQUID PSEUDOMEMBRANE TECHNIQUE PROCESSES WITH NATURAL AND FORCED CIRCULATION OF MEMBRANE PHASE

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The problem of utilization of waste energy sources requires the search for modern effective environmentally safe technologies for their processing. Solvent extraction with deep eutectic solvents solves the problems associated with the use of traditional extractants¹. One of the key advantages of deep eutectic solvents is the possibility of fine-tuning the extraction properties depending on the process conditions, which makes them promising for the realization of multistage extraction separation processes using the liquid pseudomembrane technique. Under these conditions, the deep eutectic solvent acts as a mobile liquid membrane, which is not discharged from the apparatuses, but circulates between the phases of the initial solution and the re-extractant².

The paper presents the results of mathematical modeling and experimental study of the influence of the way of organizing the circulation of the membrane phase in a cascade of mixed-swell extractors organized on the principle of liquid pseudomembranes on the efficiency of Fe^{3+} extraction by hydrophobic deep eutectic solvent based on dodecanoic acid, menthol and tributyl phosphate. The analysis of the obtained data showed that the efficiency of Fe^{3+} extraction increases with the increase of the circulation rate of the organic phase, with the maximum efficiency being achieved when the unit is operated without forced circulation.

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THE EFFECT OF THE CHARGE AND SIZE OF THE DISPERSED PHASE ON INCREASING THE EFFICIENCY OF ELECTROFLotation EXTRACTION OF FRESHLY DEPOSITED BARIUM SULFATE

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Barium sulfate is an important component for the negative electrode paste of a lead-acid battery¹. Due to its high reflectivity, BaSO₄ is of interest and practical importance as a reflective pigment on thermoregulating coatings of spacecraft and rockets². The aim of the work is to study the effect of the physico-chemical characteristics of the dispersed phase on the efficiency of electroflotation extraction of freshly deposited BaSO₄ from wastewater. Isolation of it as a valuable component from liquid man-made waste.

Studies of the particle size (d_{av} , μm) and the values of the electrokinetic potential (ζ , mV) of the dispersed phase of BaSO₄ were carried out. It was found that the use of Superfloc C-454 cationic flocculant and neon Praestol 2500 is able to increase the degree of BaSO₄ extraction to 99.7% and reduce the residual concentration in the filtrate to 0.15 mg/l. Such a high degree of BaSO₄ extraction is due to the adhesion of particles, up to the d_{av} values of the dispersed phase in the range of 26-31 microns. In this case, the ζ -potential becomes positive (12.7 mV) or approaches the isoelectric point (-4.4 mV).

Flocculant	Btand	d_{av} , μm	ζ , mV	α_{ef} , %	α_{ef+p} , %	$C_{res(ef+p)}$, mg/l
Without flocculant		20	-32	59	99,2	0,38
Kationic	Superfloc C-454	31	12,7	95,7	99,7	0,15
	Praestol 854	24	20	88,2	97,2	1,4
Anionic	Superfloc A-110	31	-35	75,3	97,5	1,25
	Praestol 25-40	26	-35	87,9	97,7	1,15
Non-anioinic	Superfloc N-300	42	-4,4	77,5	98,7	0,65
	Praestol 2500	20	-4,4	95,2	99,7	0,16

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PRODUCTION OF FUNCTIONALIZED BIOCHARS FROM BIOMASS WASTE

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Biochar is a carbon-containing product obtained from biomass. Biochar is widely used as energy sources, sorbents, catalysts, etc. The composition and properties of biochar strongly depend on the nature of the feedstock and production methods. Pyrolysis, torrefaction and hydrothermal carbonation can be distinguished among the methods of producing biochar. The conversion of biomass waste into useful materials, such as bio-coal, is a promising method of increasing the value of waste. The development of biochar production technology makes it possible to obtain nanoscale materials with an increased surface area and improved structural characteristics. However, further research is needed to ensure a high yield of bio-carbon, as well as the development of methods for the production of carbon-containing composites. Existing technologies for the production of biochar from waste need to be improved. The key direction for increasing the efficiency of the carbonation process is the optimization of technological conditions, the development of new approaches to carbonation, etc. This will increase the yield of biochar and its quality.

In this work, a comparison of biochar from birch and pine sawdust obtained by pyrolysis, torrefaction and hydrothermal carbonation was carried out. The yield of biochar, specific surface area, carbon content and abrasion resistance were studied when using sawdust and solid residues after hydrolysis and liquefaction as feedstock. For the first time, the effect of the addition of modifying agents (metal salts and alkoxysilanes) on the properties of biochar was studied. It was found that the introduction of additional mineral components into the composition of bio-carbons increases their abrasion resistance by creating an additional inorganic «framework».

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THE RISKS OF LONG-TERM MONAZITE ENRICHMENT TAILINGS STORAGE AND POTENTIALS WAYS OF THEIR RECYCLING

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The results of performed field dosimetry on the territory of an unused monazite enrichment tailings storage facility located in Baley, Zabaikalsky region¹⁻³, and laboratory gamma spectroscopy of soil samples indicate an unfavorable radioecological situation on the research site.

The assessment of equivalent doses of radiation to the city's population indicates the need to dispose of the accumulated mineral waste. An additional factor necessitating the recycling of accumulated waste is the distribution of radionuclides by soil size classes – the actinoids specific activity in a fine fraction of the soil (particle diameter <100 µm) is close to the value corresponding to very low-level radioactive waste.

The possibility of the actinoids and rare earth elements (REE) concentration with gravitational separation and sieving methods is being considered. The redistribution of target elements in the enrichment process has been studied. The use of a combined approach based on combination of gravitational separation and rock classification methods makes it possible to reduce the waste with a high nuclides content volume by 5 times, concentrating the target elements in a fine fraction of the soil. Monazite and associated minerals (ilmenite, rutile) obtained during the conversion can become raw materials for useful elements (REE and titanium) extraction. Purified soils can be involved in a construction.

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OBTAINING FUNCTIONAL MATERIALS FROM SILICON-CONTAINING TECHNOGENIC WASTE

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An important task of the metallurgical industry is to create technology for extracting valuable components from industrial waste. We used hydrofluoride technology to open silicates included in both red mud and wet magnetic separation tailings to obtain a mixture of simple fluorides, complex fluorometalates and a solution of $(\text{NH}_4)_2\text{SiF}_6$, suitable for the synthesis of amorphous silica.¹ It was shown that the reactions of complex silicate phases $\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$ and $\text{NaAl}_2\text{Si}_2\text{O}_5(\text{OH})_4$ with NH_4HF_2 occur with exothermic effects and are characterized by negative values of ΔG^0_{298} . It was found that when leaching with solutions of 20-30 wt. % NH_4HF_2 , T:L=1:100 and temperature ~95 °C, the degree of silicon extraction reaches at least 98% in 2-3 h.² Materials synthesized on the basis of such highly dispersed silica can be used as pigmenting fillers. It is proposed to obtain cobalt aluminate CoAl_2O_4 in a SiO_2 matrix by impregnating wet silica gel with solutions of aluminum and cobalt salts, followed by evaporation of the suspension to dry salts. The formation of bright blue color CoAl_2O_4 in the products of annealing against the background of SiO_2 was established at temperatures of 800-1050°C. Thus, the temperature of formation of the flower-bearing phase from a stoichiometric mixture of metal formate is reduced to 850°C. In this case, cristobalite crystallization begins at 1200°C, which allows maintaining a high specific surface area of the samples. Increasing the annealing temperature to 1050°C leads to a shift of color coordinates in the CIE 1931 system to a brighter blue region up to $x = 0.158$; $y=0.176$, which allows the use of $\text{CoAl}_2\text{O}_4/\text{SiO}_2$ as a pigment material.

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METALLURGICAL ASSESSMENT OF TECHNOGENIC WASTES FROM FERROMANGANESE PRODUCTION

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In the technological schemes used in modern conditions for the production of manganese ferroalloys from ore to the finished alloy, about 50% of the manganese extracted from the subsoil is lost, a large number of by-products are formed: slag, screenings of small fractions of ore raw materials and finished products, sludge, dust and a number of other materials, the use and processing of which can reduce the consumption of initial mineral raw materials, increase the efficiency of main production, and significantly reduce environmental pollution. The main directions for increasing the end-to-end recovery of manganese are improving technological processes for producing ferroalloys and additional recovery of valuable elements from industrial waste.

Using the methodological approach previously developed by the authors for the assessment of technogenic waste from ferroalloy production, a metallurgical assessment of a number of manganese-containing materials was carried out. Samples of industrial waste from the production of manganese alloys were selected for study: slag, dust and sludge from the smelting of high-carbon ferromanganese grade FMn78 and ferrosilicomanganese grade MnS17. An analysis of the chemical, fractional and phase compositions of the materials was carried out. It has been shown that the most valuable in terms of manganese content are gas purification dusts generated during the smelting of manganese alloys, consisting mainly of the dusty fraction of manganese ore. The method of thermodynamic modeling shows the fundamental possibility of using up to 25% manganese-containing dusts and slags instead of ore materials in the charge, however, when using slag, a decrease in the degree of manganese extraction to 3% is observed. One of the positive aspects of the use of technogenic materials may be a change in the composition of the slag when using different types of ore materials. The most promising direction for using manganese-containing production waste is the possibility of their use in the technological chain for the production of complex ferroalloys.

The work was carried out according to the state assignment of IMET UB RAS.

THE USE OF PbO_2 -BASED ANODES IN THE PRODUCTION OF SODIUM CHLORATES

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PbO_2 -based anodes are low-wear electrodes that are used in electrochemical synthesis as insoluble anode materials¹. The electrochemical synthesis of chlorates in electrolyzers has been studied: with a membrane (MK-44), without a membrane (in the presence of sodium dichromate), without a membrane without sodium dichromate. Experimental conditions: $i = 1 \text{ A/dm}^2$, V anolyte and catholyte = 250 ml, cathode- stainless steel, anode PbO_2/Ti .

Table 1. Comparison of the efficiency of the sodium chlorate production process

Electrolysis	E_{max} , mB	pH (τ_{max})	$\tau_{\text{(мин)}}$	Current efficiency, %
With a membrane	1134,6	2,15	30	20
With sodium dichromate	940	8,75	360	37
Without sodium dichromate	909	8,803	360	11,6

The highest current output (W), 37%, is achieved by electrolysis with sodium dichromate in 360 minutes. In an electrolyzer with a membrane, W is 20% (30 minutes). Figure 1 (a, b) shows the dependences of the electrode potential and pH on the time of electrolysis under various conditions, which allows us to assess the completeness and speed of electrochemical synthesis.

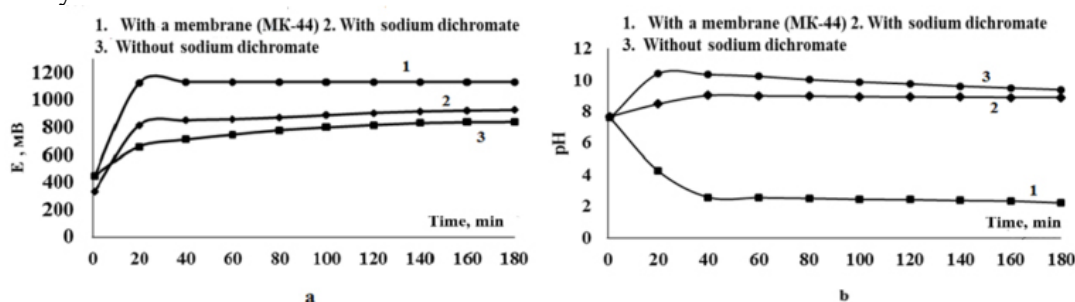


Figure 1. Physico-chemical behavior of $\text{PbO}_2 + \text{TiO}_2$ electrodes (1 g/l)

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SODA PRODUCTION WASTE AS A RAW MATERIAL FOR LOW-TEMPERATURE SYNTHESIS OF WOLLASTONITE

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Waste from chemical plants can pose a serious danger to the environment. One of the multi-tonnage wastes that have a significant impact on the environmental situation is distillery liquid. Existing recycling technologies do not completely solve the problem, and waste often accumulates in sludge accumulators or is dumped into reservoirs.

One of the directions for processing distiller liquid is to obtain from it demanded materials, such as calcium silicates, which include wollastonite, widely used in many areas of industry.

As a result of the work, well-crystallized wollastonite with a low content of impurities was obtained from distiller liquids of JSC Bashkir Soda Company and JSC Crimean Soda Plant using a low-temperature method.

Technological schemes for the production of wollastonite from distiller liquid have been developed, a preliminary economic assessment of the feasibility of such production has been carried out and a preliminary scheme has been developed for the creation of a mobile pilot plant, including 4 units:

1 block – storage of reagents (liquid glass), with the maintenance of the necessary conditions: no interaction with the atmosphere, the optimal temperature is $20 \pm 10^{\circ}\text{C}$;

2 block – is the process zone, which includes: a reactor with a mixing device, an automatic system for supplying liquid stele from block 1 and supplying distiller liquid from a sludge accumulator or production.

3 block and flushing unit, including a filter unit and a vaporizer with a condenser.

4 block – storage of products: wollastonite and sodium chloride. Storage conditions: dry air, temperature $30 \pm 10^{\circ}\text{C}$.

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METHOD OF DISPOSAL OF SULPHIDE-ARSENSIC CAKE

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When processing sulfide copper-zinc concentrates at copper smelters, sulfide-arsenic cakes are formed, which require disposal¹. To solve environmental problems associated with arsenic in the metallurgical and mining industries, it is necessary to reliably concentrate and fix arsenic in process wastes with their subsequent disposal.

Fusion of sulfide-arsenic cake with elemental sulfur (15-20% of the cake mass)² leads to the formation of glassy insoluble forms of arsenic sulfide that are less toxic and compact compared to powder cake. The resulting non-stoichiometric arsenic sulfide has high chemical stability, confirmed by the results of leaching using the TCLP method³.

The structure of the recycled sulfide-arsenic cake was identified. Using thermodynamic modeling methods, the quantity and composition of equilibrium phases were assessed, and the amount of added elemental sulfur additives was optimized. An adequate mathematical model has been constructed to analyze the process of obtaining chemically inert arsenic compounds and to minimize the number of expensive and time-consuming installation experiments. A technological scheme has been proposed for processing cakes by fusion with elemental sulfur to obtain poorly soluble sulfides suitable for long-term storage or disposal in landfills⁴.

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N-DOPING OF CARBONS, PREPARED FROM RICE HUSK ASH

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Adsorbents based on activated carbon are in demand in many fields of science and technology. The search for effective methods for producing and activating coals is an urgent task today¹. On the other hand, there is another pressing problem - the disposal of agricultural waste. For example, during rice production, a waste product remains, rice husk (RH), and its quantity reaches 150 million tons per year². At the same time, rice husk can be used as a raw material for producing activated carbon, which will be a good method for processing this waste.

In this work, we studied coals isolated from rice husk ash after pyrolysis and treated with nitrogen-containing reagents by thermal and hydrothermal methods. The possibility of chemical modification of the surface of carbon and a change in textural characteristics when doping the surface with nitrogen using urea, ammonia and aminoguanidine was investigated. As a result, activated carbons were obtained containing up to 6.8 at. % nitrogen on the surface and having a specific surface area up to 910 m²/g.

The adsorption properties of coals in relation to diclofenac and benzoic acid were also studied. It was shown that the resulting carbons exhibit high adsorption capacity. At the same time, nitrogen doping of coals opens up the possibility of targeted improvement of adsorption characteristics.

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PHYSICOCHEMICAL BASIS FOR THE SEPARATION OF RADIOACTIVE NOBLE METALS

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Radioactive noble metals (RNM - Rh, Ru, Pd, Ag) are found in spent nuclear fuel (SNF) in the form of metallic inclusions - Mo-Pd-Tc-Ru-Rh alloy with actinides and fission products (FP), as well as compounds of non-metallic PD such as palladium iodide¹. During SNF reprocessing in the Purex process RBMs are distributed into two products: metal alloy particles mainly into insoluble precipitates (IP) of the dissolution unit and the rest into the raffinates of the first extraction cycle². The separation of actinides and RBM from LE will reduce radiotoxicity of the waste, prepare the waste for disposal, return actinides to the fuel cycle and, at the expense of short-lived and stable isotopes of rhodium and ruthenium, pay for a part of SNF reprocessing³. Chloride melts are a stable technological medium for heat release, radiolyses, which causes secondary sedimentation. For the development and optimization of noble metal separation processes, data on their physicochemical properties and behavior in working media are required.

In the present work the temperature stability of rhodium and palladium chlorides in LiCl-KCl-CsCl melt up to 923 K was proved by absorption spectroscopy. The temperature of decomposition of ruthenium trichloride in LiCl-KCl-CsCl was determined by the combined method. Temperature dependences of conditional standard potentials of ruthenium, rhodium, palladium and silver, changes of Gibbs energy, enthalpy and entropy of formation of chlorides of these PMs and corresponding activity coefficients in LiCl-KCl-CsCl were determined by the EMF method. The maximum purification coefficients and radioactivity of the final electrolysis products during the intragroup separation of RNMs were estimated.

Processing will make it possible to separate and purify from impurities rhodium and ruthenium which after decay of their short-lived isotopes will have activity less than MSA and will be suitable for various uses.

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COMPLEX SULFATES IN HYDROMETALLURGY OF SCANDIUM

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The development of effective technologies and of promising areas of materials science is necessary for the transition to resource saving. Equally important is the search for promising raw materials of rare metals, including scandium. In order to develop the selective isolation of scandium from multicomponent sulfuric acid solutions, we propose its precipitation in the form of complex sulfates. Crystallization of complex sulfates of rare earth elements (REE) is a classical method of their separation¹. At the same time, scandium, unlike AREA, exhibits some anomaly of their properties and great similarity to metals with a small ionic radius (Fe^{3+} , Al^{3+} , Ti^{4+} , Zr^{4+} et al.). Therefore, in multicomponent solutions, it is possible to manifest the effects of mutual influence of elements, precipitation, co-precipitation and masking of the properties of individual compounds. This behavior explains the need for a detailed study of complex sulfates $\text{MSc}(\text{SO}_4)_2$ and $\text{M}_3\text{Sc}(\text{SO}_4)_3$ in systems $\text{M}^+\text{SO}_4\text{--Sc}_2(\text{SO}_4)_3\text{--H}_2\text{O}$.

For the separation of scandium and associated metals of scandium concentrate obtained from red slurries of alumina production², we have already developed some methods that can be implemented before the separation of pure compounds. The selective precipitation of scandium complexes is based on a decrease in solubility through increase in acidity in the presence of complexing ions. By studying model systems, structural data for some compounds were obtained for the first time, and new optical materials were synthesized on the basis of complex scandium sulfates^{3,4}.

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COMPARISON OF OPTICAL CHARACTERISTICS OF DISSOLVED ORGANIC MATTER EXTRACTS AND SEAWATER OF THE ARCTIC SHELF

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More than 50% of the world's organic matter reserves are concentrated in permafrost sediments in the Arctic region¹. Permafrost degradation leads to the release of dissolved organic matter (DOM) into the Arctic shelf seas, which affects their molecular composition and optical characteristics of DOM.

The aim of the work is to compare the optical characteristics of DPS of seawater of the Arctic shelf and DPS samples isolated by solid-phase extraction (SPE). Water samples (22 pcs.) and DOM samples (22 pcs.) were collected in 2020 during the 82nd expedition of the R/V «Akademik Mstislav Keldysh» in three shelf areas: the Kara Sea, the Laptev Sea and the East Siberian Sea. Optical properties of the samples were studied using fluorescence and UV-visible spectroscopy.

SUVA₂₅₄ values of 0.026 and 0.029 cm*L/mg in the Kara Sea, 0.017-0.022 and 0.017-0.029 cm*L/mg in the Laptev Sea, 0.010-0.013 and 0.010-0.020 cm*L/mg in the East Siberian Sea were obtained for seawater samples and DOM extracts. The ASM₃₅₀ values for the same samples were 4.77 and 4.88 in the Kara Sea, 4.85-5.72 and 4.32-5.52 in the Laptev Sea, 5.28-6.06 and 5.01-5.90 in the East Siberian Sea. The obtained values of the SUVA₂₅₄ parameter decrease in the direction from west to east, which corresponds to a decrease in the content of aromatic structures. An increase in the ASM₃₅₀ value was observed as the influence of freshwater runoff and terrigenous export decreased, reflecting the transition to an autochthonous type of DOM. The asymmetry values for seawater samples exceed the values for DOM extracts (7-20%) for all studied samples, indicating a greater «blue» contribution of protein-like aliphatic structures to the seawater DOM pool.

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REGENERATION OF USED MOTOR OIL WITH SILICA SORBENT

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The growth in production and consumption of lubricants causes a constant increase in the volume of used oils. On the territory of the Russian Federation, about 2 million tons of various waste oils are collected in just one year, while only 3.3% of the total consumption is sent for processing, the rest, due to the lack of an appropriate collection and recovery system, is burned. In order to save resources and reduce the environmental load on the environment, there is an urgent need to create a regeneration technology with the release of valuable oil components.

The purpose of the research is to develop an effective technology for the regeneration of used motor oils using vacuum distillation and sorption purification and to obtain an oil base with physical, chemical and operational characteristics that meets regulatory compliance.

A prototype «Silinom-SORB», developed at the Department of Inorganic Substances and Material Engineering of the Kazan National Research Technological University, was tested as a sorbent. The sorbent is a micronized mesoporous amorphous silica with a specific surface area of 460 m²/g. It was determined that the synthesized sorbent has high adsorption activity, selectivity towards resinous substances and sulfur-containing compounds of waste oils, achieving a deodorizing effect. The total sulfur content in the original waste oil is 0.229% wt., after vacuum distillation it decreases five times to 0.045% wt. and after sorption purification – 0.016% wt. The content of resinous substances also changes significantly, in used oil it is 24.6% wt., in recovered oil it is 0.2% wt.

As a result of the research, it was established that the complex method of regenerating used motor oils using a silica sorbent makes it possible to restore oil quality indicators to the requirements for the I-12A industrial oil grade in accordance with GOST 20799 – 2022 (Industrial oils. Specifications).

PLASMA ARC EJECTOR FOR THE PLASMA TREATMENT OF WASTE

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To solve environmental problems, one relevant solution is to place plasma waste treatment plants on mobile platforms, such as railway wagons and sea and river ships and others. One promising solution to this problem is the use of plasma arc ejector. We have developed a mathematical model for such an ejector.

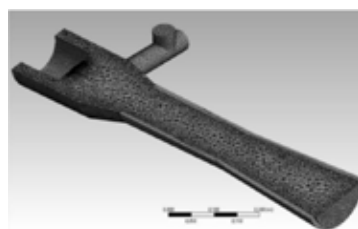


Figure 1. Mesh model of the plasma arc ejector.

The mathematical model of the plasma ejector is based on an analysis of the literature¹ and calculations of the material and heat balance in the plasma ejector. The purpose of the model is to determine the main characteristics of this device.

The main parameters and distributions of temperature, gas velocity, and gas streamlines are calculated using the model. It is planned to design an experimental installation based on the calculations.

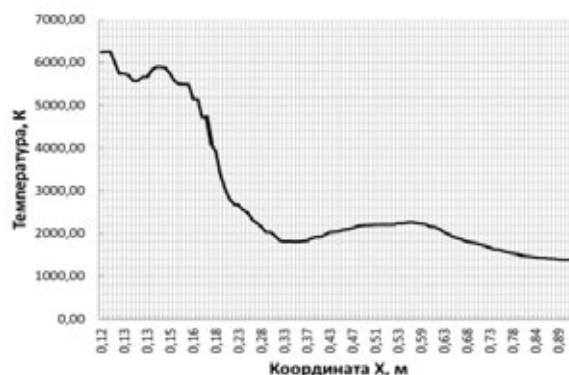


Figure 2. The temperature distribution along the ejector axis.

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SMALL-SIZED MOBILE DUAL-PURPOSE INSTALLATIONS FOR PLASMA WASTE TREATMENT

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Mobile small-sized plasma waste treatment installations with capacities of 1,300 and 6,250 tonnes/year have been developed for operation in both stationary and mobile forms. The units are assembled into one (1,300 tonnes of waste per year) or three (6,250 tonnes of waste per) standard 40-foot containers. The installation shown in Figure 1 is manufactured in the factory.

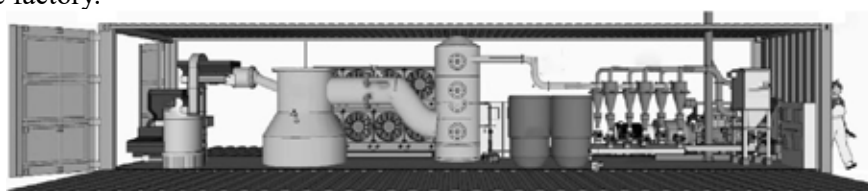


Figure 1. Plasma waste treatment installation with a capacity of 1300 tons of waste per year.

The installation shown in Figure 2 is assembled on site from three containers with equipment manufactured at the factory.

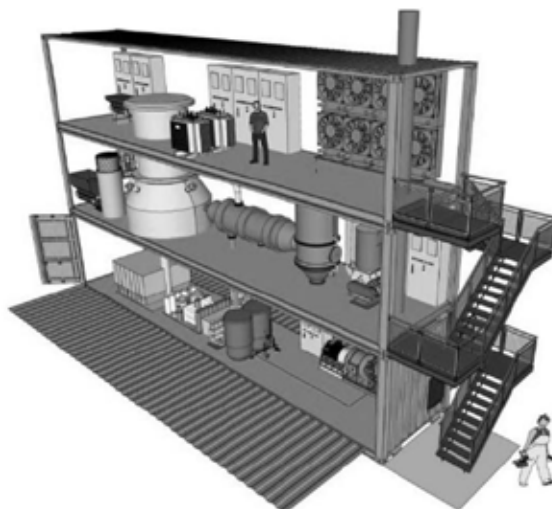


Figure 2. Modular plasma waste treatment installation with a capacity of 6250 tonnes of waste per year.

Mobile and modular plasma waste treatment installations can be used in the civilian sector and on ships and bases of the Russian Navy on the Arctic coast, for the treatment of all types of waste, including hazardous waste.

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2,2,3,3,4,4,4-HEPTAFLUOROBUTYL ACETATE. SYNTHESIS AND PURIFICATION BY REACTION DISTILLATION METHOD

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Fluorinated esters are a widely used product in various industrial fields. At the same time, the classical chemical method of obtaining such substances is characterized by a significant number of process stages and apparatuses, low conversion and yield of the target product, which is the reason for the high cost of these compounds. This classical scheme of the process design can be significantly improved as a result of integration of reaction and separation subsystems in one multifunctional apparatus.

The object of research in the present work is the process of reactive distillation aimed at obtaining heptafluorobutyl acetate, which is used in pharmaceutical aerosol compositions; appears as a copolymer in surface treatment; is used in the production of radiation-sensitive resins, ethyllithium sulfate, charge retention medium (electret), in the production of film external batteries and battery modules (membranes and diaphragm inserts); is a precursor of controlled radical initiators.

At synthesis of heptafluorobutyl acetate the reactive distillation process was based on the esterification reaction of heptafluorobutanol and acetic acid in the presence of acidic catalyst. The process was carried out on a batch distillation column. Such integration allowed to increase significantly the efficiency of the process: thermodynamic constraints on the process of separation of the reaction mixture were overcome, high conversion and product yield were achieved and, quite importantly, the process was carried out by a simplified procedure in one apparatus within one operating cycle.

This study was financially supported by the Russian Science Foundation, project no. 23-79-01164. The research was carried out using the equipment of the JRC PMR IGIC RAS

ELECTROCHEMICAL PROCESSES IN OXIDES IN THE REDUCTION OF METALS IN METALLURGICAL UNITS

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It is known that there are no atoms or molecules in the materials processed in metallurgical units – in the metals themselves, as well as in solid ores and molten slags. In metals and ores there are metal cations and so-called «free» (valence) electrons, and in ores and slags there are the same cations and the same valence electrons of metal atoms, which are largely already bound to anions and provide a bond of the latter with cations. The essence of the transformation of metals into oxide during oxidation by any oxidizer and the reduction of metals from ore by any reducing agent is the redistribution of valence electrons of metal atoms. These processes are described by the electrochemical reactions $\text{Me}^0 = \text{Me}^{2+} + 2\text{e}$ and $\text{Me}^{2+} + 2\text{e} = \text{Me}^0$ respectively. In addition, it is known that with the parameters T and P_{O_2} , existing in reducing units, the gas phase is a low-temperature plasma in which ions and free electrons are present along with molecules and atoms, and oxides have ionic and electronic conductivity.

Based on the concepts of redox reactions as processes of exchange of external («valence») electrons, we have developed an electronic theory and methods for selective non-contact reduction of metals in the volume of solid oxides based on different affinity of cations to an electron¹. According to the electronic theory, an electrochemical reduction reaction can occur inside the solid oxide phase if free electrons enter the cations or the cations drift to the electron source. In the first case, a scattering of particles of non-contaminated reduced metal is formed in the oxide, in the second - a metal shell on the oxide. The results of experimental confirmation of the theory and the possibility of practical use of the results in new technological processes are presented.

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*The research was carried out at the expense of a grant from the Russian Science Foundation No. 23-29-10119,
<https://rscf.ru/en/project/23-29-10119/>*

PROCESSING OF HIGHLY MINERALIZED TECHNOGENIC WASTE

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Currently the system of waste management in the Russian Federation is undergoing significant changes. Major innovations are related exactly to especially dangerous waste of the first and the second hazardous classes¹. A significant amount of similar wastes is a liquid fraction with high mineralization. Examples include spent sulfuric acid solutions, galvanic sludge etc. Currently such wastes are buried or thermally neutralized.

Obviously, this approach does not meet modern waste management requirements. More and more scientific research is being dedicated to obtaining new products from various wastes. The work provides a review of the most perspective areas as well as the research results which obtained for now by scientific workers of MUCTR.

At the present time reverse osmosis (RO) is the most promising technology in the utilization liquid wastes area. This is confirmed by domestic and foreign research². A properly designed RO technology allows you to simultaneously achieve a significant degree of waste concentration and obtain purified water. Highly mineralized RO stream is a promising raw material for obtaining useful products. Synthesis of new useful components is possible using electrochemical technologies.

Currently, a concept diagram of such a technology has been formed, and several experiments are being carried out to obtain useful products, including: peroxodisulfuric acid, hydrogen peroxide³, sodium hydroxide and hypochlorite. The results of the work will find application in general chemical technology and in government projects, for example, in complexes for the processing of highly hazardous waste, designed by the Federal State Unitary Enterprise «FEO».

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EXTRACTION OF LI, FE AND AL FROM SPENT LFP BATTERIES

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The widespread increase in demand for lithium-ion batteries, as well as modern environmental trends in sustainable development, lead to the need to create new technologies for processing chemical current sources, such as lithium iron phosphate batteries (LFP), which are one of the most promising energy sources in electric vehicles of the future.

The hydrometallurgical method has already been successfully used to recycle current sources¹, but the process of complex recycling of LFP active materials has not yet been described. In addition, the use of more environmentally friendly modern extractants – eutectic solvents² – is promising.

In this work, the leaching process of LFP was studied using several mineral acids under varying conditions. The research results showed that the highest degree of leaching (>85%) of Li, Fe and Al is achieved with a 2M HCl solution at room temperature and solid:liquid ratio = 1:25 for 5 hours.

A hydrophobic eutectic solvent tributylphosphine sulfide/menthol (3:7) was proposed for the extraction of Li(I), Fe(III) and Al(III) from chloride leaching solutions of LFP. Its extraction ability to selected metals has been studied. The dependences of the degree of metal extraction on the concentration of HCl and NaCl, the phase ratio and the initial metal concentration have been established, which makes it possible to characterize the mechanism of interphase distribution of metals in the proposed system. Thermodynamic parameters of the extraction process have been established. Simple regeneration of the eutectic solvent and the possibility of its repeated use are shown.

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*This work was financially supported by RSF, project No. 23-79-10275,
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OBTAINING SOILS FOR BIOLOGICAL RECULTIMATION

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An important element of the reclamation of disturbed lands is the stage of biological recultivation¹, the purpose of which is to implement a set of agrotechnical measures aimed at the restoration of flora and fauna. In this regard, it is relevant to develop soils and sub-soils that provide the necessary set of agrochemical indicators. One of the options is soils based on coal combustion residues (CCR), brown and oxidized hard coal². At the same time, oxidized coals are a source of humic substances, which in turn have a positive effect on plant growth, and ash and slag waste is an alkaline reactant for the extraction of humic substances and act as suppliers of such agrochemically important elements as phosphorus, potassium, manganese, etc.

In this work, an assessment of soils of different composition was carried out: soil #1 – 90 mass % ash and 10 mass % of oxidized coal; soil # 2 contained an equal amount of both components. The soils were obtained by mechanochemical activation by ultrasound for 1 hour with the addition of water (in the ratio S: L – 2:1). After activation, the mixture was dried at 80 °C to an air-dry state.

The data showed that an increase in the content of oxidized coal in soil # 2 leads to a decrease in the pH of the aqueous extract to 8.1 (for soil # 2, pH was 10.1). To assess the biological activity of soils, the germination of Vilensky oat seeds was determined on different base mixtures. Potentially fertile mixtures of rocks from loam and quaternary sediments, as well as sand, were used as substrates. It is shown that the introduction of 10% of the soil into the substrates leads to loosening of clay rocks, increases the germination of plants by 2.2 times. The best results were obtained on soil # 2 containing an equal amount of oxidized coal and ash.

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GAS ANALYTICAL SENSOR DEVICES FOR CONTINUOUS ENVIRONMENTAL MONITORING

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To prevent accidents and conduct continuous environmental monitoring in hard-to-reach places, it is necessary to use gas analytical instruments of a new generation. Semiconductor metal oxide gas sensors make it possible to determine hydrogen sulfide, carbon monoxide, nitrogen oxides and other toxic gases even when their concentration is significantly lower than the maximum permissible concentration. However, the need to maintain the operating temperature of the gas-sensitive material at 200-400 °C limits the battery life of the device, as it requires significant power consumption.

Our group uses several approaches to reduce the power consumption of metal oxide gas sensors. The first of them is associated with the synthesis of semiconductor nanomaterials that are sensitive to toxic gases even at low temperatures and do not require heating.

The second approach is associated with the development and fabrication of special dielectric substrates with miniature gas-sensitive element located on a tip of thin console. In 2024, industrial production of such substrates began at the C-Component enterprise.

The third approach involves the use of temperature modulation. Pulsed heating of the sensor solves several important problems. Firstly, increasing the duty cycle (the interval between thermal pulses) leads to significant energy savings. Secondly, non-stationary modes make it possible to identify the individual characteristics of the analyte gas and significantly increase the selectivity of analysis¹. Thirdly, pulsed heating leads in some cases to a significant increase in the sensitivity and stability of the analysis².

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The research was funded by the Russian Science Foundation (project No. 23-23-00329).

EFFICIENCY OF SEPARATION OF ALCOHOL – ESTER SYSTEMS USING DEEP EUTECTIC SOLVENTS

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Deep eutectic solvents (DES) are a breakthrough alternative to ionic liquids and volatile organic solvents in various fields of chemical technology. Recently, DESs have proven themselves as extractants for the separation of azeotropic mixtures. In this work, a set of studies of evaluating the efficiency of separation of various alcohol – ester systems using DES based on choline chloride was carried out. To achieve the goal, the study of the liquid-liquid equilibrium in pseudo-ternary systems alcohol – ester – DES was carried out, and then the distribution coefficients and selectivity values were calculated. Quantitative analysis of equilibrium phases was carried out using ^1H NMR spectroscopy. Figure 1 shows an example of the dependence of the separation selectivity of the 1-butanol – 1-butyl formate system on the alcohol content in the system using 4 different DESs.

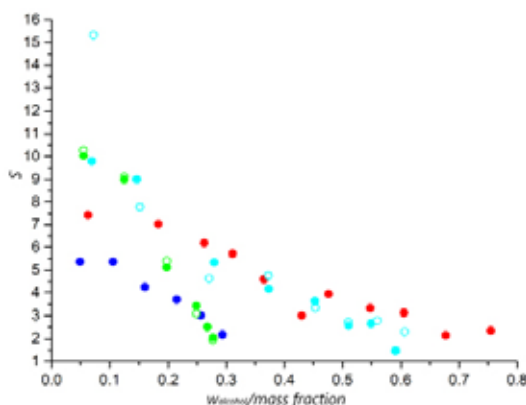


Figure 1. Dependence of selectivity (S) on the mass fraction of alcohol in the system 1-butanol – 1-butyl formate – DES

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INTEGRATED PROCESSING OF METALLURGICAL SLAG WITH HEAT RECOVERY

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Metallurgical slag is a renewable raw material resource for the construction industry, metallurgy, agriculture and other industries. The main processes of slag processing are cooling, crushing, metal recovery and size sorting.

The issues of heat recovery from slag melts, improving the quality of products and reducing dust and gas emissions remain unresolved.

The scientific foundations of the technology and design of equipment samples for the processing of metallurgical slag in a liquid state have been created¹. The technology was sold in China and is widely used at enterprises in Southeast Asia.

The essence of the technology is the supply of melt to moving metal bodies (balls) placed in the grate drum-rotor. In the interspherical space, the melt intensively gives off heat and forms a developed heat exchange surface, which makes it possible to extract and utilize heat with different energy characteristics as the melt cools down².

By changing the nature of the movement of the ball packing, the ratio of the masses of the melt and the balls, the parameters of the cooling media, conditions are created for controlling the cooling process, the type and quality of the resulting product, and the localization of vapor-gas emissions³.

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INFLUENCE OF THE METHOD FOR DETERMINING GAS TRANSPORT CHARACTERISTICS OF MEMBRANE ELEMENTS ON THE RESULTS OF MODELING GAS SEPARATION SYSTEMS

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Today there are many scientific publications devoted to the simulation of membrane gas separation processes, most of which are based on models with a large number of simplifications, namely modeling taking into account only the ideal transport characteristics of membranes. This approach does not allow us to adequately assess the effectiveness and feasibility of developing the proposed technological solutions.

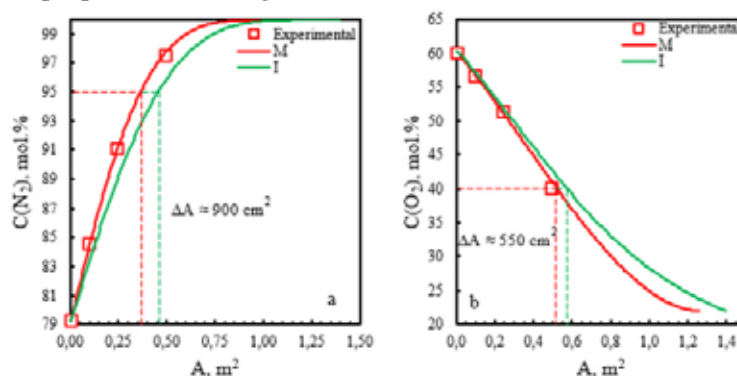


Figure 1. Dependence of concentration (a - N_2 in the retentate flow; b - O_2 in the permeate flow) on the effective area of the PPO membrane over the entire stage-cut range.; M – modeling based on effective gas transport characteristics; I – modeling based on ideal characteristics.

As a result of the study, it was found that when modeling based on ideal gas transport characteristics for pure gases, the difference in the concentration of the target component in the product flow ranges from 1.5 to 8.8% compared to the experimentally obtained values for the same module. This can lead to errors in the design of devices and incorrect technical and economic assessment of the process. When designing technological lines using mathematical modeling tools, it is necessary to use effective gas transport characteristics of the material and/or product.

The work was carried out with financial support from the Ministry of Science and Higher Education of the Russian Federation within the framework of a scientific project under state assignment No. FSSM-2023-0004

CONTINUOUS MEMBRANE COLUMN CASCADE - AN ENERGY-EFFICIENT SOLUTION FOR CARBON DIOXIDE CAPTURE FROM FLUE GASES OF THERMAL POWER PLANTS

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Over the last century, the concentration of carbon dioxide in the atmosphere has increased dramatically from 275 ppm to 387 ppm and has led to a measurable increase in global temperatures, namely, the average surface temperature of the Earth in the 21st century is 0.8-1.2°C higher than in the 20th century. If CO₂ accumulation continues at the current rate, it will exceed 560 ppm by 2060, more than double the pre-industrial level. Developed climate models predict that the established trend will adversely affect the global climate by 2100.

The present work is devoted to a comprehensive modelling of the CO₂ capture process using a membrane cascade of the «Continuous Membrane Column» type on the case of separation of a four-component gas mixture (O₂ / H₂O / CO₂ / N₂ = 4.4 / 11 / 11.6 / 73 mol.%). This membrane cascade includes three membrane units: one unit in the depletion section (produces carbon dioxide depleted stream), two units in the enrichment section (produces CO₂ concentrate).

A multi-parametric analysis established the influence of membrane gas transport characteristics on the membrane area required for each section of the cascade and determined the relationship between the membrane area of each section, CO₂ recovery and purity. Under optimum conditions, the membrane cascade provides efficient separation, capturing more than 90 % of CO₂ with its concentration in the product stream greater than 95 mol.%.

The work was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation within the framework of the scientific project under the state assignment № FSSM-2022-0005

CAVITATION EFFECT ON HYDROBIONTS

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The analytical study of fishery products requires technological methods with minimal use of intermediate stages. A promising way to obtain extracts from natural objects is the use of a rotary pulsation apparatus in which the raw materials are subjected to a cavitation process¹. We have demonstrated a method for studying the biochemical composition of fish products (Pacific herring) using cavitation sample preparation (temperature 40 °C, rotor rotation speed 3000 rpm, exposure time 5 min) and chromato-mass spectrometric measurement (LCMS-8060 Shimadzu spectrometer). Table 1 shows the composition of the lipid fraction of the sample.

Table1. Biochemical composition of the sample.

decryption	polyunsaturated fatty acids	relative intensity 100%		
		free acid	methyl ether	ethanolamide
ALA	18:3 ω 3	280	290	322
SDA	18:4 ω 3	276	287	320
ARA	20:4 ω 6	305	320	350
EPA	20:5 ω 3	300	315	345
Osbond acid	22:5 ω 6	330	350	375
DHA	22:6 ω 3	329	340	370

18:3 ω 3 – α - linoleic acid (ALA). 18:4 ω 3 – stearidonic acid (SDA); arachidonic acid (ARA)
20:5 ω 3 – eicosapentaenoic acid (EPA); 22:5 ω 6 – docosapentaenoic acid (Osbond acid);
22:6 ω 3 – docosahexaenoic acid (DHA)

The data obtained indicate the possibility of measuring biological samples in this way without multi-stage stages of decomposition and extraction.

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INFLUENCE OF HEATING CONDITIONS AND COMPOSITION OF FUEL BLENDS ON PRODUCTS OF THERMAL CONVERSION STAGES

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The production of energy and value-added products from biomass and waste is considered a promising direction for solving a range of environmental and energy problems. A wide range of components can be used for direct combustion, pyrolysis and gasification. Fuel properties, chemical composition, and the presence of impurities affect atomization, mixing with air, evaporation rates, ignition and burnout. One of the problems in the designated scientific and technological area is the low efficiency of the full cycle of use of raw materials, high emissions of hazardous substances, high cost of installations and their maintenance.

This work analyzes the intermediate and final gas products formed during the thermal decomposition and combustion of fuels based on low-grade fuels (brown coal, wood, coal sludge) and liquid components (vegetable oils, waste petroleum oil). The experimental technique included heating small volumes of fuel in a model combustion chamber, ensuring the collection of a gas sample for composition analysis. The results obtained in the experiments confirmed the significant influence of heating temperature and the composition of the carrier medium (air, a mixture of air and nitrogen, water vapor) on the formation of products of complete and partial conversion of feedstock. A non-additive relationship between the concentrations of flue gas components and the component composition of fuel mixtures was obtained, which indicates a synergistic effect in the chemical reaction.

The possibility of reusing intermediate and final reaction products has been shown, and rational conditions have been determined to ensure minimum concentrations of harmful combustion products and ash deposits. The results obtained are the basis for the design of installations for the joint thermal conversion of new types of composite liquid fuels derived from industrial waste, gas hydrate compounds, as well as traditional fossil fuels.

The study was supported by a grant from the Ministry of Science and Higher Education of Russia, Agreement No. 075-15-2024-543 dated April 24, 2024.

THE INFLUENCE OF METAL IONS AND PH OF THE MEDIUM ON THE PROCESS OF HYDROGEN SULFIDE OXIDATION BY IRON SALTS

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Hydrogen sulfide is a common environmental pollutant with high toxicity and an unpleasant odor. Typical sources of its emissions in urban areas are municipal wastewater treatment facilities and oil loading terminals. Reducing the harmful impact of hydrogen sulfide on the environment is possible by oxidizing it with oxygen from the air to elemental sulfur - a solid substance with a low hazard class (IV). Liquid catalysts based on variable-valence metals, most commonly iron, are used for economically viable oxidation processes [1,2]. The aim of the work was to study the mechanism of hydrogen sulfide oxidation and to find ways to accelerate it.

A study was conducted on the kinetics of the reaction of hydrogen sulfide oxidation using catalysts of different compositions. Gas emissions were simulated using a synthetic mixture of air with controlled hydrogen sulfide content. The concentration of hydrogen sulfide in the air was determined using a Chromos GC-1000 gas chromatograph (Russia) with a flame photodetector.

It was shown that at typical emission concentrations, the rate-determining step of the process is the transfer of hydrogen sulfide across the gas-liquid phase boundary. The oxidation rate of dissolved hydrogen sulfide has little influence on the overall process rate. The transfer of hydrogen sulfide across the phase boundary occurs with the formation of HS⁻ and S²⁻ ions. The process of phase boundary transfer is accelerated by increasing the pH of the catalyst solution or by adding transition metal ions that form sulfides with low solubility product, such as Cu²⁺ and Ag⁺. Increasing the concentration of Cu²⁺ ions leads to an increase in the process rate.

The results obtained in laboratory tests were validated on a real wet scrubber installed in the exhaust gas treatment system of a sewage pumping station.

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DESILINIZATION OF MAGNETITE CONCENTRATE BY AMMONIUM HYDROFLUORIDE

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The iron-containing concentrate obtained from ash and slag waste from thermal power plants by wet magnetic separation contains numerous impurities in its composition (Table 1), which complicate its application in ferrous metallurgy.

Table 1. Chemical composition of iron-containing concentrates, K1- obtained by magnetic separation from ash, K 2 desiltered with ammonium hydrofluoride K1

% mac.	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	P ₂ O ₅	SO ₃
K1	0.26	2.36	9.65	23.40	0.50	1.98	0.45	1.41	59.50	0.28	0.21
K2	0.28	3.14	12.30	2.18	0.24	2.49	0.55	1.82	76.24	0.32	0.44

Due to the complex composition of the ferrospheres belonging to the system - FeO–SiO₂–Al₂O₃–CaO–MgO^{1,2}, physical enrichment methods have known limitations. One of the effective methods of opening silicate rocks is based on fluoroammonium technology³. The mechanism of interaction of silicon oxide with ammonium bifluoride can be represented as follows:

$\text{SiO}_2 + 3\text{NH}_4\text{F} \cdot \text{HF} \rightarrow (\text{NH}_4)_2\text{SiF}_6 + \text{NH}_3 + 2\text{H}_2\text{O}$. The ammonium hexafluorosilicate formed as a result of the reaction sublimates upon heating, thereby removing silicon oxide from the system. Fluoridation of concentrate K1 at a temperature of 400°C allows to remove silica from it by 90.6%.

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CHARACTERISTICS OF GAS-DUST WASTE FROM FERROALLOY PRODUCTION

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When manganese, chromium and siliceous ferroalloys are produced in ore-reducing electric furnaces in the Russian Federation, about 1.5 million tons of dust are formed. For the rational use of these materials, it is necessary to know their basic physical, chemical and technological characteristics.

To study the characteristics, industrial samples of dusts formed during the production of: crystalline silicon (CC), 75% ferrosilicon (FeSi75), low and high carbon ferrochrome (LCFeCr and HCFEcr), silico- and ferromanganese (SiMn17 and FeMn 78) were used. The chemical composition of the dusts is shown in Table 1.

Table 1. Chemical composition of dusts, %

Name	Cr ₂ O ₃	MnO	SiO ₂	FeO	CaO	Al ₂ O ₃	MgO	Si _{мет}	C
Dust CC			92,0	0,9	0,4	0,6		0,3	3,3
Dust FeSi75			87,0	3,5	1,1	1,3		0,2	1,3
Dust LCFeCr	26,5		16,5	9,8	21,3	7,5	19,2		0,3
Dust HFeCr	15,5		67,3	7,6	0,8	1,7	7,2		6,5
Dust SiMn17		21,7	29,4	0,6	9,6	5,9			
Dust FeMn 78		45,1	10,6	2,6	7,3	4,6			

CC and FeSi75 dust is a highly siliceous dispersed material – microsilicon. Its mineral phases are represented by β -cristobalite, α - quartz, SiC carbide, Al₂O₃•SiO₂ aluminosilicates, FeO•Al₂O₃ spinel and amorphous silicon.

Fractional composition of microsilicon (microsilica): less than 2 microns is 90.1%, the rest is no more than 15 microns. Gas purification dust during the smelting of high- and low-carbon ferrochrome has a particle size of less than 0.038 mm about 50%, and less than 0.22 mm 90%. The density of LCFeC dust is 3.44 g/cm³, HCFEcr dust is 2.74 g/m³. The melting point of LCFeC dust is 1590-1690°C, HCFEcr dust is 1460-1480°C. The basis of the phase composition of the LCFeC dust is, %: 56.4 spinelides, 23.0 – mervinite CaMg (SiO₄)₂, 12.6 calcium orthosilicate. HCFEcr dust consists of, %: 32.0 spinelide, 55.0 glass phase, 8.0 monticellite.

The composition of the FeMn 78 gas purification dust contains: gausmanite Mn₃O₄, tetragonal manganese oxide, K_{0.7}Na_{0.7}Cl, SiMn17 dust- gausmanite, quartz, K_{0.7}Na_{0.3}Cl. The size of the dust particles of SiMn17 and FeMn78 alloys less than 0.15 mm is up to 50%, and less than 0.54 mm is up to 90%.

The work was performed according to the State assignment of the Institute of Metallurgy of Ural Branch of the Russian Academy of Sciences.

COMPUTATIONAL APPROACH TO ASSESSING COMPLIANCE OF CHEMICAL PROCESSES WITH PRINCIPLES OF GREEN CHEMISTRY

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Modern society is facing an urgent need for the development of innovative high-tech approaches to implement chemical processes and careful planning of syntheses aimed at minimizing negative impact on the environment¹. This paper presents the process of developing the functional part of software for assessing chemical processes using numerical indicators based on the 12 principles of green chemistry. For each principle of green chemistry a mathematical formula has been developed to calculate indicators that allow to compare chemical-technological processes. The algorithms are based on known mass indicators of the process, taking into account specific coefficients that influence the ecological efficiency of the process. The initial formulas were developed taking into account available data from regulatory documents of the Russian Federation, including GOST 32419-2022 «Classification of the Hazard of Chemical Products. General Requirements»². The created mathematical algorithms provide the ability to compare the environmental efficiency and safety of various chemical processes aimed at obtaining the same chemical product.

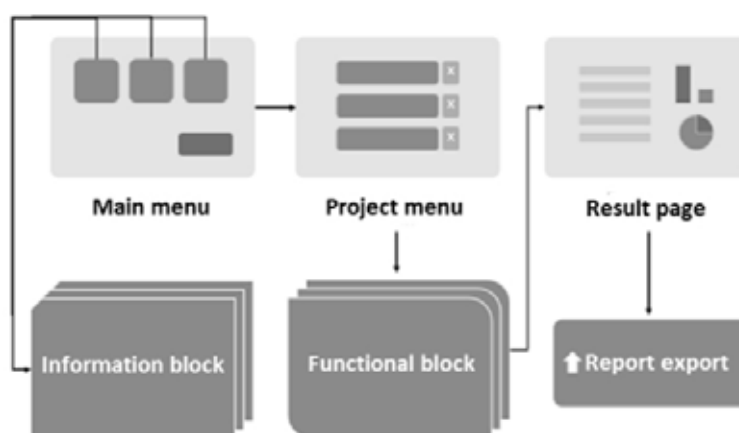


Figure 1. Block diagram of the software functionality

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IMPROVING THE EFFICIENCY OF USING BAKAL SIDERITES IN STEEL METALLURGY

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The high purity of siderites in phosphorus and non-ferrous metals makes them high-quality raw materials for the production of metallized concentrate using coke-free metallurgy methods.

The cost of electricity per scrap melt and metallized siderite concentrate in an electric furnace, containing 30 % of the waste rock and loaded into the furnace at temperatures above 1000 °C, is close. This allows to exclude reduction of the waste rock content in a metallized concentrate using magnetic separation from the siderite processing flow.

A technique including the reduction of the initial siderite ore in a rotating furnace to 95 % degree of metallization and separation melt of the resulting metallized concentrate, hot loaded (at temperatures above 1000 °C) into a furnace is proposed. The resulting liquid metal can be refined in a ladle furnace or, after casting, used as semi-finished metal to replace scrap.

The empty rock of the metallized siderite concentrate contains a large amount of magnesium oxide, which makes it refractory. To obtain liquid slag, it is proposed to use an additive of boric anhydride in the form of colemanite.

The addition of 60-120 kg of colemanite per 1 ton of concentrate makes it possible to obtain slag having a low viscosity (less than 3.65 P) at the tapping temperature (1600 °C).

The work was carried out according to the state assignment for IMET UB RAS using equipment of the Collaborative usage centre «Ural-M».

METALLOTHERMY AS A METHOD FOR PYROMETALLURGICAL ENRICHMENT OF POOR RARE-METAL RAW MATERIALS

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The need to develop the production of rare metal products using domestic natural resources is driven by the demand for niobium and tantalum in various industries. There is currently no viable technology for extracting tantalum and niobium that meets the economic and environmental criteria for processing low-grade ore and industrial waste. The proposed solution in this work is based on the application of pyrometallurgical enrichment of low-grade concentrates and metal wastes. This approach increases the integrated processing and extraction of target metals.

Using the example of poor tantalum concentrates from Orlovskoye and Etykinskoye deposits ($\sim 3.5\% \text{ Nb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$) and Ta-W cakes of rhenium leaching from oxide-metallic wastes, the regime parameters were optimized and technological parameters of metallothermic concentration of niobium and tantalum into iron-based alloy were evaluated. Aluminium or calcium-aluminium master alloy¹ were used as reducing agents. The experimental results demonstrated the possibility of enriching and selecting metals during the reduction of tin-rare-metal Etykinskiy concentrate. Refractory rare metals were transferred into an iron-based alloy, while tin was converted into sublimate at a temperature of 1700-1750 °C with an aluminum consumption of 8.5 %, CaO of 30 %, and Fe_2O_3 of 9 % of the concentrate mass. High extraction rates (99.9 % Ta and 97.7 % Nb) of metals were obtained by reductive smelting with both Al and Ca-Al master alloy. Under optimum conditions, the distribution coefficients of tantalum and niobium between the alloy and slag during the aluminothermic reduction of Orlovskiy concentrate were 71 and 135, respectively. The degrees of extraction into the alloy were 93 % for tantalum and 87 % for niobium. During the smelting process of technogenic raw materials, specifically Ta-W cake, nearly all tungsten, 95.7 % of molybdenum, and 92.5 % of tantalum were successfully transferred into the iron alloy. It is important to note that all zirconium remained in the slag.

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The work was carried out according to the state assignment for IMET UB RAS using equipment of the Collaborative usage centre «Ural-M».

ALTERNATIVE TYPE OF FLUX USED IN THE COURSE OF PELLET PRODUCTION FOR BLAST FURNACE AND METALLIZATION PROCESS

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The Waelz zinc oxide production process, while using a gas treatment dust trapped in the EAF shops, is feasible in the rotary kilns. Meanwhile, zinc and other volatile metals are sublimated to a gaseous phase, off-taken by gas flow from the operating space of a thermal unit and captured in the gas treatment units. A zinc-free solid material discharged from a rotary kiln is represented as a clinker, which contains the iron (30...40%) in the form of oxides, calcium and magnesium oxides (25...35%), silica and residual content of zinc (up to 0.6%) and carbon (up to 7%). The chemical composition of clinker is deemed a promising material to be used as a fluxing agent in the production of oxidized pellets with partial or complete substitution of conventional flux (limestone)¹.

Lab-scale and pilot tests have been conducted with different content of clinker in the pellet feed for versatile applications, as follows: blast furnace and metallization furnace of a solid-phase reduction with determination of properties, in particular physical-chemical and metallurgical. The concentrates of Mikhailovsky GOK JSC (Zheleznogorsk town) and Lebedinsky GOK JSC (Gubkin town) were used as the main source of iron ore feed.

The results obtained lead to the following conclusions:

the content of clinker in the blend as part of Waelz process may reach

- - up to 75% for blast furnace pellets;
- - up to 50% for DR pellets.

Process and heat engineering calculations revealed an efficiency of clinker addition in the pellet blend, namely:

- reduction of CO₂ emissions (reduction of carbon footprint);
- reduction of natural gas consumption;
- reduction of concentrate consumption per 1 ton of fired pellets;
- ramp up of induration machine capacity.

Reference list

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SORPTION PROPERTIES OF TRICALCIUM PHOSPHATE IN RELATION TO Sr(II) IONS

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The sorption behavior of $\text{Ca}_3(\text{PO}_4)_2$ (β -TCP) in aqueous solutions containing Sr(II) traces was studied. The influence of pH, Sr(II) concentration, humic acid and phosphate anions on sorption was determined. Before the experimental study, thermodynamic modeling of β -TCP solubility was performed, which showed that, depending on the solution pH, the $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ and $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ (hydroxyapatite) are the main equilibrium phases. The stoichiometric Ca/P ratio in solution is consistent with the presence of these phases, however, analysis of the sorbent after contact with the solution showed the presence of only the $\text{Ca}_3(\text{PO}_4)_2$ phase. The assumption was made that the established equilibrium refers not to the bulk of the initial β -TCP grains but to a certain small surface layer.¹

A comparison of the sorption isotherm with the change in phosphate solubility in terms of Ca, P ions showed that the interaction with the sorbent surface occurs in the form of a phosphate complex. The results obtained in the Henry region ($<5 \mu\text{mol/L}$) were interpreted according to the mechanism of surface complexation: formation of an electroneutral strontium complex SrHPO_4^0 and its absorption by negatively charged phosphate groups ($\equiv\text{P}-\text{O}-$). It was found that in this region, tricalcium phosphate is able to extract Sr(II) from aqueous solutions with K_d up to $2 \cdot 10^3 \text{ mL/g}$ in the pH range 7–11. As the concentration of Sr(II) increases above $10 \mu\text{mol/L}$, the transition to ion-exchange mechanism of Ca(II)–Sr(II) substitution in the solid phase begins with a drop in K_d to $2 \cdot 10^2 \text{ mL/g}$. The presence of humic acids up to a concentration of $\sim 150 \text{ mg/L}$ does not affect sorption.² Introduction of additional phosphate ions into the solution significantly changes the character of pH– K_d dependence and the mechanism of β -TCP dissolution due to the disappearance of hydroxyapatite on the $\text{Ca}_3(\text{PO}_4)_2$ surface. The results of the study will find application in biology, geochemistry, agrochemistry, in particular in the decontamination of soils and agricultural lands.

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The research was carried out according to the 2023-2028 plans of ISSC UB RAS.

STUDY OF PHASE EQUILIBRIUM IN THE ALCOHOL-ESTER SYSTEM WITH THE DEEP EUTECTIC SOLVENT BASED ON CHOLINE CHLORIDE

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The problem of separating esters and alcohols is quite common in industrial processes involving an esterification reaction, since these two components can form azeotropes. Currently, organic solvents are used to separate such mixtures. However, modern trends in eliminating the use of toxic and aggressive solvents and reagents lead to the need to use more environmentally friendly reagents. Therefore, the main directions of research recently have been focused on the search for new effective environmentally friendly solvents and extractants for the separation of homogeneous mixtures. One of the results of such research was the introduction into chemical practice a new type of solvent - deep eutectic solvents. These solvents are made from natural components such as choline chloride, citric, tartaric, succinic, oxalic acids, glycerin, urea, sugars, and are currently positioned as environmentally friendly analogues of organic solvents and ionic liquids. It is believed that the formation of a eutectic solvent occurs due to the formation of a hydrogen bond between a hydrogen donor and a hydrogen acceptor, which in turn causes a decrease in the melting point of the final solvent and makes it liquid at room temperature.

In this work, a synthesis of a deep eutectic solvent based on choline chloride and glycerol was carried out. The liquid-liquid equilibrium data at different temperatures (293.15, 313.15 K) for the n-butanol - n-propyl acetate - DES (choline chloride and glycerol), n-butanol - ethyl acetate - DES (choline chloride and glycerol), n-butanol - n-propyl propionate - DES (choline chloride and glycerol), n-butanol - ethyl propionate - DES (choline chloride and glycerol) was generated. Based on experimental data, the distribution coefficients of alcohol and ester, as well as the selectivity of alcohol extraction, were calculated.

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SYNERGETIC INFLUENCE OF POLYSACCHARIDES AND ELECTROMAGNETIC FIELD ON THE PROCESS OF CALCIUM CARBONATE CRYSTALLISATION

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Undesirable crystallisation of CaCO_3 is a complicating factor in production processes of oil and gas production [1,2]. Inhibitors mainly phosphonate, carboxylate and sulfonate compounds are used to prevent crystallisation and salt deposition. In this work, the combined effect of modified polysaccharides and electromagnetic field (EMF) on CaCO_3 crystallisation was investigated. Among a large group of monosaccharides, disaccharides, oligosaccharides, polysaccharides and their carboxy- and sulfo- derivatives, arabinogalactan functionalized with phosphate groups and Na-carboxymethylcellulose (NaCMC) showed high inhibitory efficacy on calcium carbonate crystallisation (more than 90% at non-stoichiometric dosages). At simultaneous exposure of polysaccharides and EMF, a synergetic effect was observed, manifested in the reduction of the size of the formed crystals (d) and the shift of the distribution interval to the nanoscale region and the preferential formation of vaterite and aragonite.

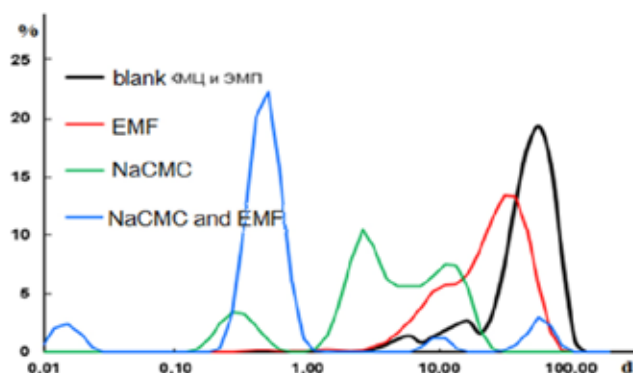


Fig.1. Effect of simultaneous EMF and NaCMC on the size distribution of CaCO_3 particles.

The mechanism of mutual influence of EMF and polysaccharides on crystallisation is proposed.

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TECHNICAL CHARACTERISTICS OF MIXED FUELS BASED ON BROWN COAL AND FIR MEAL

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The energy sector is of strategic importance in every country and has an important impact on economic and sustainable development.

The main purpose of geothermal energy is to replace traditional fossil fuels, whose reserves are being depleted, and combustion causes environmental pollution¹.

The use of geothermal thermal energy sets special conditions for drilling wells, developing fields and constructing power supply systems. Electrical energy is generated at single-circuit direct cycle stations with a geothermal source temperature of more than 160 °C. Binary circuit stations can generate electrical energy using a geothermal source with a lower temperature.

More than 60 deposits of hydrothermal springs with water temperatures from 40 °C to 250 °C have been explored in Russia, the predicted reserves of which are about 20 million m³/day, which corresponds to the combustion of 40 million tons of fuel equivalent. per year². Hydrogen sulfide, nitrogen, nitrogen-carbon dioxide, hydrogen sulfide-carbon dioxide, carbon dioxide, methane and nitrogen-methane waters are found in these deposits².

The purpose of the work is to establish the possibility of testing a pilot installation on an operating well. A basic thermal diagram of a GeoPP with an organic Rankine cycle (ORC) with a power of 25 kW has been developed.

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The work was carried out within the framework of the transport hub development program «Priority-2030-EB-016-202-2024».

TECHNICAL CHARACTERISTICS OF MIXED FUELS BASED ON BROWN COAL AND FIR MEAL

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Enterprises involved in the production of useful cosmetic products from fir wood are faced with a significant problem in the disposal of production waste. The solution to useful energy-efficient processing of these wastes is an urgent task for enterprises working in the field of biotechnology. One of the possible solutions to this problem could be the joint utilization of waste biomass (fir meal) together with coal. The fuel preparation technique is given in work¹. Technical analysis of mixed fuels was carried out in accordance with well-known GOSTs¹. Table 1 shows the results of the study of mixed fuels.

Table 1. Complete technical analysis of mixed fuel compositions based on coal and meal after its processing

Samples	Indicators Name						
	Schrot Org.	Brown coal	B/Sho 90/10	B/Sho 85/15	B/Sho 80/20	B/Sho 75/25	B/Sho 50/50
W ^a	5,3	13,7	8,24	8,0	8,04	7,73	7,24
A ^a , %	3,9	2,96	3,43	3,38	3,51	3,48	3,68
V ^{daf} , %	81,1	46,66	48,27	49,52	51,87	53,07	62,02
Q ^{daf} _{fir}	22,19	29,07	28,71	28,21	27,99	27,87	25,92
Coke residue	Powdered						

Analysis of experimental studies has shown that mixed fuels obtained by adding meal to coal are suitable for combustion in low-power power boilers.

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HYDROGEN-GENERATING SYSTEM BASED ON ALUMINUM AND INORGANIC SALTS

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Aluminum is a promising energy carrier, with high energy density and stability, which allows it to maintain its energy properties for a long time. However, the main disadvantages of using it as an energy carrier remain the complexity of its activation, the selection and use of expensive catalysts, as well as the difficulty in the technological process of the oxidation of aluminum with water.

A hydrogen-generating system has been developed to activate aluminum of any type under normal conditions by introducing electrolytes - inorganic salts (sodium chloride and copper chloride).

Based on experimental data, the main characteristics of the hydrogen-generating system are shown: degree of extraction - more than 90%; the average rate of hydrogen release is ~ 100-150 ml/min. The option of gradually adding an electrolyte solution at a rate of 1 ml per 30 seconds was considered, which showed the possibility of producing hydrogen in portions while maintaining the total amount. An option for scaling this hydrogen-generating composition has been considered, showing successful results without losing the quantitative characteristics of the system.

All of this leads to the conclusion about the effectiveness of using the pro-posed system as a hydrogen-generating composition and the wide possibilities of its use as parts of autonomous power sources, in particular portable mobile power plants.

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EXTRACTION IN NEODYMIUM MAGNET UTILIZATION PROCESSES

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Neodymium magnets are currently the most powerful and commercially available. They are widely used in a variety of industries ranging from household appliances and medical equipment to industrial wind turbines and wind turbines, hybrid electric motor vehicles and laser equipment¹⁻³.

The amount of waste from the production of permanent neodymium magnets is 15-40% of the mass of the magnets themselves and this is a huge loss of valuable raw materials. In addition, NdFeB magnets that have reached their end of life are a potential secondary resource of strategic rare earth elements. Even though in recent years, methods of recycling neodymium magnets have been actively developed, they are not yet applied in practice.

In the present work, a hydrometallurgical method of processing neodymium magnets using selective hydrophobic deep eutectic solvents based on organophosphorus compounds is proposed. The conditions of quantitative acid leaching of metal ions from neodymium magnets have been determined. Physicochemical regularities of extraction of a number of rare-earth elements (Nd, Pr, Dy, Tb) from acidic solutions by the proposed deep eutectic solvents have been established. The prospectivity of application of the extraction method for processing of neodymium magnets is shown.

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PYROMETALLURGICAL PROCESSING OF INDUSTRIAL WASTE FOR METAL EXTRACTION AND PRODUCTION OF CONSTRUCTION MATERIALS

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In Russia, there is a large accumulation of various iron-containing industrial waste, including over 600 million tons of red mud, 1.5 billion tons of fly ash from thermal power plants, tens of millions of tons of dust and sludge from blast furnace production, over 140 million tons of copper slag, and a significant amount of spent catalysts. With the introduction of recycling technology in the Russian Federation, such as the Waelz process for recycling dust from electric arc furnace steelmaking, up to 100,000 tons of Waelz clinker with a high iron content is produced annually. The accumulation of these wastes poses significant harm to human health and the environment, and significant resources are spent annually on the maintenance of landfills and sludge storage facilities. Moreover, these wastes contain valuable elements such as Fe, Al, Ti, Cu, Zn, and rare earth elements (REE).

Currently proposed technologies for processing such waste primarily involve extracting iron from them using pyrometallurgical methods in the initial stage, followed by extracting the remaining valuable elements from the resulting tails or slag using hydrometallurgical methods. While these schemes allow for the production of a large quantity of valuable products, they are also characterized by the complexity of implementation, the need for a large initial investment volume, limited market demand for the produced products, and environmental consequences due to the formation of spent acid and alkali solutions..

An alternative approach is to utilize such waste to produce construction materials alongside metallic products. These processes require lower initial costs as they can be implemented directly in the smelting unit in a single stage. Moreover, the ability to control the cooling rate of the resulting slag allows for the regulation of the properties of the construction materials.

This report presents a review of the works of both foreign and domestic researchers on this issue, as well as developments by the IMET RAS in this field.

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EXTRACTION RECYCLING OF LFP BATTERIES USING HYDROPHOBIC EUTECTIC SOLVENTS

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The hydrometallurgical approach is the most relevant for recycling active materials of spent lithium-iron-phosphate batteries due to its advantages: high recovery efficiency, low energy consumption, negligible emission of harmful gases and low capital costs¹. Recycling used LFP batteries will help to fill the shortage of valuable cells, reduce the exploitation of natural resources, reduce the negative impact on the environment and ensure sustainable development of human society. To date, there is limited research on the recycling of spent LFP batteries, and the technologies developed are still in their early stages^{2,3}.

In the present work, solutions are proposed for the problems of isolation and separation of metal ions, which are the main ones in the composition of the cathode and anode of LFP batteries, from aqueous media using hydrophobic eutectic solvents. Optimal conditions for complete leaching of metals from active materials were determined. Physicochemical foundations of Li(I), Cu(I), Fe(III) and Al(III) extraction with hydrophobic eutectic solvents based on organophosphorus reagents have been developed. The possibility of regeneration and reuse of eutectic solvents has been established. The prospects of using the proposed solutions for the creation of modern closed-cycle technologies are shown.

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An abstract visualization of a complex molecular structure, possibly a protein or a large organic molecule, rendered in a dark blue and purple color scheme. The structure is composed of numerous small, glowing spheres (atoms) connected by lines (bonds), forming a dense, interconnected network. The overall shape is irregular and complex, with many protrusions and recessions. The background is a deep, dark blue, making the glowing structure stand out.

SECTION 5

CHEMISTRY OF FOSSIL AND RENEWABLE HYDROCARBON RESOURCES

NEW NANO-SIZED CATALYSTS FOR THE CONVERSION OF TERPENOIDS INTO PRODUCTS FOR VARIOUS FUNCTIONAL PURPOSE

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Natural halloysite nanotubes (HNT) are the basis for the creation of novel catalysts for the conversion of renewable terpenoids into high-value products [1]. Thus, in the presence of HNT modified with hydrochloric acid, the condensation of terpenoids (-)-isopulegol and *p*-mentha-1,8-diene-5,6-diol with a number of carbonyl compounds occurs with highly selective (up to 90.0%) formation of substituted chromenols, which have high analgesic activity [2, 3]. The reaction of 2-carene with anisaldehyde on HNT leads to isobenzofuran compounds (71.0%), which exhibit neuroprotective properties [4]. Commercial montmorillonite K-10 effectively catalyzes the cascade Prins-Friedel-Crafts condensation of 4-hydroxymethyl-2-carene with aromatic aldehydes into polycyclic compounds with cytostatic activity [5]. It has been established that SO₃H-functionalized halloysite and carbon nanotubes, as well as biochar, are effective catalysts for the Prins-Ritter reaction for the synthesis of bioactive amides based on (-)-isopulegol [6].

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FUNDAMENTAL FEATURES OF THE IGNITION OF METHANE- AND HYDROGEN-CONTAINING GASES AND THEIR EFFECT ON THE KNOCK RESISTANCE OF GAS FUELS

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The observed climate changes and their supposed connection with anthropogenic CO₂ emission are stimulating the wider use of low-carbon fuels such as natural gas and hydrogen-containing gases – methane-hydrogen mixtures, syn-gas.¹ The most common range of initial conditions for their use in energy (GT) and transport (ICE) $T = 500\text{--}900\text{ K}$ and $P = 1\text{--}40\text{ atm}$, which is practically inaccessible to the most popular method of studying high-temperature reactions – the shock tube method. But it is in this range that serious changes occur in the oxidation mechanisms of both methane and hydrogen, associated with a change in the role of peroxide compounds in them,² seriously complicating the prediction of the parameters of their ignition and knock resistance.

In this temperature range below 900 K, which is practically important not only for optimizing the operating modes of power plants and automotive internal combustion engines using methane, hydrogen and gases containing them, but also for ensuring the safety of working with them, a number of unexpected and paradoxical phenomena are observed. Such a critical parameter determining the propensity of methane-hydrogen mixtures to ignite, as the activation energy of their ignition delay, near 900 K can vary 4–5 times. Addition of H₂ can inhibit methane ignition, and addition of CO can promote hydrogen ignition.

The sharp difference between the effect of hydrogen and the heavier alkanes inevitably present in real natural gas on the ignition of methane³ greatly complicates the problem of determining the knock resistance of gas motor fuels, making such widespread characteristic, as the methane number (MN), inapplicable.

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PROGRESS IN HYDROPROCESSING CATALYSTS FOR OIL REFINING

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Hydrotreating and hydrocracking are among the key processes in the petroleum refining industry. In the medium term, the global capacity of hydrotreating and hydrocracking units will increase, which in turn will determine the growing demand for catalysts for these processes. Tightening requirements for the characteristics of petroleum products, along with the changing feedstock base of oil refining processes, in turn, determines the need to research and development of new, more advanced catalysts.

The report provides an analysis of research in the field of catalysts for hydrotreating and hydrocracking of petroleum fractions, presents new research and promising directions for the development of hydroprocessing catalysts. Among the trends that will determine the further development of hydroprocessing catalysts, one can highlight a change in the feedstock base due to heavier crude oils, an increase in the share of distillates of secondary processes and an increase in the share of feedstocks of non-petroleum origin (pyrolysis products of biomass, plastics, etc.), as well as changes in the demand for various petroleum products.

POTENTIAL OF BIOMASS AND POLYMER WASTE FOR THE PRODUCTION OF FUELS AND BASIC PETROCHEMICAL PRODUCTS

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One of the directions of the Russian Federation's Scientific and Technological Development Strategy for the transition to environmentally friendly and resource-saving energy is the creation of closed carbon cycle technologies, implying a zero balance of carbon consumed and released into the atmosphere. The transition to an economy of this kind is possible due to the gradual abandonment of fossil energy sources simultaneously with the involvement in the processing of renewable raw materials and carbon-containing waste.

One of the possible substitutes for fossil raw materials can be biomass of various kinds. It contains carbon, hydrogen, and oxygen; accordingly, it can potentially be used for the production of fuels and chemical products. The main advantages of using biomass as an energy source and chemical raw materials are the huge amount of available resources, their rapid reproduction and the absence of influence on the carbon balance in the biosphere.

Despite the development of a number of biofuel production processes, the main task the conversion of lignocellulose biomass into motor fuels and raw materials for petrochemistry is far from complete due to the complexity of the full deoxygenation of biomass and derived products. The most promising is an integrated approach based on partial hydrogenation of biomass derived products followed by catalytic cracking or hydroconversion. A significant feature of this approach is the conversion of joint feed, containing biooil and conventional oil fractions, which allows for the full hydrodeoxygenation of bio-oil under mild conditions.

Polymer waste is another potential type of carbon-containing feedstock. Considering that a significant part of polymers is used in packaging, with the current volume of production and consumption of plastics in the modern world, billions of tons of synthetic polymer waste have already accumulated, and this amount is continuously increasing. Deep chemical processing of such waste is one of the global challenges for world chemical science and industry. At the same time, it is obvious that such processing can become the basis of a highly profitable industry if the methods and processes used for this are not only effective, but also environmentally friendly, meeting the strict requirements of "green chemistry".

HYDROFORMYLATION: NOVEL CATALYSTS AND APPROACHES TO REALISATION

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Hydroformylation (HF) – the interaction between syngas and olefins with the formation of aldehydes – underlies industrial processes for the production of plasticizers, surfactants, solvents, components of medicals and cosmetic products. Industrial HF uses predominantly homogeneous catalytic systems containing cobalt or rhodium hydridocarbonyls¹. The most relevant scientific problems associated with HF are the difficulty of separation of an expensive catalyst from the reaction products, the toxicity of some components of catalytic systems (CO and phosphine ligands), and the need for additional stages to obtain valuable products from aldehydes, such as primary alcohols, acetals, and amines. Our research group conducts the investigations in these areas. Easily separated biphasic catalytic systems have been developed for tandem reactions of reductive HF, hydroaminomethylation² and HF-acetalization³. Such systems make it possible to obtain products with higher added value in one technological step. All the systems are shown to be reusable. Interesting results have been achieved in the development of solid HF catalysts based on polymers⁴ and hybrid polymer-silicate materials⁵, including those active in reductive HF⁶. In collaboration with colleagues from Southern Federal University and the Kurchatov Institute, our group is engaged in *in situ* studies using synchrotron radiation aimed at the investigation of the state of active metal under conditions of tandem reactions⁷. Also, we study the possibility of using alternative syngas sources for HF reaction⁸.

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CONTRIBUTION OF RUSSIAN SCIENTISTS TO THE DEVELOPMENT OIL REFINING TECHNOLOGIES

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After the withdrawal of leading foreign technology companies from the Russian market, domestic scientists and specialists faced the most important task of maintaining Western technologies existing at refineries and replacing them with Russian analogues. What has changed in two years? Firstly, the transition to Chinese catalysts, additives, and equipment has been observed. Secondly, ample opportunities for the implementation of Russian developments have arisen.

Many Russian technologies have long been mastered at refineries. These are, for example, primary processing of crude oil, isomerization of light hydrocarbons C_5 - C_6 , catalytic cracking of vacuum gasoil, delayed coking of tar, sulfuric acid alkylation of isobutane with butylenes. An almost complete line of Russian-made refining catalysts is available to refineries, with the exception of hydrocracking catalysts, reforming with a moving bed of catalyst, and hydrotreating gasoline obtained from catalytic cracking.

New technologies of leading Russian scientists are of great interest. First of all, these are hydroconversion of tar, alkylation on zeolites of the IPS Academy of Sciences of the Russian Federation, production of needle coke and pitch (developed by the Ufa State Petroleum Technical University together with the Gubkin University), catalysts for hydrotreating and hydrocracking of LLC Rosneft Research and Development Center, Russian Scientific Research Institute for Oil Refining and Federal Research Center “Institute of Catalysis of the Siberian Branch of the Russian Academy of Sciences”, isomerization of C_7 , LLC “Scientific and Production Enterprise Neftekhim”, reforming of Federal Research Center “Institute of Catalysis of the Siberian Branch of the Russian Academy of Sciences” and Gubkin University. Significant achievements have been made in the field of Russian catalysts. New technological solutions of Rosneft scientific Institutes have made it possible to take leading positions in such processes as hydrotreating, hydrocracking, and catalytic cracking, where today Russian catalysts are actively replacing foreign ones. Full-scale industrial tests confirm Russian developments. The main problems today remain with Russian hydrocracking technologies. At this difficult time, it is necessary for the entire Russian scientific community to stand together to produce joint developments and introduce modern Russian technologies for the processing of petroleum.

PRODUCTION OF SYNTHETIC HYDROCARBONS FOR FUEL AND PETROCHEMICAL PURPOSES FROM BIOMASS

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Biomass is currently considered as a renewable raw material for the production of thermal and electrical energy. However, as is known, the areas of application of biomass are not limited to energy.

First of all, these are processes that allow deep processing of carbon-containing raw materials using the principles of “green” chemistry, including «carbon footprint» reduction processes: synthesis gas conversion into a range of products for fuel and petrochemical purposes.

At the TIPS RAS systematic research is being carried out on the processing of plant biomass by hydrothermal carbonization into biochar - an intermediate product for the production of synthetic hydrocarbons. This can be in one stage and through the stage of synthesis gas producing by gasification.

The possibility of obtaining environmentally friendly fuel components by the method of hydrogenation catalytic liquefaction of biochar and by the Fischer-Tropsch method from synthesis gas of gasification of biochars of different nature has been shown.

At the Institute of Petrochemical Synthesis of the Russian Academy of Sciences, research is underway to reduce the oxygen content in biomass conversion products for their further use in road construction in the form of modifiers for traditional brands of bitumen or even individual biobitumens.

The most suitable type of deoxygenated biomass liquefaction product is synthetic oil (bio-oil), obtained by thermal dissolution of biochar, that was produced by hydrothermal carbonization of biomass to minimize the oxygen content.

Such studies are completely new, since they have practically no analogues described in the world scientific literature.

The work has been carried out in frames of the State Program of TIPS RAS.

NEW CARBONATION-RESISTANT CATALYSTS FOR THE DRY REFORMING OF METHANE TO SYNTHESIS GAS

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Dry reforming of methane (DRM) is a catalytic process that allows efficient utilization of greenhouse gases – CO₂ and methane to produce an equimolar mixture of CO and H₂ (synthesis gas). Synthesis gas of this composition is a convenient intermediate for the Fischer-Tropsch synthesis of hydrocarbons and a number of oxygenates, and can also be used in various fuel cells. The catalysts of the DRM process also make it possible to obtain synthesis gas from biogas, other products of biomass processing, and waste. However, the DRM process is complicated by high endothermicity, the formation of coke deposits and the “sintering” of active catalyst centers. The implementation of the DRM process requires the creation of stable and carbonation-resistant catalysts. The approaches we have developed to create such catalysts include: synthesis of catalysts containing nickel and/or cobalt dispersed in a matrix of oxide carriers with a developed surface (zeolites, alumomagnesium oxides, REE oxides)¹⁻³ or perovskite-like complex oxides of cobalt and REE dispersed in a silicon carbide matrix⁴. These catalytic materials ensure the quantitative formation of synthesis gas for at least 50 hours of DRM, and are practically not subject to carbonation and “sintering” of the formed nanoscale metal active centers.

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CATALYTIC TRANSFORMATION OF PLANT BIOMASS COMPONENTS IN THE PRESENCE OF BIFUNCTIONAL SYSTEMS

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Conversion of lignocellulose biomass into value-added chemicals and components of liquid fuels requires an integrated approach, including the development of effective methods of catalytic transformation of biopolymers such as cellulose, hemicellulose and inulin. Among the “platform compounds” obtained by processing plant biomass, levulinic acid (LA) and the product of catalytic hydrogenation of LA – gamma-valerolactone (GVL), as well as furan compounds (furfural (FF) and 5-hydroxymethylfurfural) are of special interest.

The processes of transformation of plant polysaccharides, as well as the production of GVL and furan compounds, take place in the presence of heterogeneous catalysts, which should contain active centers of various natures to ensure high efficiency. In the framework of this work, it was shown that bifunctional systems (catalysts based on metal-containing particles immobilized in the pores of oxidic supports; catalysts based on mono- and bimetallic metal nanoparticles (Ru, Pd, Co, Cu), as well as magnetic particles immobilized in the pores of cross-linked aromatic polymers) can be successfully used in the transformation processes of plant polysaccharides, as well as such “platform compounds” as LA and FF.

It was shown that in the case of catalytic systems based on oxidic supports, the strength of acid centers is important: too high acidity allows a noticeable increase of reaction rates, however at the same time leads to carbonization of the catalyst surface and to corresponding decrease in its activity during reuse. Catalytic systems based on non-functionalized polymeric supports do not have this drawback. In this case, it is possible to increase catalytic activity by introducing a second metal-modifier.

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POSSIBILITIES OF REDUCTION OF CO₂ EMISSION IN GAS-CHEMICAL PROCESSES

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Reducing carbon dioxide emissions during production is one of the main trends in modern oil and gas chemistry. One of the most realistic possibilities for achieving this is to involve carbon dioxide generated in technological processes as a feedstock for producing gas chemical products. The maximum effect can be achieved in the production of large-scale chemicals, such as syngas, hydrogen, and methanol. In addition to the widely discussed catalytic processes of carbon dioxide reforming and tri-reforming of natural gas to syngas, CO₂ can also be successfully used in various non-catalytic autothermal processes based on natural gas. For example, the emission of carbon dioxide during the production of methanol and hydrogen can be almost completely avoided in a combined process for their production based on autothermal matrix conversion of natural gas, in which most of the carbon contained in natural gas is converted to the resulting methanol. This eliminates the emission of carbon dioxide into the atmosphere during methanol production, which even in processes based on tri-reforming reaches 0.91 t CO₂/t CH₃OH.

The CO₂ hydrogenation reaction is of interest as an approach to implementing the CCU strategy in the fight against man-made greenhouse gas emissions. At high temperatures (1200 K and above), the thermodynamically favored hydrogenation route is the endothermic RWGS reaction to form CO and H₂O, whereas at temperatures of 500–900 K in the presence of catalysts (typically those based on nickel), an exothermic Sabatier reaction occurs (reaction of CO₂ with H₂ to form methane). The use of catalysts makes it possible to combine these reactions with the subsequent selective conversion of CO to methanol, DME, and C₂₊ hydrocarbons, in particular, olefins.

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SYNTHESIS AND MODIFICATION OF CATALYSTS FOR PRODUCTION OF LOWER OLEFINS USING SUPERCRITICAL FLUID MEDIA

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The production of lower olefins – ethylene and propylene – in the processes of conversion of light alkanes (LA) – the main components of natural and associated petroleum gases – helps to expand the base for the production of petrochemical and basic organic synthesis products. Of particular interest are the oxidative transformations of LA with the production of olefins (oxidative coupling of methane, oxidative dehydrogenation of ethane and propane, oxidative cracking of C_{3+} alkanes), which do not have thermodynamic limitations. The practical implementation of this type of processes is significantly hampered by the lack of active, selective and stable catalysts.

From the point of view of synthesizing materials with a certain phase composition, porous structure and surface area, as well as optimizing their catalytic properties, approaches involving the use of supercritical (SC) fluids as a medium and reactant are of particular interest. Unlike “traditional” methods of thermal synthesis, processing in an SC fluid media involves the possibility of varying several parameters (in addition to temperature and process duration), such as density and composition of the fluid, and in the case of processing materials in water fluids (WF), regulation of acid-base and redox potentials of the medium.

As examples are considered

- formation of the phase composition and morphology of silicon and aluminum oxides used as catalyst supports during the processing of their precursors in a WF medium;
- synthesis of aluminates of alkaline earth and rare earth elements in a WF medium;
- the effect of synthesis conditions and structure of materials on their catalytic properties in the production of ethylene and propylene via the oxidative transformations of C_1 - C_3 alkanes.

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SAF-AND LCAF-FUELS: CHALLENGES ACHIEVEMENTS

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The main motivation in the world in SAF and LCAF fuels is to reduce the carbon footprint of aircraft operations¹. These categories of fuels are also called alternative aviation biofuels. There are examples in the world of creating SAF fuels in the component mixture form produced from traditional hydrocarbons with the addition of components from plant raw materials². There are known examples of creating 100% biokerosene using a plant component. Most often, in programs for the development of aviation industries in a number of countries (Europe, USA, China), compositions are introduced with the addition of 1-15% components based on plant raw materials to traditional hydrocarbon fuels³.

An analysis of the available data in the international periodical literature shows that the raw materials for the production of SAF fuels are: rapeseed, tall, camelina oils, and cooking fats. The most commonly used technologies for the production of SAF fuel components are: Fischer-Tropsch synthesis, hydrocracking, and catalytic cracking. Taking into account the simultaneous consideration of environmental, energy, economic, technological, operational and other indicators, it is possible to use other technologies and raw materials. At the same time, LCAF fuels are characterized by a multi-stage reduction in the hydrocarbon footprint down to zero^{2,3}.

In Russia today there are no technologies for the factory production of SAF and LCAF fuels. The need to create such fuels is acute, since such fuels are available in developed and developing countries, and they are included in the registers for aviation equipment. The absence of such fuels in Russia imposes restrictions on the sale of domestic equipment abroad, flights of ships of domestic companies over the territories of states that impose restrictions on anthropogenic emissions.

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IN-SITU UPGRADING AND ENHANCED OIL RECOVERY OF HEAVY OIL USING IN-SITU COMBUSTION PROCESS

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Heavy oil deposits are considered as one of the promising sources of hydrocarbons to meet the growing demand for energy due to the depletion of conventional light oil reserves. However, the development of such deposits is not as simple as in the case of light oils. In-situ combustion (ISC) may be a promising method for heavy oil recovery. During the injection of air and oxidation of oil, a combustion front is formed, which allows the displacement of oil into production wells due to the combined mechanism of action of hot water, steam and flue gases, which, thus, allows achieving a high oil recovery factor. This is a more energy-saving, environmentally friendly and effective method of enhancing oil recovery compared to other thermal methods. In addition, when using it, it is possible to carry out upgrading (partial processing) of heavy oil in the reservoir.

As part of the work, systematic studies of the oxidation processes of oil of various types and compositions, its individual group fractions and their mixtures, as well as model compounds in the presence and absence of rock were carried out. To determine the parameters of the oxidation processes of the systems under study under various conditions, methods of thermal analysis and calorimetry were used, which made it possible to evaluate the number of main stages of the process and their mechanism. To analyze the stability of the combustion front and evaluate the efficiency of oil displacement, special techniques for studying ISC under dynamic conditions were used - combustion tubes. To solve the problem of stabilizing the combustion front and regulating oxidation processes in reservoir conditions, a series of catalytic systems were used.

The new data obtained during the research will make it possible to create new technologies for heavy oil production based on the combined use of air injection and oxidation catalysts.

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CATALYTIC APPROACHES FOR PLANT BIOMASS PROCESSING

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The role of catalysis in the processing of bio-based materials seems to be key. The specifics of the processed plant raw materials (as a rule, a complex mixture of compounds, high oxygen and water content, low thermal stability, the presence of various functional groups in the compounds) imposes certain requirements on catalysts and process conditions as a whole.

In this regard, the development of catalysts for selective hydrogenation, oxidation, as well as for the processes of esterification, hydrolysis and dehydration of primary processing products is relevant and is presented in this work. Primary processing should be understood as the processes of rapid pyrolysis, extraction, steaming, hydrolysis, as a result of which plant biomass passes into a liquid, dissolved state and can be subjected to further catalytic processing.

In this work, emphasis is placed on the catalytic processing of furfural, a product of hemicellulose hydrolysis, to furfuryl alcohol and 2-methylfuran, which are positioned as a “building” material for organic synthesis and octane-enhancing additives. Cu-Fe- and Ni-Mo - catalysts with high selectivity to the target product were used as catalysts. The fuel direction is represented by the processes of transesterification with methanol and hydrogenation of vegetable lipids – triglycerides of fatty acids and free fatty acids to produce biodiesel and normal alkanes, respectively. To obtain a better fuel from the obtained alkanes, hydroisomerization to isoalkanes is required, which proceeds on Pt catalysts using acidic carriers, such as zeolites ZSM-5, Y.

The work was supported by the Ministry of Science and Higher Education of the Russian Federation within the governmental order for Boreskov Institute of Catalysis (project FWUR-2024-0038)

FEATURES OF ASPHALTENES AND RESINS COMPOSITION IN THE RESIDUAL PRODUCT OF VACUUM RESIDUE HYDROCRACKING AND PROSPECTS FOR ITS USE TO OBTAIN HIGH-MARGIN PRODUCTS

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TAIF-NK Refinery operates a complex for deep refining of heavy residues, including a unique unit for suspension combined hydrocracking, which ensures light oil product yield up to 98.2% and refining depth up to 98.6%. At the first stage in the process of liquid-phase hydrocracking of vacuum residue at temperature 430–470 °C and pressure 200–210 atm. in the presence of a special carbon additive without catalytic effect is obtained hydrogenysate purified from metal compounds, with up to 5% of unconverted heavy residues as a by-product. TAIF specialists have developed a fundamentally new technological approach that allows extracting an additional amount of distillate fractions from this residue, which also results in a unique product KOGG, which is a concentrate of asphaltenes and resins. At present it is known about the possibility of obtaining various high-margin products on the basis of oil asphaltenes - carbon materials, catalysts, polymer fillers, sorbents, etc. The new technological approach allows for extraction of additional distillate fractions from this residue.^{1,2} In order to determine the most promising directions of practical use of KOGG, its research was carried out with the identification of features of composition and structure of asphaltenes and resins by various methods (FT-IR, EPR, TGA, MALDI). It is shown that the use of KOGG in a mixture with polyethylene and polypropylene increases the melt flow properties of compositions by 1.5–2 times at filling up to 15 wt.%. In addition, the use of KOGG as a filler allows increasing the decomposition temperature of polyolefin compositions (up to 200°C according to TGA data).

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DESIGN OF TECHNOLOGY FOR RECYCLING FLUE GASES OF GPU AND TPP TO SECONDARY ENERGY CARRIERS

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One of the largest sources of CO₂ emissions in Russia is flue gases from thermal power plants (TPPs) and gas pumping units (GPUs). On the territory of the Russian Federation there are more than 370 large thermal power plants with a total installed capacity of about 163.5 GW, using natural gas as fuel in the European part of the country, as well as coal, APG, coke oven gas or peat¹. In addition, as of 2020, 3,781 GPUs with a total capacity of 46,8 million kW² operating on natural gas combustion were in operation in Russia.

In this work, we calculated technological schemes for processing flue gases from thermal power plants and gas compressors of various compositions and parameters into secondary energy carriers (methanol and DME), and also assessed their energy efficiency and carbon footprint.

Based on the results of modeling various technological schemes, a route for flue gas utilization was proposed, including the stages of adsorption CO₂ release, high-temperature electrolysis of a CO₂-steam mixture and methanol synthesis. This scheme was implemented at a pilot plant with a capacity of 1 ton of methanol per year and was successfully tested at the Shatrovskaya compressor station in the Kurgan region in December 2022.

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The calculation work was carried out at the expense of the state assignment of the TIPS RAS (FFZN-2022-0004 Capture and utilization of carbon dioxide No. 123012300040-4).

Design and engineering work, as well as a trial run, were carried out at the expense of an agreement with Gazprom Transgaz Ekaterinburg LLC.

PRODUCTION OF NAPHTHENIC OILS FROM HEAVY NAPHTHENIC-AROMATIC CRUDE

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Naphthenic oils are a popular product and are widely used as transformer oils, lubricant bases, plasticizer oils, etc. Currently, there is no production of naphthenic oils in the Russian Federation, and the need for them is satisfied by imports. Considering the availability of production capacities, catalysts and a raw material base in our country - naphthenic-aromatic oils (oils «Russkaya», «Severo-Komsomolskaya», «Pangodinskaya», «Yaregskaya» and others)¹, the development of domestic technology for obtaining naphthenic oils is relevant.

In world practice, various technologies for obtaining naphthenic oils are used: acid, contact purification, however, the most effective is hydrocatalytic processing of naphthenic-aromatic raw materials.

Research was conducted on hydrougrading of NK-350, 350-400, 400-450, 450-500 °C fractions of naphthenic-aromatic oil in the presence of a domestic industrial Al-Ni-Mo catalyst at 360-400 °C, 4-10 MPa, space velocity of 0,5 h⁻¹, hydrogen : feedstock ratio of 1200 nm³/m³.

An analogue of imported low-viscosity naphthenic oil with a pour point below minus 72°C was obtained from the hydrogenated fraction NK-350°C. Using this sample, an experimental batch of hydraulic oil MGE-10A was prepared and tested with positive results by qualification methods. Also, the hydrogenated fraction obtained from this fraction can be used as a component of winter and arctic diesel fuel and other base oils.

The possibility of obtaining a line of naphthenic oils with a kinematic viscosity at 40°C from 2.0 to 180.0 mm²/s from fractions of 350-400, 400-450, 450-500 °C is shown. The obtained oils can be used as components of engine, transmission, turbine and other oils, for lubricating high-pressure compressors, as softening oils, solvents and dispersants in the production of various technological preparations, in the manufacture of rubber, etc.

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ELECTROCHEMICAL EXTRACTION OF PURE HYDROGEN FROM METHANOL, DIMETHYL ETHER AND DIMETHOXYMETHANE STEAM REFORMING PRODUCTS

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The report discusses the feasibility of the electrochemical separation of pure hydrogen from reformat of oxygenates such as methanol, dimethoxymethane (DMM) and dimethyl ether (DME) using a electrochemical hydrogen pumping (EHP) with a polymer proton-exchange membrane based on polybenzimidazole [1].

Recently, we proposed efficient bifunctional $\text{CuO-ZnO}/\gamma\text{-Al}_2\text{O}_3$ and $\text{CuO-CeO}_2/\gamma\text{-Al}_2\text{O}_3$ catalysts for methanol, DME and DMM steam reforming reactions [2,3]. In particular, the catalysts, provide complete conversion of raw materials and the production of hydrogen-rich (~ 70 vol.%) with low CO content (< 1 vol.%) at atmospheric pressure and 300°C . Gas mixture of this composition can be fed directly to the EHP, which provides 90-95% hydrogen recovery and ~ 99.95 vol.% of purity at current densities of $0.33\text{-}0.35\text{ A/cm}^2$. The current-voltage characteristics of the EHP were studied and its power efficiency was evaluated as well. For example, the power efficiency of electrochemical separation of pure hydrogen from the reformat exceeds 80%. Thus, the results obtained allow as to propose the portable device for producing pure hydrogen.

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SHS CATALYSTS FOR SELECTIVE CO₂ HYDROGENATION TO HEAVY HYDROCARBONS

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Based on complex intermetallides obtained by self-propagating high-temperature synthesis (SHS), polymetallic catalysts with a nanostructured surface of the composition (95-x)Co-xCu-5La (x=10,30,50) (I), 80Mo-20Ni (II), 80Mo-20Co (III) and 80Mo-10Ni-10Co (IV) have been developed. The catalysts were tested in the process of CO₂ hydrogenation under relatively mild conditions (T = 200°C and P = 1 MPa), H₂:CO₂ ratio = 3:1 and gas space velocity of 2 NL·g⁻¹·h⁻¹. On the catalyst of group (I) (x=30), a CO₂ conversion of ~ 40% was obtained with a selectivity for heavy hydrocarbons (HC) C₂₊ of 39%, including 25% for liquid HC¹. The highest performance under the same conditions was obtained on catalyst (IV), where at a CO₂ conversion of 58%, the selectivity for the liquid fraction reached 46.9%. Increasing the temperature to 300°C increases CO₂ conversion to 96%, but methane predominates in the products (98%).

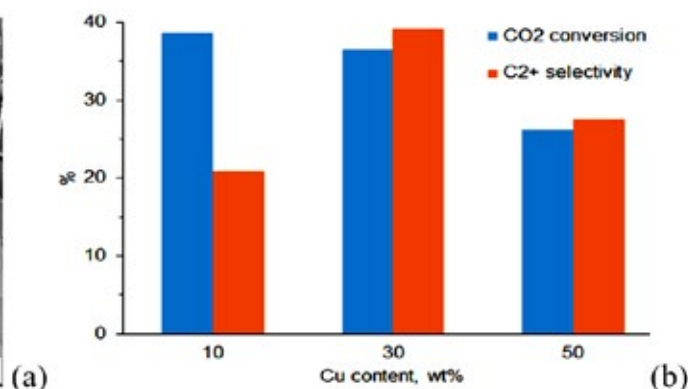
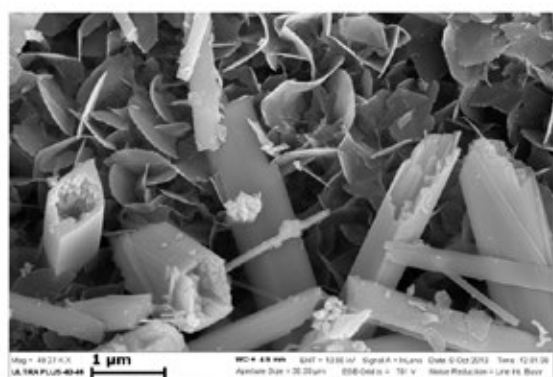


Figure 1. The surface morphology of the catalyst (95-x)Co-xCu-5La, x=30 (a), and effect of x on CO₂ conversion and C₂₊ selectivity (b).

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COMPARISON OF THE SULFIDE CATALYSTS ACTIVITY IN HYDROGENATION REACTIONS OF TWO-COMPONENT SYSTEMS

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The process of recycling polyvinyl chloride is harmful and hazardous to the environment. Thermal processing (pyrolysis) is considered to be the main way to recycle polyvinyl chloride, which results in the formation of a liquid product (pyrolysis oil) with a high content of chloroaromatic unsaturated cyclic and non-cyclic compounds of various structures [1, 2]. The presence of even a small amount of chlorine-containing compounds in pyrolysis oil/wax does not allow to use the refined products as fuel or petrochemical feedstock, and the content of organochlorine compounds in the liquid product should not exceed 10 ppm.

Previously, we have already attempted to study the course of the hydrodechlorination process of a model mixture of 1,4-dichlorobenzene in the presence of bimetallic sulfide catalysts [3]. However, the real feedstock has a more complex composition. Liquid products of processing (pyrolysis, hydrothermal processing, hydroconversion) of polyvinyl chloride are characterised by high chlorine content (1-20 wt.%) and are characterised by extremely complex composition. Involvement of several types of polymer waste (e.g. polyvinyl chloride, polyethylene, polystyrene, polyethylene terephthalate) in processing leads to even greater complexity of the composition of the resulting products. The target raw materials may have chlorine, sulphur and nitrogen-organic compounds in their composition.

The aim of the work was to compare the activity of unapplied sulphide bimetallic catalysts in the reactions of hydrodehydrogenation reactions of two-component model mixtures.

Sulfide catalysts synthesised in situ, were investigated in the processes of hydrodechlorination-hydrodeoxygenation co-processing of (1,4-dichlorobenzene - terephthalic acid) (Figure 1).

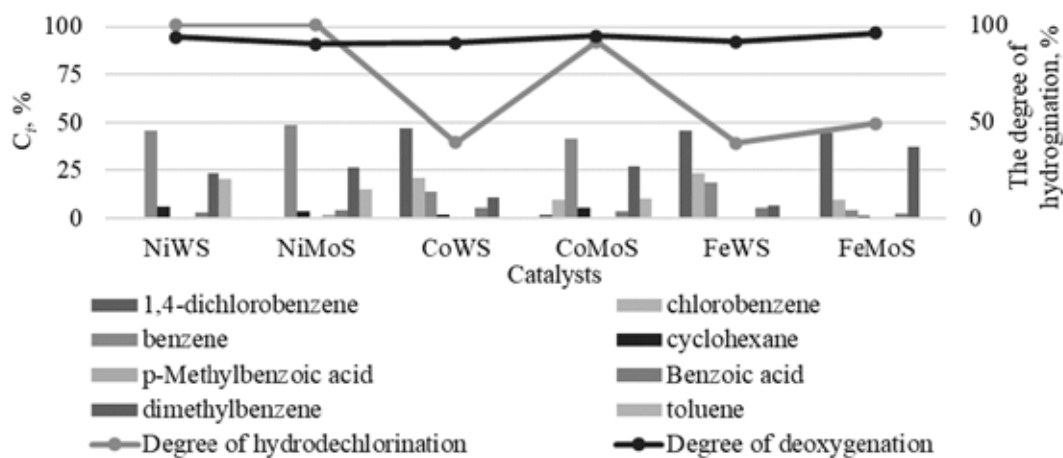


Figure 1. Effect of catalyst nature on the co-hydrodechlorination of 1,4-dichlorobenzene and terephthalic acid. where C_i is the content of the component in the mixture, %. Conditions: Pressure H_2 6 MPa, temperature 340°C, solution of 1,4-dichlorobenzene and terephthalic acid (molar ratio 1/1) in 10% n-hexadecane solution, molar ratio in the two-component system W(Mo)/component 1/40.

The maximum degree of hydrodechlorination and hydrodeoxygenation is achieved in the presence of NiWS catalyst. FeWS and FeMoS are low active in the hydrodechlorination reaction but show high activity in the hydrodeoxygenation reaction. The results obtained require a more thorough study of the competition for the active catalyst centres when several hydrodeoxygenation processes are carried out together.



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LOW-TEMPERATURE CONVERSION OF CO₂ INTO VALUABLE PRODUCTS BY PLASMA CATALYSIS

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Currently, the amount of carbon dioxide entering the atmosphere equals to tens of billions of tons per year. This is due to the developed global industry, the increase in the number of vehicles with internal combustion engines and other anthropogenic factors. To transform CO₂ molecules into valuable chemical raw materials, a significant amount of thermal energy is required to break the bonds in the initial molecules and shift the thermodynamic equilibrium¹. An alternative way to activate CO₂ molecules is plasma-chemical conversion – in this approach, high-energy plasma electrons promote the breaking of C–O bonds in the molecule, while the gas temperature remains relatively low. Combining plasma chemical and catalytic approaches for CO₂ conversion is also promising. When the catalyst is placed in the discharge region, there is both a change in the physical characteristics of the plasma due to the occurrence of microdischarges at the contact points of the granules, and catalytic effects due to the adsorption of CO₂ on the surface of the catalyst. For example, the use of Mg and Ce oxides allows increasing the conversion of CO₂ to CO in the barrier discharge from 10% up to 30%². In addition to direct decomposition of CO₂ to CO and O₂, the plasma catalysis method allows carrying out reactions of carbon dioxide reforming of methane³, hydrogenation of CO₂⁴ and other processes, while a distinctive feature of the reaction in plasma is obtaining liquid oxygenates in a single stage⁵. Development of new catalysts for plasma-catalytic conversion of CO₂ into valuable products is an urgent task in the fields of gas and petroleum chemistry.

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CATALYSTS BASED ON TRANSITION METAL COMPOUNDS IN HYDRODEOXYGENATION REACTIONS

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Involvement of renewable carbon feedstocks in the processing for the production of fuels and valuable chemicals contributes to the sustainable development of the fuel and energy industry. The main source of these feedstocks is plant biomass, which contains various oxygen-containing groups in its structure. One of the most effective pathways of biomass processing is hydroprocessing, which results in hydrogenation and hydrodeoxygenation reactions. Transition metal compounds are of considerable interest as catalysts for these hydroprocesses. While sulfides and oxides can be considered as traditional catalysts, phosphides, carbides, and nitrides are considered as alternatives.

In the present work, the hydroprocessing of various oxygen-containing substrates^{1,2,3,4} (phenols, aromatic esters, lignin) was studied using dispersed oxides and phosphides of Mo, W, and Ni synthesized *in situ* in the reaction medium. It was found that deoxygenation of substrates proceeds partially or completely depending on the type of catalyst used and reaction conditions. It is shown that oxygen-containing compounds with aromatic structure provide opportunities for obtaining various classes of substances that can be used as components of fuels, solvents, and intermediates for organic, petrochemical and polymer synthesis.

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CHANGES IN OIL COMPOSITION DURING MODEL BIOOXIDATION PROCESSES BY AEROBIC MICROFLORA STRAINS

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The paper presents the results of a study of the destruction processes of the oil system and changes in the composition of oil samples in the processes of model biodegradation in the aquatic environment under the influence of various strains of aerobic microflora. It has been established that under the same experimental conditions the rate of decrease in the mass of polluting oil in an aqueous substrate at concentrations from 2 to 5 wt% practically does not depend on the species composition of the studied microflora (*Koccoria sp. G+*, *Pseudomonas sp. G-* and a consortium of native species of microflora extracted from oil-contaminated soil samples on the territory of the Khanty-Mansiysk Autonomous Okrug).

It has been shown that in the oil biooxidation process not only petroleum hydrocarbons are destroyed, but also its polar high-molecular components – petroleum resins and asphaltene fractions. In one of the experiments (duration 30 days) with a total mass reduction of polluting oil of 84.7%, the reduction in the content of the saturated substances fraction was 87.2%, aromatic 79.1%, resins 91.0% and asphaltene 74.1%. Probably, partial destruction of supramolecular structures – resins and asphaltene occurs during the biooxidation of an oil system. And resulting low-molecular destruction products are utilized by microflora according to the usual mechanism of enzymatic attack.

A detailed analysis of changes in the component composition of the saturated hydrocarbons fraction by chromatography-mass spectrometry methods showed that after a 7-day exposure only isoprenoids remain in paraffins composition. Later they completely disappear and as a result the fraction of saturated compounds by the end of the experiment is represented exclusively by naphthenes. Already at the first stages of biooxidation all alkylbenzenes almost completely disappear in the fraction of aromatic substances. Condensed aromatic compounds are more resistant to the enzymatic attack of microflora and by the end of the 30-day experiment only naphthalene was completely utilized. For all classes of polycycloaromatics an increase in stability is observed with increasing molecular weights.

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CHEMICAL PROCESSING OF POLYMER WASTE

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The rapid growth of production and widespread consumption of plastic goods in modern society has led to the formation of an increasing flow of plastic waste (PW), the pollution of which poses a serious threat to the environment. Chemical processing of polymer waste is a promising way to increase the efficiency of the use of hydrocarbon raw materials

The report presents the results of a study on the depolymerization of separately polyolefins and mixtures of PW with a heavy vacuum residue (VR) by the method of hydroconversion using suspensions of dispersed catalysis. The influence of technological parameters on the yields and properties of the products of hydroconversion of a mixture of VR+PW has been studied.

With an increase in pressure in the reactor to 7.0 MPa, an increase in the concentration of fr. > 500 °C is observed, a decrease in the content of olefins and sulfur in distillate fractions, an increase in pressure to 10.0 MPa does not significantly improve the hydroconversion indicators. An increase in temperature above 400 °C and a reaction time of more than 2 hours is accompanied by a decrease in the yield of distillate fractions and an increase in the content of olefins in them, while the yield of coke increases sharply.

An increase in the content of polyolefins in the raw material mixture contributes to an increase in conversion, an increase in the content of paraffin and naphthenic hydrocarbons and a decrease in aromatic hydrocarbons in light and medium distillate fractions. The involvement of polymers of irregular structure in the raw material mixed with tar - rubber or a rubber component of styrene butadiene rubber, leads to an increase in the yield of olefins, naphthenes and aromatic hydrocarbons.

At a concentration of 0.05-0.1% wt in the reaction zone, the best results are achieved in the conversion of fr. > 500 °C and the structure of the reaction products during the hydroconversion of tar and a mixture of tar with polymers.

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ETHYLENE HOMOLOGIZATION OVER POLYFUNCTIONAL Ni-Mo-CONTAINING CATALYSTS

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Ethylene is a very essential raw material for petrochemical industries. The annual ethylene capacity growth reaches 4%. Majority of the ethylene produced is used for the development of polyethylene (about 65%). Today, a promising trend for ethylene conversion is not only the production of polymers, but also the production of propylene, butenes, and higher molecular weight hydrocarbons.

The most effective catalysts for the homologation of ethylene to C_3 - C_{18} alkenes are considered to be polyfunctional heterogeneous systems. Active sites for ethylene di- and oligomerization, acid sites, as well as sites for alkene metathesis present on the surface.

In this work, a comprehensive study of Ni-Mo-containing catalysts was carried out in order to apply them for the conversion of ethylene to propylene, butenes and C_{5+} alkenes. It has been shown that the ethylene oligomerize to butenes, hexenes, etc. on Ni-sites to the mechanism of metal complex catalysis. The isomerization and oligomerization reactions of C_4 - C_6 alkenes catalyze by acid sites. The production of propylene from ethylene is carried out by sequential reactions of ethylene dimerization, isomerization of 1-butene to 2-butenes and metathesis of ethylene and 2-butenes on Mo-containing sites. The use of aluminum oxide support for Ni-Mo-containing catalysts provides the maximum propylene content in the reaction products. Catalysts based on the higher acidic supports (B_2O_3/Al_2O_3 , SO_4^{2-}/Al_2O_3) have a high C_{5+} hydrocarbon fraction in the products. Increasing the temperature and weight hourly space velocity also leads to an increase in the contribution of oligomerization reactions with the formation of butenes and higher molecular weight alkenes.

Thus, varying the support nature of Ni-Mo-containing catalysts and the reaction conditions allows regulating the activity and the distribution of the ethylene conversion products.

This work was supported by the Ministry of Science and Higher Education of the Russian Federation within the governmental order for Boreskov Institute of Catalysis (project FWUR-2024-0039).

CHEMISTRY OF ADAMANTANE: ACHIEVEMENTS AND DEVELOPMENT PROSPECTS

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The lecture is dedicated to the 90th anniversary of the development of adamantane chemistry after its discovery in oil in 1933.

The following questions will be covered:

1. Introduction
2. Methods for selective functionalization of adamantane
3. Porous materials based on polyaryladamantanes
4. Adamantane in catalysis
5. Application of adamantane derivatives in medicine
6. Use of adamantane derivatives in nanotechnology
7. Development of adamantane chemistry in Russia
8. Conclusion

This work was performed according to the state program "Petrochemistry and Catalysis. Rational Use of Carbon-Containing Raw Materials" (project no. 121031300092-6).

COMPOSITIONS BASED ON THE PRINCIPLES OF “GREEN CHEMISTRY” TO INCREASE OIL RECOVERY OF DIFFICULT OIL RESERVES

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The share of hard-to-recover oil reserves (HTR) in Russia is constantly growing and currently exceeds 65%. In recent years, to solve the problem of increasing oil recovery from fields with HTR, the Institute of Petroleum Chemistry SB RAS on the principles of “green chemistry”, using deep eutectic solvents (DES), has created the fundamental foundations of new physical and chemical methods for increasing oil recovery, allowing production in mild, environmentally friendly conditions promising chemical reagents for increasing oil production. To develop fields with HTR, acid and alkaline oil-displacing compositions with adjustable physical-chemical, surface-active and rheological properties have been created, their solid and liquid low-freezing (minus 20–minus 60 °C) commodity forms, ensuring good logistics and manufacturability of use in fields in the northern regions and the Arctic. High efficiency for carbonate and terrigenous reservoirs has been shown by multifunctional compositions of a new type based on surfactants, three- and multicomponent DESs containing Lewis acids, polyols, carbamide, ammonium and aluminum salts, and fluorine-containing reagents. Of practical importance is the high efficiency of the action of acid compositions based on surfactants, inorganic polybasic Lewis acid, coordinating solvents and complex compounds with thermal methods – significant increase in the production of viscous oil, the oil displacement coefficient was 91-96%. The effectiveness of compositions and technologies of the Institute of Petroleum Chemistry SB RAS is confirmed by successful pilot testing and industrial use of technologies in the fields of Western Siberia and the Komi Republic, including in combination with thermal methods.

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STABILIZATION OF BIO-OIL FOR SUBSEQUENT CATALYTIC PROCESSING

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Bio-oil can be used as a feedstock for the production of various chemicals is a relevant problem. However, catalytic processing of pure bio-oil is not possible due to oligomerization reactions occurring under heating. The result is clogging of the process feed systems. Carrying out an additional step of bio-oil stabilization may be a successful solution¹.

Hydrogenation and alkali treatment of bio-oil were carried out in this work. After the stabilization, the bio-oil was tested for thermal stability according to the original method. The technique consisted of feeding stabilized bio-oil into a reactor heated to 530°C (inert) until the pump jammed. Bio-oil and stabilization products were subjected to qualitative analysis.

This work has shown that both approaches can increase the thermal stability of bio-oil by several times as shown in Fig. 1 (the stability of pure bio-oil ~350 s). Hydrogenation of bio-oil allows to reduce the amount of oxygen contained in bio-oil compounds, which are prone to oligomerization. Treatment of bio-oil with ammonia reduces the pH of the system to alkaline, which reduces the intensity of oligomerization reactions. The achieved results allow to use of stabilized bio-oil as a raw material for industrial processes, such as catalytic cracking.

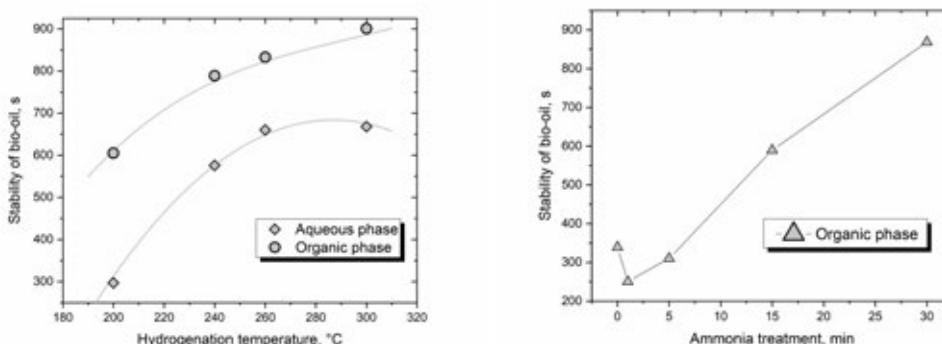


Figure 1. Stability of bio-oil after hydrogenation 7.5 MPa H₂, Ru-Ni catalyst (left) and ammonia alkalization (right)

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HYBRID ZEOLITE CATALYSTS FOR THE SYNTHESIS OF HYDROCARBONS WITH VARIOUS COMPOSITIONS

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The CO₂ converting into liquid hydrocarbons via methanol and dimethyl ether is currently actively developing. It includes both the production of C₅C₁₁ fractions with a high content of aromatic compounds and the production of C₈C₁₄ fractions similar in composition to aviation fuel. Zeolite catalysts with an MFI structure are the most well-known for conducting these reactions. To increase the yield of the liquid product and vary its composition, the synthesis of zeolites with a hybrid structure, combining different zeolite structures such as MFI and MEL, MFI and MTW, MFI and BEA, MFI and AEL, MFI and FER, is actively being developed. Hybrid materials can be obtained through the co-crystallization of zeolites with different structures or in the synthesis of “core-shell” type structures. Several studies have shown that such zeolites have an additional amount of mesopores, which promote the formation of cyclic and aromatic compounds.

This study summarizes and analyzes the experimental research results on the production of liquid hydrocarbons of various compositions using zeolite catalysts with hybrid structures in the conversion of oxygenates and lower olefins. The influence of the acidic and textural characteristics of hybrid zeolite catalysts on the yield of liquid hydrocarbons and their group composition has been investigated. The observed patterns are explained in terms of a dual-cycle mechanism of reaction.

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ONE-STEP SYNTHESIS OF TRIPTANE-ENRICHED LIQUID HYDROCARBONS FROM CO₂ AND H₂

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Converting of CO₂ into valuable organic compounds is one effective way to reduce the growing volume of carbon dioxide emissions. Reactions involving CO₂, a very stable molecule, are thermodynamically allowed when reducing agents, such as H₂, are used as coreagent. In this regard, the hydrogenation of CO₂ into hydrocarbons (HCs) may become one of the most important reactions, given that hydrogen can potentially be produced using renewable energy. Of particular interest is the obtaining of a product with a high content of isoalkanes, the most valuable among which is triptane (2,2,3-trimethylbutane), due to its high octane number (112) [1].

Previously, we demonstrated the possibility of synthesis of C₅₊-HC with the triptane content 2.2 wt. % from mixtures of CO and H₂ in an integrated unit via the stage of oxygenates formation [2]. In this work, a product containing triptane 5 wt. % was obtained by one-step synthesis from CO₂ and H₂ (340°C, 10 MPa) with a selectivity of 30 wt. % (52 wt.% of the total HCs amount) on bifunctional combined catalysts, which include CuZnAl-Al₂O₃ oxide and Pd-LaHY zeolite (La is introduced into HY by the traditional ion exchange method at 0.1 MPa and 90 °C) catalysts in the ratio 2:1. It has been shown that the introduction of La into HY by the method of single-step ion exchange in an autoclave at elevated temperature and pressure (185 °C, 2 MPa) provides a more uniform distribution of lanthanum cations in the volume of the zeolite, which has a positive effect on CO₂ conversion (27%) and gasoline selectivity 36 wt.% (60 wt.% based on the total HCs amount) and triptane content (10 wt.%).

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OIL DISPERSE SYSTEM TRANSFORMATION UNDER THE CAVITATION TREATMENT

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Research results of cavitation treatment of dark oil products for change their physicochemical characteristics are presented. The treatment was carried out in hydrodynamic mode. The pressure gradient ranged from 20 to 50 MPa, with 1 to 5 acts of exposure. Samples of straight-run and commercial fuel oils, catalytic cracking and straight gasoils, hydro-purified gas oils were used as research objects.

It is shown that cavitation processing leads to increase the yield of fractions boiling up to 400 °C, reduction the density and refraction coefficient of these fractions. Changes increase with the pressure and number of processing cycles. There is a correlation between the group hydrocarbon composition of raw materials and the yield of fractions boiling up to 400 °C. Fraction yield was higher the higher the resin content. As a result, the resin and asphaltene content in sample reduced and the share of arenas in the oil fraction increased.

Localization of cavitation bubbles is possible at phase boundaries¹. For oil disperse systems, this is the boundary of contact of complex structural units formed by resins and asphaltenes and dispersion of lower molecular weight hydrocarbons. Therefore, resins and asphaltenes should be more susceptible to degradation under the heat influence produced by the collapse of cavitation bubbles. X-rays and Raman spectroscopy recorded structural changes of asphaltenes: increasing the diameter of the layer, lowering the height of the aromatic layers packet, increasing the degree of their aromaticity. Data on asphaltene degradation under the cavitation influence are consistent with results of the samples elemental analysis. After cavitation treatment sulphur content in fractions boiling above 480 °C decreased, and its concentration increased in fractions with lower boiling points.

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DEVELOPMENT OF HIGH-PERFORMANCE MEMBRANE MATERIALS FOR STEAM REFORMING REACTORS

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Palladium-based membranes are used for gas separation and production of high-purity hydrogen.¹ Alloying such membranes with other base metals will increase their strength in a hydrogen atmosphere and reduce the cost of the final product.

During the work, samples of Pd-Cu_{40%} alloy foils were obtained, manufactured by three methods: melting and rolling with intermediate annealing (a), magnetron sputtering using a solid target (b) and a composite target (c). Samples of the resulting Pd-Cu40% films were studied as membranes in hydrogen transport processes.

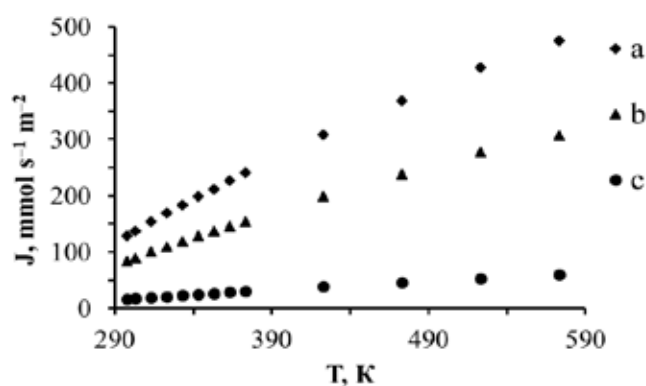


Figure 1. Temperature dependence of the density of the penetrating hydrogen flux at an excess pressure of 0.1 MPa on the inlet side of Pd-Cu_{40%} alloy membranes.

A significant increase in the flux density of membranes obtained by magnetron sputtering was recorded. This is probably due to a decrease in film thickness and ordering of the solid solution with the formation of a β -phase with a less dense crystal lattice. This method makes it possible to obtain defect-free thin-film materials with high permeability and low cost.

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The research was carried out with a grant from the Russian Science Foundation and with the financial support of the Kuban Science Foundation in the framework of the scientific project Num. 24-19-20070.

RHEOLOGY OF OILFIELD WATER-IN-OIL EMULSIONS: FEATURES OF FLOW/BEHAVIOUR DEPENDING ON COMPOSITION AND TEMPERATURE

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Oil produced as an emulsion is a dispersed oil system with a dispersed phase of two types – aqueous and solid (due to the presence of asphaltenes and waxes in the oil, as well as various mechanical impurities). The wide variety of oils and emulsions formed from them significantly complicates the identification of the relationship between the factors influencing their stability and rheological properties.

The steady state analysis using shear rheological measurements showed that the flow character of oilfield emulsions varies from Newtonian to non-Newtonian fluids depending on water content, composition (amount of natural emulsifiers, “gel” and solids) and sample temperature.

Table 1. Rheological types of oilfield water-in-oil emulsions when flow curves are approximated by Newton, Ostwald-Weil and Bingham models (n – constant characterizing the degree of deviation of non-Newtonian fluid properties from those of Newtonian fluids; τ_y – yield strength, Pa)

Samples	Water/»gel» content, wt%	Temperature, °C			
		10	20	30	60
Emulsion 1	9,7 / 0	Newton	Newton	Newton	-
Emulsion 2	15,0 / 0	Ostwald-Weil, $n=0,79$	Newton	Newton	-
Emulsion 3	37,5 / 0	Ostwald-Weil, $n=0,76$	Ostwald-Weil, $n=0,79$	Ostwald-Weil, $n=0,91$	-
Emulsion 4	62,0 / 8,0	-	Bingham $\tau_y=3,4$	-	Bingham $\tau_y=3,0$
Emulsion 5	53,0 / 25,0	-	Bingham $\tau_y=16,7$	-	Bingham $\tau_y=23,7$

Data obtained on the rheological behaviour of emulsions can be used to understand their stability during transport, storage and processing processes and to predict the demulsification process of such emulsions.

This work was supported by the State Assignment of the GEOKHI RAS.

ENSURING FLOW ASSURANCE AND ENERGY STORAGE: HOW TO MANAGE GAS HYDRATE FORMATION?

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Inclusion compounds called gas hydrates contain a framework of hydrogen-bonded water molecules enclosing one or more types of guest species, typically low molecular weight gases. High pressure and low temperature limit the thermodynamic stability zone of these compounds. Although gas hydrates are generally considered a severe complication in oil and gas production, they have been increasingly proposed as a means of solving various technological problems such as associated petroleum gas and carbon dioxide utilization, gas storage and transportation, gas mixture separation, water desalination, and even cold storage.¹

One of the urgent tasks is to find new, more effective reagents to control the hydrate formation process. Reagents that modify gas hydrate formation can be broadly categorized into two classes according to their mechanism of action: thermodynamic and kinetic ones. Thermodynamic agents change the water activity in the solution or incorporate into the cavities of the hydrate framework, causing a shift in the equilibrium conditions of hydrate formation (e.g., alcohols, saline solutions, tetrahydrofuran, etc.). Reagents that act kinetically do not affect the equilibrium conditions of hydrate formation but slow down (inhibitors) or accelerate (promoters) hydrate nucleation and growth.

This study comprehensively examined the impact of a wide range of reagents on the equilibrium conditions of hydrate formation (methanol, dimethyl sulfoxide, urea, acetonitrile, ethylene carbonate, and diethylamine) as well as their nucleation and growth (Gemini surfactant and humic acid extract). In the latter case, the wettability of the cell wall and the medium pH were varied. The data demonstrated that by properly selecting gas hydrate modifiers, the conditions for obtaining hydrates can be altered depending on the requirements, which can offer a resource-saving solution to energy storage.

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MODERN APPROACHES TO CO₂ CAPTURE USING LOW-TEMPERATURE COMPOSITE SORBENTS

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The task of reducing greenhouse gas emissions is relevant for the whole world, including the Russian Federation. In order to reduce the CO₂ level in the atmosphere, sorption systems are being developed for its capture from various gas sources (flue gases, biogas, and atmospheric air). These sources are characterized by different levels of CO₂ and other components, which may have an adverse effect on the sorbent's operation. CO₂, extracted using the sorbent from a gas source, can later be stored or used in industry or agriculture. Moreover, methods for catalytic CO₂ conversion into valuable chemical products have recently been actively developed.

The most advanced absorption systems available today are those that use aqueous amine solutions to capture CO₂. The drawbacks of the technology include the corrosive nature of amines, their volatility and instability in oxygen-containing environments. To solve the problem of volatile amine leaching, it is proposed to use non-volatile amine-containing polymers or ionic liquids dispersed on porous supports to accelerate the sorption process.

An alternative to amine-containing materials are low-temperature sorbents based on potassium carbonate (K₂CO₃), which is an inexpensive and widely available chemical reagent. Potassium carbonate is able to absorb CO₂ in the presence of water vapor: $K_2CO_3 + CO_2 + H_2O = 2KHCO_3$. Unfortunately, pure solid K₂CO₃ demonstrates a very low rate of CO₂ absorption, so it needs to be dispersed in the pores of a support. Since K₂CO₃ is a non-oxidizable compound, the approach based on using potassium carbonate allows to extract CO₂ from moist gas mixtures with a high oxygen content. The main shortage of composite sorbents based on K₂CO₃ is quite high energy consumption for thermal regeneration of the material.

In general, the choice of CO₂ sorbents for industrial applications is likely to be determined by the optimal balance between the cost of the material and its physical and chemical characteristics, which determine the energy efficiency of the CO₂ capture process and the service life of the sorbent.

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CLUSTER OF PRODUCTION OF CHEMICALS BASED ON VEGETABLE OILS

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The chemical and technological aspects of the use of renewable raw materials based on vegetable oils for the domestic production of a wide range of high-value chemical products are considered.

Due to their chemical structure (the presence of reactive ester and olefin groups in triglycerides), vegetable oils and their derivatives are widely used in various sectors of the national economy¹. Various ways of conversion vegetable oils into valuable products are shown. Physical methods make it possible to obtain vitamin concentrates (vitamin E), lecithins, squalenes, etc., which are used in the cosmetic and pharmaceutical industries. Using chemical methods, oils can be effectively converted into fuel hydrocarbons², fatty acids (including dicarboxylic acids) and alcohols³, environmentally friendly plasticizers and PVC stabilizers⁴, a wide range of functional additives for the needs of small-scale chemistry, paint and varnish, polymer industry, as well as the synthetic detergent industry.

Additionally, ways of conversion the by-product - glycerol - into valuable products of organic synthesis were demonstrated.

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The work is performed in the framework of the development program “Priority-2030” of the Mendeleev University of Chemical Technology of Russia.

DEHYDROGENATION OF PROPANE ON $\gamma\text{-Al}_2\text{O}_3$ MODIFIED WITH MANGANESE

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Currently, propane dehydrogenation is receiving much attention as an important source of propylene. Aluminum oxide is most often considered as a catalyst for propane dehydrogenation. Among the various crystalline phases of Al_2O_3 , $\gamma\text{-Al}_2\text{O}_3$ has attracted much attention due to its lower formation of by-products. To increase activity and thermal stability, various modifying additives are added to the aluminum oxide catalyst. The purpose of this work is to study the effect of the addition of manganese to $\gamma\text{-Al}_2\text{O}_3$ on its physicochemical and catalytic properties in the process of propane dehydrogenation.

The modification of $\gamma\text{-Al}_2\text{O}_3$ was carried out by impregnating it with aqueous solutions of manganese salt ($\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$). The manganese concentration (in terms of metal) varied from 2 to 12 wt.%.

The activity of the catalysts under study was studied in a flow-type installation with a fixed catalyst bed ($V = 3 \text{ cm}^3$) at a reaction temperature of 550 to 650 °C.

The results of studies of the catalytic properties of the samples showed that modification of $\gamma\text{-Al}_2\text{O}_3$ with manganese helps to increase its activity in the process of propane dehydrogenation. At the same time, the modified catalysts exhibit a higher dehydrogenation ability in the process under study at a lower temperature ($T = 600 \text{ °C}$). It has been established that for Mn-containing catalysts the optimal concentration is 4.0% manganese content in the catalytic system. A further increase in the concentration of manganese in the catalyst to 8% does not lead to a significant change in its activity and selectivity in the process of converting propane into olefins.

Thus, based on the results obtained, it was established that the $\text{Mn-Al}_2\text{O}_3$ catalytic system can be used as an active and accessible catalyst for the production of olefinic hydrocarbons from propane.

The work was carried out with the financial support of the Russian Science Foundation, project 24-23-00386.

HIGHLY DISPERSED SULFIDE CATALYSTS FOR HYDROUPGRADING OF PETROLEUM FEED UNDER WATER GAS SHIFT REACTION

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The one of the promising strategies for hydroupgrading of water-containing feed is water involvement into Water Gas Shift Reaction (WGSR), where their interaction with carbon monoxide leads to generation of *in situ* hydrogen for hydrogenation of aromatics and heteroatomic compounds. For thus one-stage processing, where water removal and hydroupgrading occurs simultaneously, the unsupported dispersed catalysts are supposed to be the most justified¹⁻³.

This work evaluates the catalytic behavior of unsupported dispersed catalysts based on transition metal sulfides and produced through high-temperature decomposition-sulfidation of water- or oil soluble metal precursors in hydrotransformation of model feed, containing of aromatics and organosulfur compounds, under WGSR conditions. For multicomponent model feed with the competitive sorption of reactants, the mutual influence of aromatics and sulfur compounds on their transformation, products distribution, and reaction pathways depending on the composition of catalyst and reaction media, temperature, pressure were estimated. For model feed and oil fractions the effectiveness of hydroupgrading, that accounted sulfur removal and aromatic hydrogenation, were evaluated in view of the influence of water content, hydrogen source, and syn gas composition.

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DEVELOPMENT AND IMPLEMENTATION OF THE ALGORITHM CONDUCTING RESEARCH AND EVALUATION THE EFFECTIVENESS OF THE SOLVENT

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To date, the problem of the formation of asphalt-resin-paraffin deposits (ARPD) is particularly relevant for most oil fields at a late stage of development. Therefore, it is important to select an effective and safe hydrocarbon solvent for removing deposits from downhole equipment during oil production. Moreover, an individual solvent is selected for each ARPD, depending on the composition of the deposit. This process is long and complicated. During the research of borehole ARPD from various fields of Bashkiria, the authors developed an optimal algorithm.

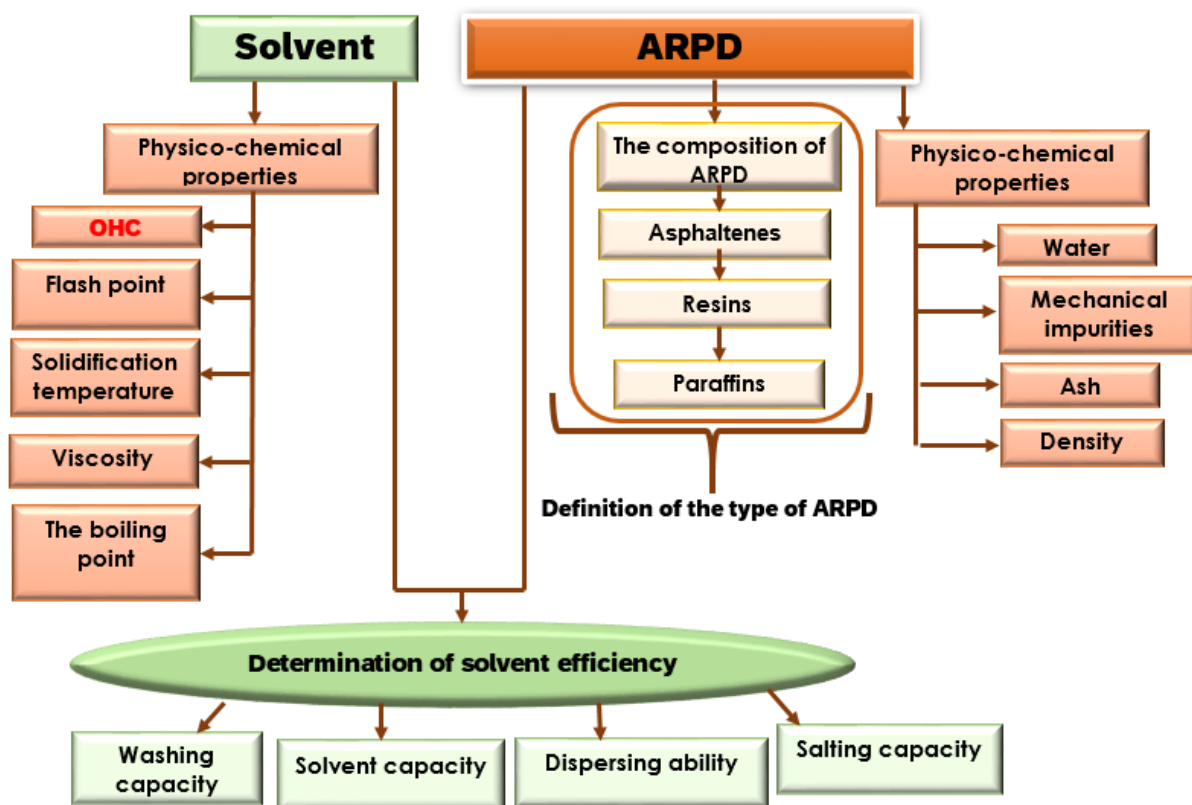


Figure 1. Algorithm for determining the effectiveness of the solvent

As a result of the implementation of the algorithm, comprehensive information on the composition of the ARPD was obtained, a comprehensive assessment of the effectiveness of solvents was performed, and recommendations were issued for conducting pilot tests at real deposits.

COMBINING HYDROCONVERSION PROCESSES AS A METHOD FOR PRODUCING MOTOR FUELS FROM RENEWABLE HYDROCARBON FEEDSTOCK

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The work assessed the feasibility of obtaining environmentally friendly components of motor fuels from renewable hydrocarbon feedstock (vegetable oils) through sequential catalytic hydroprocessing using two types of catalyst: a hydrotreating catalyst and a zeolite catalyst (structural type ZSM-5). The process was carried out in a laboratory flow-through catalytic unit with the following technological parameters: temperature 375 °C, pressure 70 bar, feedstock space velocity 1 h⁻¹, hydrogen consumption 0.0021 m³/hour.

For the obtaining products, the main physicochemical characteristics and hydrocarbon composition were determined.

Table 1. The characteristics of obtained products

Characteristics	Unit	Rapeseed oil product	Corn oil product	Sunflower oil product
Density at 15°C	g/cm ³	0.794	0.779	0.776
Dynamic viscosity at 15°C	mPa/s	1.248	0.797	0.757
N-alkanes content	% wt.	3.47	7.58	5.95
Iso-alkanes content		3.14	17.63	18.75
Cycloalkane content		1.81	6.86	6.37
Aromatic hydrocarbon content		68.31	48.26	48.58

As a result of the study, it was also found that the obtaining products are characterized by a wide fractional composition (the average boiling point of 10% vol. was 61 °C, 50% vol. was 154 °C, 90% vol. was 322 °C), which makes it possible to separate from them gasoline and diesel fuel fractions.

The main characteristics and composition of the separated fractions were also determined, and the possibility of using the obtained fractions in the production of motor fuels was shown.

The study was supported by a grant of Russian Science Foundation No. 23-23-00101, <https://rscf.ru/en/project/23-23-00101/>.

CRITERIA FOR THE PROPENSITY OF COALS TO SPONTANEOUS COMBUSTION, ESTABLISHED IN THE ZONE OF LOW-TEMPERATURE THERMAL DECOMPOSITION

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The identification of coals prone to spontaneous combustion allows you to take measures in advance to prevent the occurrence of a fire¹. One of the methods for studying processes and parameters capable of characterizing coal prone to spontaneous combustion is the TGA method².

The paper presents the approbation of the self-ignition criteria³ obtained for the hard coals of the Pechora deposit on the coals of the Kuznetsk coal basin. Fig.1 shows the rates of mass change of the studied coals during thermal decomposition in a low-temperature region: No. 1-2 – seam Chetvertye (Komsomolskaya), No. 3-4 – seam Moshchnye (Vorgashorskaya), No. 5-6 – seam 69 (Taldinskaya-Zapadnaya), No. 7-8 – seam Polysaevsky-II (A.D. Ruban).

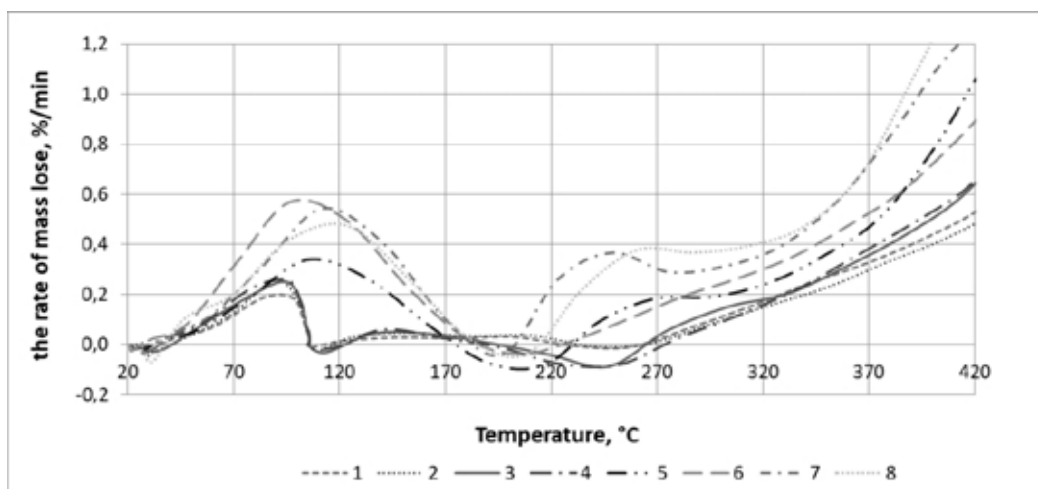


Figure 1. DTG-curves of non-prone (No. 1-2) and prone (No. 3-8) to spontaneous combustion of coals

It is found that the rate and magnitude of the thermal effect of carbon oxidation can be indirectly determined by TG dependencies.

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EXTRACTION DESULFURIZATION OF LIGHT VACUUM GAS OIL WITH DEEP EUTECTIC SOLVENTS IN A MINICHANNEL

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The composition of oil and petroleum products includes sulfur compounds of various natures, the control of the content of which is important for solving a number of technological, analytical and research tasks. Sulfur compounds have a negative impact on the environment, operational and transport properties of petroleum raw materials, and their cost. Various methods are used to isolate sulfur compounds from petroleum products. Currently, one of the new directions for solving the above problem is extraction desulfurization in micro- and mini-recorders using a new class of extractants - deep eutectic solvents. Minichannels are simple flow-through devices that allow to intensify the extraction process by increasing the area-to-volume ratio of phases, reducing the path of diffusion of molecules and phase contact time. Deep eutectic solvents, unlike classical organic solvents, are recognized as a safe, environmentally friendly, biodegradable class of reagents.

In this work, desulfurization of vacuum gas oil was carried out in minichannels using deep eutectic solvents based on salts of quaternary ammonium bases and carboxylic acids. During the study, the influence of the structural and operating parameters of the minichannels (diameter, length, capillary material, flow velocity, volume ratio of the phase supply) on the efficiency of extracting sulfur compounds from petroleum products was revealed, flow maps were built for each case. It is shown that the most effective flow model in the minichannel is the projectile (Taylor flow), in which the degree of extraction of sulfur compounds from vacuum gas oil using deep eutectic solvents reaches 36% in 50 seconds, the phase contact time required to establish extraction equilibrium during the process in the traditional mode (shaker, magnetic stirrer) is 30 minutes. Multistage extraction in a minichannel made it possible to extract up to 70% of aromatic sulfur compounds from vacuum gas oil within a few minutes.

The work was carried out within the framework of the state assignment of the GEOKHI RAS.

TROPICAL RESISTANCE OF UREA GREASES

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The complex impact of environmental factors, such as temperature, humidity, and UV radiation, can lead to the deterioration of the physicochemical properties of lubricants during storage and use. As part of a joint research project between TIPS RAS and the Russian-Vietnamese Tropical Centre, the features of changes in the properties (dropping point, oil separation, ultimate strength, and anti-wear performance) of urea greases for different purposes were studied for the first time through long-term exposure to natural conditions for 6, 14, 19, 24, and 30 months. It has been demonstrated that urea-based lubricants have significant potential for resistance in tropical environments, which is attributed to the nature of the organic thickener. The thickener is a stable, structural framework composed of diurea molecules, which retains the components of the base oil thanks to strong hydrogen bonds. Additionally, the urea thickener exhibits antioxidant and biocidal properties. This is confirmed by the lack of signs of biological corrosion. It has been found that thermal effects, in combination with UV radiation, are the causes of the destruction of lubricants due to a gradual change in colloidal stability, during the loss of a portion of the base oil. Chemical changes that occur in ureate grease upon exposure have been studied using infrared (IR) spectroscopy.



Figure. The change in the appearance of urea grease when exposed to tropical climates.

The work was carried out within the framework of the State assignment for the Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences.

THE USE OF ADDITIVES TO IMPROVE THE PROPERTIES OF WATER-BASED DRILLING FLUIDS

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Oil and gas companies are challenged to develop new fields characterized by low penetration and difficult drilling conditions. As a result, more and more demands are being demanded for drilling fluids (DF) in solving current challenges in the oil and gas industry, including increased oil recovery.

During drilling wells emergencies may occur due to corrosion of equipment, swelling of wellbore, overheating of equipment, wear of drill bit, etc. To prevent such accidents, various additives are introduced into drilling fluids.

Many companies use water-based drilling fluids due to their lower environmental impact and stability at high temperatures. To improve rheological and filtration properties, additives are added to the mud¹. These additives could be surfactants, nanoparticles, polymers or other substances.

The effect of surfactants is their adsorption on the surface of well walls, drill bit and all other surfaces. This slows down corrosion of equipment, swelling of well walls and decreases the friction coefficient of the drill bit against the surface.

Polymers are used to increase the viscosity of the drilling fluids. Polymers are fairly well soluble in water, and their use also reduces fluid loss.

Nanoparticles are used to preserve formation penetration and increase well productivity. They help in solving a number of critical problems: dealing with pipe adhesion to well walls, reducing corrosion, reducing drilling fluid loss, etc.

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OPTIMIZATION OF MODES AND FEEDSTOCK COMPOSITION OF CATALYTIC CRACKING WHEN INVOLVING VEGETABLE FEEDS IN PROCESSING

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One of the promising research direction today is the involvement of renewable feedstocks in the processing of petroleum fractions, including the catalytic cracking, as well as the optimization of the composition of catalytic cracking catalysts, the composition of mixed feedstock, and the modes of its processing [1].

The research is aimed at optimizing the composition of mixed feedstock of the catalytic cracking, containing up to 50 wt% of vegetable feeds (rapeseed, tall oil, and linseed oil) and the process conditions using the laboratory unit MAK-10 (ASTM D 3907). We also defined the spent catalyst and fractional composition of liquid products of catalytic conversion (ASTM D 2887). The research results (Table 1) showed that the products yield and fractional composition of products from catalytic conversion of feedstock mixture significantly differ for two types of catalysts.

Table 1. Experimental study of the catalytic cracking involving a mixture of vacuum gas oil (VGO) and rapeseed oil (RO) as feedstock.

Feedstock	Catalyst 1				Catalyst 2	
	VGO	VGO + RO 10% (mass.)			VGO	
Temperature of the process, °C	502	482	492	502	502	502
Gas volume, ml	74,9	99,9	111	117	184,9	174,9
Liquid products yield, % mass.	89,09	82,52	78,5	78,73	63,55	78,14
Conversion, % mass.	15,11	23,71	27,05	30	78,44	71,66

The involvement of 10% rapeseed oil in vacuum gas oil processing increases the feedstock conversion at a temperature of 502 °C by 14.89% and 6.78% by mass for two different catalysts (conditions of steam treatment at 788 °C in a 100% steam environment). Such effect is associated with the intensification of catalytic cracking reactions due to the thermal destruction of fatty acids. According to the results of TGA-DSC, the coke content on the catalyst surface increases. It must be account for optimizing the thermal operating mode of the catalytic cracking reactor and regenerator.

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ASSESSMENT OF THE CHEMICAL COMPOSITION OF TUBERS OF ANTHOCYANIN-CONTAINING POTATO VARIETIES IN THE CRNZ OF THE RUSSIAN FEDERATION

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Currently, potato varieties with dark-colored flesh are of interest because they contain anthocyanins, which are strong antioxidants in the human body¹. In this regard, the chemical composition of tubers of such potato varieties requires study. In 2023, an assessment of the chemical composition of tubers of three potato varieties with different flesh colors was carried out, selected when considering the harvest in the experimental field of the Tver State Agricultural Academy on sod-medium podzolic soil. The nitrogen content was determined by the Kjeldahl method, phosphorus by the Kirsanov method. The results of the studies are presented in Table 1.

Table 1. Yield and chemical composition of tubers of potato varieties

Variety	Coloring of the pulp	Dry matter content, %	Crude ash content, %	Nitrogen content (according to Kjeldahl), %	Phosphorus content (according to Kirsanov), %
Gala	yellow	15,9	0,97	0,27	0,049
Syurpriz	red-mottled	21,8	1,29	0,30	0,052
Severnoe siyanie	blue-mottled	24,1	1,11	0,29	0,039

As a result, it was revealed that the anthocyanin-containing varieties Surprise and Northern Lights were characterized by a higher content of dry matter, crude ash and total nitrogen, as well as a moderate starch content (10.1 - 12.4%), which makes them more suitable for dietary nutrition.

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GAS HYDRATES OBTAINED IN SOLUTIONS OF ALCOHOLS: STABILITY AND COMPOSITION

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The relevance of hydrate-based technologies for storing and utilizing gases in low-temperature formations has increased with the exploration of oil and gas fields in Arctic regions. However, the possibility of using these technologies depends on the stability of hydrate-containing sediments when they encounter used reagents. To assess the feasibility of introducing such technologies, we conducted a study on methane hydrates obtained from aqueous solutions of lower alcohols, including unsaturated ones. The study examined the equilibrium of methane hydrate with gas and alcohol solution, including under conditions of ice crystallization. The composition of double hydrates of methane and alcohol (if any) was also analyzed. The data obtained on the stability of the hydrates help to identify the temperature and pressure ranges of formations that can be used to store gas in hydrate form.

The work was supported by the Russian Science Foundation (grant 23-79-01312).

HYDROCHEMICAL METHODS OF RESEARCH AS CRUCIAL TOOLS OF OIL FIELD EXPLORATION AND DEVELOPMENT

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Both oil and natural gas are deeply connected to the hydrosphere of underground. This is due to the fact that the hydrosphere of underground is an environment in which hydrocarbons are formed and accumulated according to their placement patterns in the depths. All aquifers bear the imprint of a long geological chain of oil and gas ontogenesis.

Massive volumes of water formed as a result of mixing and chemical interaction of reservoir, pumped and technological waters is extracted together with hydrocarbons during the process of field development. This makes information about the nature, properties and composition of associated waters crucial both during stages of exploration and development.

Hydrochemical methods of research made solving problems that arose during the exploration and development of fields in the Northern Caspian Sea, the Volgograd region and Western Siberia possible. Conducted research included diagnostics of the nature of water samples obtained during formation testing within geological exploration; identification of regional hydrochemical correlatives based on regular monitoring of the composition of associated waters; determination of the nature of reservoir saturation based on the study of drilling mud; prediction of water compatibility and salt deposition risks; discovering the influence of solutions and reagents used in experimental-industrial work on the composition of associated waters.

Wide range of applications of hydrochemical research makes it an integral part of a complex of studies of building geological models and monitoring field development.

NEW ASPECTS OF GAS SEPARATION USING HYBRID TECHNOLOGY BASED ON GAS HYDRATE CRYSTALLIZATION AND MEMBRANE TECHNOLOGY

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Gas hydrate crystallization is one of the promisingly developing methods for natural gas processing. On one hand gas hydrates are the negative phenomena in gas and oil industry, because they are formed in gas pipes, blocking flow and give the risk to shutting-down. But on the other hand gas hydrate are found to be applied for benefit purposes, as for natural gas processing. Gas molecules are sorbed in crystalline cavities at specified thermobaric conditions.

Membrane separation was chosen to increase the efficiency of the gas hydrate crystallization method. It is based on sorption-diffusion mechanism of gas components. Both processes were made in common mass transfer module, therefore number of process stages was constant, that is obviously benefit of the hybrid technology.

Gas separation experiments based on the gas hydrate crystallization and membrane technology were made.

There were used two membrane modules: with 20 and 40 hollow fibers of polyetherimide+polyimide (PEI+PI). For the efficiency tracking different stage cut ratios were used (the ratios of gas hydrate flow to feed gas flow): 0.1, 0.2, 0.5, 0.8, 0.9. There were also different ratios of permeate flow to retentate gas flow: 1/1, 1/2, 1/5, 1/10 (all of these numbers were respective to the specified stage cut).

Experiment conditions: pressure in module was 4.25 MPa at temperature of 280.15 K. The difference between high pressure area and low pressure one was 2 MPa.

Experiments showed that hybrid method is significantly efficient for gas mixture separation compared to the only gas hydrate crystallization method. There are such components as xenon, propane concentrated in gas hydrate phase. There are carbon dioxide and hydrogen sulfide in the low pressure area. And the gas phase is rich by ethane and methane.

Performed with the support of the Russian Science Foundation, grant № 22-79-10222.

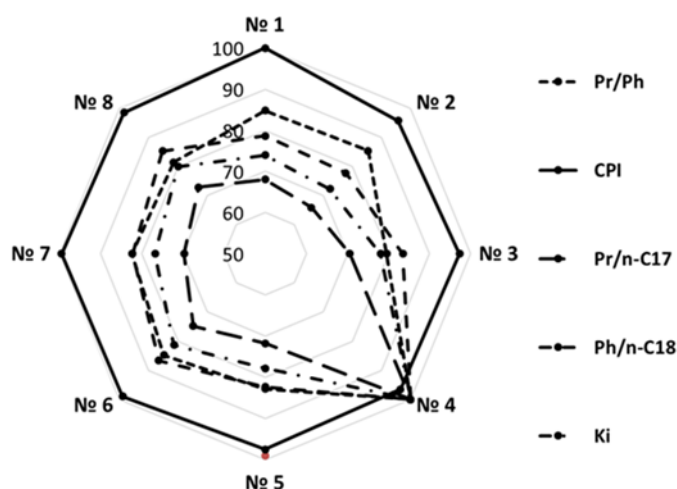
VARIATIONS OF COMPOSITION IN RECOVERABLE OIL AND WELL PRODUCTIVITY FORECAST

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The study of variations in the oil composition from different wells of the same oil field is of interest for reservoir management, optimizing oil recovery operating mode and has long been used in oil production. In this work the isomeric and homological composition of saturated and aromatic compounds in oil samples from one of the oil fields of the Khanty-Mansiysk Autonomous Okrug was studied using chromatography-mass spectrometry methods. The values of geochemical parameters were used to compare the composition of oil samples. Geochemical parameters are calculated from the absolute and relative contents of isomers of different classes of substances and are widely used in oil geochemistry.

Some of geochemical parameters calculated from the isomeric composition of paraffins in several wells (No. 1 - No. 8) are compared on the diagram. For greater clarity, the parameter values are normalized – the maximum value of any parameter in a set of samples is taken as 100%.



As follows from the diagram the composition of the oil in one of the wells (No. 4) differs significantly from all the others. There is information in the literature that wells in which the oil composition differs significantly from the statistical average for the oil field, are fueled by deep oil fluids. Therefore, well No. 4 should be considered as the most likely candidate for deep recharge and inflow. And also other geological and operational characteristics of this well should be studied in more detail.

The study was financially supported by Government of Khanty-Mansiysk Autonomous Okrug - Yugra (order from 20.06.2023 № 10-P-1534).

APPLICATION OF MESOPOROUS POLYMER SORBENTS BASED ON DIVINYLBENZENE AND 4-VINYLBENZYL CHLORIDE FOR HYDROCARBON SEPARATION

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Physical and chemical properties of lower alkanes determine their wide application in many industries. These alkanes are part of natural, shale and associated petroleum gases and are valuable raw materials. However, the process of their separation requires high energy consumption, as it is carried out by low-temperature distillation. Adsorption methods of purification are an energy-efficient alternative for hydrocarbon separation¹. In this regard, porous polymeric sorbents are of interest, the possibility of controlling the structure of which makes it possible to obtain a set of sorbents with properties that meet various separation tasks².

Polymer microspheres were synthesised by suspension radical polymerisation based on divinylbenzene and 4-vinylbenzyl chloride monomers.

The specific surface area and pore size distribution were determined by nitrogen sorption/desorption isotherms on a Sorbi MS META device (Russia). The specific surface of polymer microspheres was found to be 83.9 m²/g, pore diameter 3.96 nm.

Thermodynamic characteristics: specific retention volume (V_g), adsorption-equilibrium constants (K) and selectivity (α) were obtained by inverse gas chromatography (IGC) on a Chromos GC-1000 instrument in the temperature range of 303.15 to 333.15 K.

The parameters of hydrocarbon separation were determined based on the data obtained using the ideal adsorbed solution theory.

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OBTAINING POLYALPHAOLEFIN BASE OILS USING FISCHER-TROPSCH SYNTHESIS OLEFINS BY OXIDATIVE OLIGOMERIZATION

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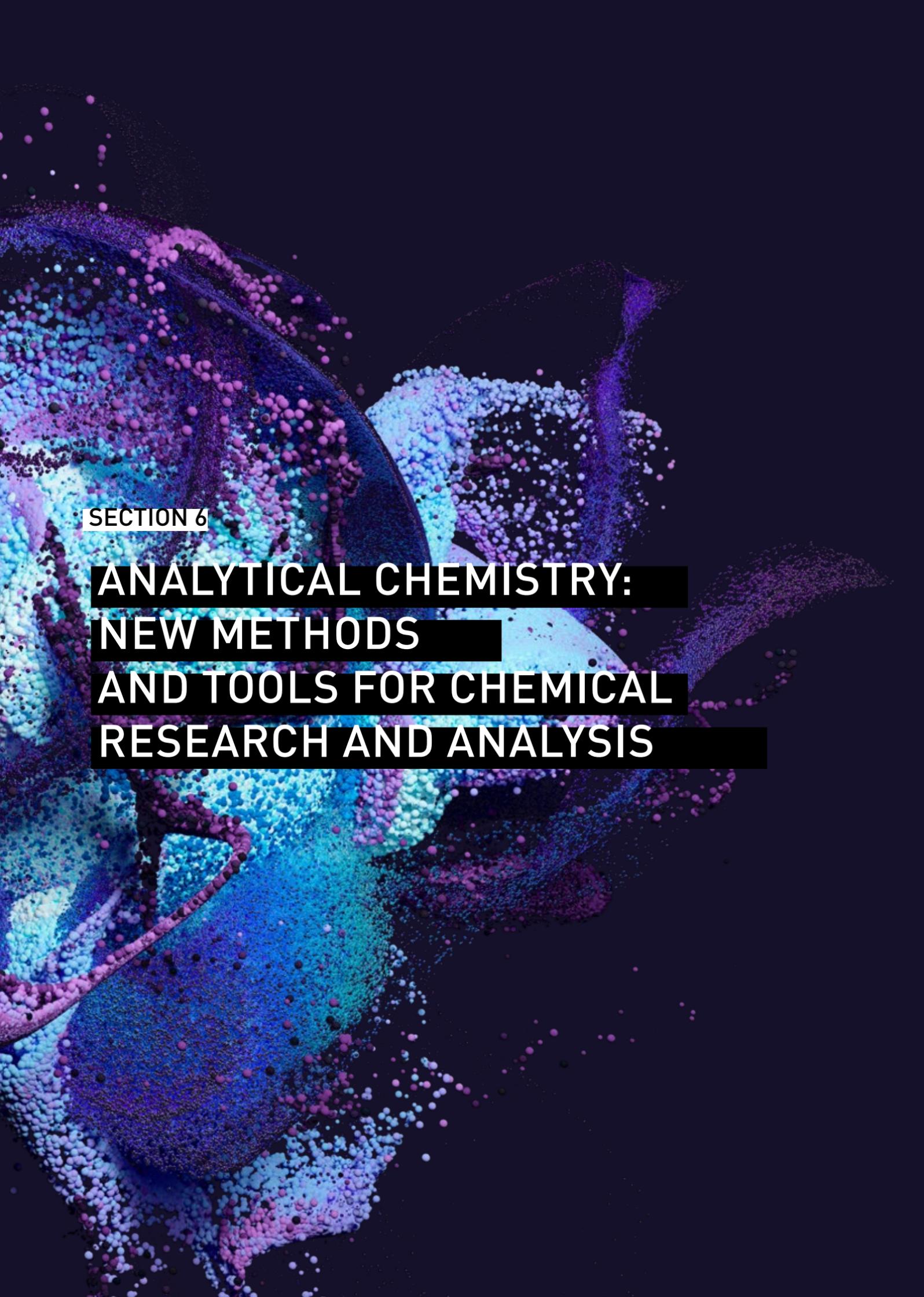
Due to the growing global demand for polyalphaolefin (PAO) oils, which are obtained by oligomerization of C_{10} - C_{12} α -olefins, research is being actively carried out to expand the base of starting materials, namely monomers, for their production. Recently, Fischer-Tropsch synthesis has been considered as an alternative method for the production of unsaturated hydrocarbons with different chain lengths. However, unsaturated hydrocarbons obtained by Fischer-Tropsch synthesis are a mixture of hydrocarbons containing, along with α -olefins, β - and γ -olefins, which are formed in significant quantities during the synthesis process, complicating the use of standard catalysts which are currently implied in industry for synthesizing PAO base oils.

This work will present the results of a study of the oligomerization of a synthetic diesel fraction enriched in unsaturated hydrocarbons, synthesized on a bifunctional cobalt catalyst [1] by oxidative oligomerization using zirconium octanoate as a catalyst. To understand the influence of the olefin structure (change in the position of the double bond, the presence of side branches) on the properties of the final product, a series of experiments were carried out under similar conditions using decene-1 as a feedstock. It has been established that the presence of a branched hydrocarbon chain in the feedstock with increasing viscosity (corresponding to the molecular weight of the oligomerization product) reduces the pour point of the products. The oligomerization product of the synthetic diesel fraction is similar in its properties (kinematic viscosity and pour point) to commercially available PAO.

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The work was carried out with the financial support of the Russian Science Foundation Grant No. 23-23-00466 using the equipment of the Center for Shared Use "Nanotechnologies" of the Southern Russian State Pedagogical University (NPI) named after M.I. Platova.

An abstract, high-resolution visualization of a molecular structure, possibly a protein or a complex crystal lattice. The structure is composed of numerous small, colored spheres (pink, blue, and white) arranged in a complex, interconnected pattern. The background is a deep, dark blue, and the overall lighting is dramatic, highlighting the intricate details of the molecular model.

SECTION 6

**ANALYTICAL CHEMISTRY:
NEW METHODS
AND TOOLS FOR CHEMICAL
RESEARCH AND ANALYSIS**

NON-LABORATORY ANALYSIS USING OPTICAL MULTISENSOR SYSTEMS

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The needs of modern high-tech society for chemical analysis is growing rapidly. The throughput of instrumental methods, including widely used optical spectroscopy, is no longer sufficient to provide the required level of analytical control of a growing number of objects and media. The traditional approach associated with sampling and centralized laboratory testing is not suitable for solving new problems, such as on-line monitoring of various processes, field and express-analysis.

Analytical chemistry responds to today's challenges by developing optical multisensor systems (OMSs) – low-selectivity analyzers designed to solve specific problems of quantitative or qualitative analysis. OMS can be based on light-emitting diodes (LEDs), optical filters, detector arrays of different sensitivities, and similar elements¹. Careful optimization of individual system sensors allows for highly accurate analysis with a small number of optical channels². This makes it possible to significantly reduce the size and cost of the analyzer, increase the measurement speed, create portable and autonomous devices. The lack of selectivity is compensated mathematically by the use of chemometrics algorithms, which play a key role in the OMS design and application.

The presented report is devoted to a discussion of scientific and technical issues of the creation and implementation of optical multisensor systems. Examples of their development and application in solving real practical problems are presented³.

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DETECTORS OF EXPLOSIVES: STATE OF THE ART

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The paper discusses typical objects that indicate the presence of explosive devices or explosive charges. These objects are detected by instrumental analyses methods and include traces of target substances on surfaces or vapors in air. The amount of explosive traces or vapors in assay samples can vary within a wide range. The mass of an explosive substance in the first latent fingerprint left on a surface just after contact with an explosive charge is $\sim 10^{-6}$ g, while in the 10-th and 100-th sample it is $\sim 10^{-7}$ g and $\sim 10^{-9}$ g, respectively. The concentration of volatile explosives in air near real objects can be ≤ 1 ng/cm³.

The main analysis capabilities, operating parameters, and cost characteristics of different detection devices are reviewed and compared. The devices of interest include fieldable mass spectrometers (fMS), ion mobility spectrometers (IMS), gas chromatographic devices (GCD), stand-off laser spectroscopic devices (SLSD), and optic, electric, electrochemical, thermometric, and mass-sensitive sensors that are used to detect and identify vapors and of explosive substances (see Table).

Table

Device	¹ LOD for traces, $\times 10^{-12}$ g	¹ LOD for vapors, $\times 10^{-15}$ g/cm ³	² t_a , s	³ Selectivity	³ Cost
fMS	10	10	1	H	H
IMS	10	10	1	M	M
GCD	0.01	10^{-1}	10^2	H	M
⁴ SLSD	10^4	10^3	10^2	M	H
Sensors	10^2	10^2	1	L	L

¹ LOD – limit of detection; ² t_a – speed of response; ³ H – high, M – medium, L – low; ⁴ for ~ 10 m distance.

NEW APPROACHES IN SAMPLE PREPARATION AND ANALYSIS OF ROCKS, ORES AND BIOLOGICAL SAMPLES BY ICP-MS/AES METHODS

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It is difficult to imagine achieving breakthrough results in conducting research in the field of geochemistry, cosmochemistry, biology, medicine, and other disciplines today without performing research of new, highest quality, including chemical analysis. The new quality of analytical results here means making determinations using unique solutions (technical, computational, etc.) with the lowest possible detection limit and with correctly estimated uncertainty of the results. These results can form a pool of reusable data (FAIR). Modern research in geochemistry and cosmochemistry in recent decades has been largely associated with the development of local analysis, both elemental and isotopic. The tasks of the bulk analysis are mainly aimed at determining extremely low concentrations of elements, primarily rare, rare earth, refractory, noble, and analyzing “difficult” samples (rocks including hard-to-open minerals, or the decomposition of which is accompanied by loss of elements for various reasons).

ICP-MS is today the leading method for determining ultra-low concentrations of elements and/or isotopes in both bulk and local analysis.

Bulk analysis usually involves decomposition of the sample. The quality of the decomposition determines the success of the analysis as a whole, making a major contribution to the overall uncertainty of the analysis results. In addition, this is a time-consuming stage that determines overall productivity.

A critical review of the methods of samples digestion, including black shales, granites, some ores, biomedical samples, etc., is presented. These are acid digestion both at atmospheric pressure and in autoclaves, digestion with ammonium bifluoride, sintering/fusion with fluxes, etc. To determine ultra-low concentrations of rare earth elements and noble metals, various concentration methods, including modified carbon nanomaterials, are considered.

To ensure data control obtained by ICP-MS/AES methods while their interpretation in the subject area, a multidimensional model of the analysis results based on a DBMS is proposed. It harmoniously integrates with the FAIR system

“MICROPLASTICS” AND “NANOPLASTICS”: A NEW PROBLEM AND CHALLENGE IN ANALYTICAL CHEMISTRY

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Microplastics and nanoplastics (MP and NP) are an invisible global problem. Nearly 80% of the plastic ever created is not recycled in any way and continues to pollute the environment where it breaks down into smaller particles. The problem of microplastics is one of the world's greatest environmental challenges. Up to 8 million tons of plastic enter the world's oceans every year, most of it as microscopic particles floating on the surface or buried in the sediment. It gets there through the use of personal care products, decorative cosmetics, paints, washing synthetic clothes. It easily passes through filters and sewage treatment plants and ends up in rivers, oceans and other bodies of water.

The main source of airborne plastic is car tyres, which rub plastic dust off the road. Plastic particles are so common that they fall to the ground in rain and snow and are even found in food and drinking water.

Chemists around the world are just beginning to recognize the problem and look for ways to solve it (including the analysis and identification of these types of materials).

This report discusses the main types of sources, compares the properties of MFs and NPs, and gives the chemical composition of the most common particles found in nature. The main types of sample preparation of objects for the separation of MFs and NPs will be considered, various modern approaches to the analysis of such particles and corresponding methods of analysis (with their advantages and disadvantages) will be presented.

X-RAY FLUORESCENCE ANALYSIS OF SMALL MASS SAMPLES FOR GEOCHEMISTRY, GEOECOLOGY AND ARCHAEOLOGY

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Quantitative elemental analysis of relatively small (less than 300 mg) samples is required for the solving of specific scientific issues. The developed technique based on the fusion of 110 mg of a sample with lithium metaborate and further X-ray fluorescence determination of Na, Mg, Al, Si, P, K, Ca, Ti, Mn, Fe and some microelements (Sr, Zr), made it possible to expand the possibilities of X-ray fluorescence method for:

- analysis of cores of bottom lake and peat sediments in order to reconstruct environmental conditions and climate in the past¹;
- analysis of archaeological objects in order to study their origin with the possibility of saving unique artifacts²;
- analysis of samples of snow cover solid phase³ and coal ashes for the environmental monitoring of potentially polluted urban areas.

The advantage of proposed technique is the possibility of further analysis of fused samples using inductively coupled plasma mass spectrometry⁴.

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RAPID TEST SYSTEMS: NEW APPROACHES FOR DETECTING LOW- AND HIGH-MOLECULAR WEIGHT COMPOUNDS

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The combination of porous planar carriers, receptor molecules and colored, highly dispersed marker particles allows rapid detection of a variety of analytes. Immunochromatographic tests (ICTs) are the best known among such analytical systems, being effective tools for out-of-laboratory diagnostics. However, new types of tests are in demand, providing lower detection limits, reduced time and increased productivity. The report will present studies of Immunobiochemistry laboratory of the Research Center of Biotechnology in this area and will evaluate the developed approaches.

Composition and antigen-binding ability of antibody complexes with gold nanoparticles of different sizes and shapes, the most common markers in ICTs, have been determined. The influence of the surface density of immobilized antibodies on parameters of ICTs using the resulting conjugates has been studied. Mathematical models have been developed to describe the formation of detectable immune complexes in ICTs, and factors limiting the decrease in the detection limit have been identified. For systems providing simultaneous detection of up to four mycotoxins and antibiotics, the choice of binding zones location has been grounded based on the immunoreagents' affinities. Variants of immunochromatography with single- and multi-stage inclusion of markers into immune complexes have been compared. ICTs with various colored and fluorescent marker nanoparticles, including those of different shapes – nanospheres, nanoflowers, nanostars, nanorods – have been implemented. Test systems with different types of signal amplification - in situ growth of nanoparticles, nanozyme catalysis, aggregation of functionalized nanoparticles, etc. – have been developed. To increase information output, multithreshold and multi-zone test systems have been developed. The achieved characteristics of the test systems have been compared with theoretical calculations. Criteria for a priori assessment of the capabilities of new candidate markers and analysis schemes have been elaborated. The proposed approaches have been successfully applied and validated for different types of analytes – low molecular weight compounds (antibiotics, pesticides, myco- and phycotoxins), proteins (specific antibodies, markers of inflammation and cardiac dysfunction), viruses and bacteria, providing 10-20-minute determination with lowering detection limits from 10 to 500 times.

APPLICATION OF LUMINESCENT SEMICONDUCTOR QUANTUM DOTS IN ANALYSIS

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Quantum dots (QDs) are semiconductor nanoparticles with optical and electronic properties that differ from bulk semiconductor. Quantum dots have been widely used in solar cells, photovoltaic devices, light-emitting diodes, displays, photocatalytical processes and as photoluminescent label for biomedical research. Small size, photo- and colloidal stability, bright turnable emission, high surface area and possibility to create desired design give prospects of wide QD application in bioanalysis as labels and nanosensors.

Luminescent QDs are widely used as labels for immunoassay. The nature of QDs makes it possible to significantly change the analytical characteristics of the methods by varying the modifying agents, the composition of conjugates, and the conditions of analysis. The report summarizes the main results on the use of CTs as labels for the determination of a wide range of analytes in various objects^{1,2}.

In this work we demonstrated the capabilities of analytical nanosensors based on the quenching of the fluorescence of alloyed CdZnSeS/ZnS QDs and ternary AgInS/ZnS QDs for operative detection of anthracycline antibiotics (ANTs). To maximize the nanosensor quenching efficiency, the spectral features of QDs and ANTs were carefully studied, and the complex nature of QD fluorescence quenching in the presence of ANTs was shown. Using optimized conditions, turn-off fluorescence nanosensors for direct doxorubicine and mitoxantrone detection in human plasma were developed^{3,4}.

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THE SENSE OF SMELL OF ANIMALS AND INSECTS AS A GUIDELINE FOR THE DEVELOPMENT OF OLFACTORY TECHNOLOGIES

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The high olfactory ability of animals and insects attracts the attention of researchers with the aim of using them to control the presence of explosives and other harmful substances by smell, and for the early detection of diseases. Biological objects are also attractive due to the possibility of developing new approaches to cyborg chemical perception, in which brain signals from bio-objects are directly used to detect and recognize vapors of various substances. If in the first case it is necessary to train animals and insects to respond to detectable odors, then in the second case methods for decoding brain signals should be developed. The report provides a brief overview of the problem of using the sense of smell of animals and insects, including the implementation of cyborg sense of smell.

Successful work on creating systems for testing humans for the presence of explosives using trained rats was started in the USA in the 1970s.¹ Since the 1990s, there have been numerous works in different countries on training mice and wasps to identify explosives by smell, ants to detect cancer cells and explosives, and bees to detect coronavirus. Advances in identifying the high sensitivity of “biological” detectors initiated work in the field of cyborg olfactory technologies. In this case, electrodes are implanted into the brain of an animal (rat, mouse) or insect (grasshopper, locust), and substances detected by a “live” detector are determined by processing electrical signals.² For trinitrotoluene, high sensitivity thresholds at the level of pg have been achieved.

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CONTRIBUTION OF CHEMOMETRICS TO GREEN ANALYTICAL CHEMISTRY

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Environmental pollution caused by human activities is one of the major problems of modern society. Chemistry is fundamentally not very environmentally friendly, including analytical chemistry. The attempts to get rid of the use of environmentally hazardous substances, techniques and procedures in chemical analysis are an important area of the modern research. Chemometric data processing is now a well-settled part of analytical chemistry. The application of chemometrics offers several valuable advantages such as reliable qualitative and quantitative analysis in cases of non-ideal, overlapping and poorly selective signals. It helps to eliminate complex physical manipulations with samples and instrumentation, resulting in significant simplification and cost reduction of analysis, and in turn leads to significant environmental benefits of chemometrics. The motto that chemometrics itself is “green” is quite popular and various authors have already emphasized this in recent literature [1, 2]. At the same time, the specific “green” impact of chemometrics has not been characterized numerically. A very popular metric for this purpose is the Analytical Eco-Scale method [3]. In this paper, we have evaluated a number of representative studies from the current literature using Eco-Scale score and compared chemometric analytical procedures with their conventional counterparts in terms of environmental friendliness. It is shown that the eco-friendliness of specific analytical problems can be significantly improved by using chemometric methods [4].

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PLASTICIZED, PASTE AND METAL OXIDE FILM ELECTRODES IN IONOMETRY, VOLTAMMETRY AND ECOLOGY

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The report presents the results of a comprehensive study that includes three interrelated areas as follows:

- potentiometric study of the electrode behavior of conventional and solid-contact ISEs based on PVC membranes, plasticized by DOP/DBP, as well as carbon paste electrodes in relation to hydrophobic onium cations, which includes the characterization of electrode functions and estimation of their selectivity coefficients (K_{ij}^{not}) unbiased by membrane and solution composition;
- voltammetric study of carbon paste electrodes, the paste of which contains redox-active complex compounds synthesized in NIIC SB RAS;
- electrolysis of nitric acid solutions with additives of the redox system Ce(IV)/Ce(III) in an electrolyzer with Nafion membrane separated cathode and anode spaces under normal conditions ($t < 100\text{ }^{\circ}\text{C}$, $p = 1\text{ bar}$) with metal oxide anodes, in which an IrO_2 film is electrolytically sedimented on a titanium base to prevent corrosion phenomenon. The mediated electrochemical oxidation (MEO) process is carried out in a reactor connected to the electrolyzer in batch or continuous dosing mode.

The main results of the research:

- thermodynamic characteristics of interphase distribution of R_4N^+ cations in the series; methods for determination of onium cations in technology process solutions and wastewaters, indirect determination of alcohols; determination of nonionic surfactants by functional fragments, etc.
- data on $E_{1/2}$ of complexes, results of electrocatalytic properties study in relation to water hydrogen and oxygen evolution reactions, electrocatalytic reduction of CO_2 , etc.
- data on the EDTA mineralization kinetics in MEO process during continuous monitoring and its mathematical model, calculations of steady-state current for continuous EDTA decomposition with simultaneous cerium (IV) electrochemical regeneration in galvanostatic mode based on the diffusion current model.

NEW EXPRESS METHODS FOR ANALYSIS OF NON-METALLIC INCLUSIONS IN STEELS AND ALLOYS

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Determining the content of non-metallic inclusions (NI) in steels and alloys is an urgent task for product quality control. The determination of NI is carried out using quantitative metallographic analysis using optical and electron microscopy with X-ray microanalysis, which are not express methods. Methods for monitoring NIs using fractional gas analysis (FGA) and optical emission spectral PDA analysis make it possible to provide results on the amount of NIs in a metal sample within 5-15 minutes, but the lack of standard samples and certified methods prevents their use in industry.

Fractional gas analysis (FGA) is a modification of the method of high-temperature extraction of oxygen and nitrogen from a sample in a carrier gas at a given heating mode. The method is based on differences in the thermodynamic stability of oxides and nitrides and the possibility of their fractional separation during monotonic heating in the analyzer oven. Due to a priori uncertainty in the number and parameters of extraction peaks, the problem of analyzing and interpreting gas release curves (evalograms) belongs to the class of incorrectly posed nonlinear inverse problems. The report presents the results of using an analysis method based on neural networks that takes into account physical information about oxide inclusions potentially contained in the sample. The developed algorithm was tested on the results of the analysis of standard samples.

A comparison was made of the results of determining of non-metallic inclusions in samples catted from steel ingots using optical metallography according to the ASTM E1245-03 standard, fractional gas analysis (FGA) and optical emission spectral PDA analysis. Based on the experimental results, a mathematical model for interpreting the results of spectral analysis using the PDA method was developed, a correlation equation was obtained, and the volume fractions of non-metallic inclusions in the studied samples were calculated. The capabilities of the method for monitoring the cleanliness of steel by NI are demonstrated. Comparison of the results of determining NI in samples using various methods showed good agreement.

ELECTROCHEMICAL (BIO)SENSORS BASED ON SMALL ORGANIC MOLECULES FOR SOLVING SOME CURRENT PROBLEMS OF ECO-, BIO- AND PHARMACEUTICAL MONITORING

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The development of methods for non-laboratory rapid analysis and portable devices based on them remains one of the most important tasks of modern analytical chemistry. Electrochemical (bio)sensors successfully complement laboratory methods as primary rapid screening tools in the field of medical diagnostics, environmental monitoring and food safety. Structuring the receptor layer of such devices using small organic molecules can significantly expand the analytical capabilities and prospects for their application. Original small molecules - promising drug candidates and targeted functionalized with a pronounced effect of intramolecular charge transfer - can act as functional elements of electrochemical biosensors. We have proposed original approaches and electrochemical (bio)sensors based on nitrogenous heterocycles - derivatives of azoloazines, diazines, carbazoles as cross-linkers, electrocatalysts, labels and elements of biomolecular recognition for the rapid determination of some clinically significant parameters of blood, infectious agents, nitroaromatic compounds in samples with different matrices. Molecular recognition in some cases is closely related to the type of biological activity, and wide synthetic capabilities make it possible to ensure effective immobilization of molecules on the surface of an electrode and nanomaterials. The use of an integrated approach, including computational and physicochemical research methods, makes it possible to carry out the most effective search for complementary analyte-receptor pairs, as well as predict the response change as a result of supramolecular interaction and/or immobilization. The proposed approaches and sensors are universal and can be integrated both into the commercially available portable electrochemical analyzers and the original laboratory prototypes of electrochemical test platforms developed by us.

The research was financially supported by the Russian Science Fund (project No. 20-13-00142-P, project No. 20-73-10077-P).

NEW SENSORS FOR PERSONAL HEALTH DIAGNOSTICS

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People want to know more about their health and prevent diseases that can lead to unpleasant consequences. A personal device for diagnosing the condition of the skin odor, or more precisely, by volatile products of excretion through the skin, has been developed using one nanostructured gas piezo sensor.

Unlike typical gas analyzers, the sensor of the BioScan health bioscanner is not tuned to any specific gas, but belongs to the universal detectors due to the sorbent, which shows affinity for many volatile organic chemical compounds - markers of physiological processes in the body.

The scanner is designed for personal use and in terms of its functionality occupies an intermediate place between gadgets and diagnostic equipment. The gas sensor has been repeatedly tested in different conditions, on different batches and mass of sorbent phases with different intervals of skin expiratory monitoring: for a long time and for a short time in specific abnormal situations (stress, disease).

The property of the development is a personal program with an intuitive interface, in which not only measurement, its storage and processing are carried out, but also the measurement results are translated into a three-level interpretation for an untrained user: the final numerical value of the health index, the verbal characteristics of the most probable reasons for deviations from the norm and the generation of an image by artificial intelligence based on the final conclusions.

New features of skin exhalation at different points – projections of organs (Zakharyin-Gede zones) have been established, which make it possible to assess the hidden processes of vasospasm and ducts; stability of the functioning of important organs and systems, for example, liver/heart; the degree of symmetry of the left and right organs, etc.

In 2024, the device received a declaration of compliance with the requirements for the production of a small batch. There are no analogues in Russia and abroad.

GRAND OPTICAL SPECTROMETERS: A NEW INSTRUMENT FOR MEASURING ANALYTE MASS FRACTIONS

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Currently, Grand¹⁻³ optical spectrometers have been developed and mastered in production. The spectrometers are recorded under No. 89108-23 in the State Register of Measuring Instruments of the Russian Federation and are available in ten modifications:

1. Atomic emission spectrometers:
 1. Grand-SVCh nitrogen microwave plasma spectrometer and Grand-ISP argon inductively coupled plasma spectrometer for liquids;
 2. Grand-Globula, Grand-Kristall, and Grand-Express air arc and spark spectrometers for the direct analysis of powders by evaporation from the channel of a graphite electrode and for metal analysis;
 3. Grand-Potok arc spectrometers for the rapid analysis of geological powdered samples using the spill-injection method, including scintillation atomic emission analysis for reducing the limits of determination of noble metals;
 4. Grand-Expert and Grand-Favorit argon spark vacuum spectrometers for the rapid analysis of metals and alloys with simultaneous determination of non-metallic inclusions;
 5. Grand-Pavlin flame spectrometer for the determination of alkali and alkaline earth elements in solutions.
2. Grand-AAS high-resolution continuum-source atomic absorption spectrometers with an electrothermal atomizer for the simultaneous determination of elements in a liquid.

Spectrometers based on assemblies of new photodetector arrays provide high-sensitivity simultaneous acquisition of spectra in the entire working spectral range at a rate of up to 1000 spectra/s, which significantly improves the results of chemical analysis.

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USING OF PRELIMINARY CONCENTRATION OF TRACE ELEMENTS AND NON-STANDARD METHODS OF SAMPLES INTRODUCING INTO THE EXCITATION AND IONIZATION SOURCES FOR ANALYSIS OF HIGH PURITY SUBSTANCES

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Mass spectrometry and atomic emission spectrometry with inductively coupled plasma (ICP-MS and ICP-AES) are one of the most informative methods of quantitative chemical analysis (QCA) both in terms of the number of analytes and their detection limits (LODs). Along with these common methods, the atomic emission spectrometry with two-jet arc plasma (TJP-AES) using an excitation source with a power an order of magnitude higher than ICP, is very promising. At the same time, the capabilities of QCA methods for the analysis of high-purity substances are limited by the need to dissolve samples for ICP-MS and ICP-AES analysis and dilute them with graphite powder for TJP-AES analysis. This leads to an increase in the contribution of the blank experiment, a decrease in the signal of the analytes and deterioration of their LODs. The LODs of analytes can be significantly improved by:

- using of preliminary concentration of trace elements,
- using of non-standard high efficient methods of introducing of the samples and trace elements concentrates into the excitation and ionization sources.

The report will present the results of using of the preliminary concentration of trace elements (reactive distillation of the sample base, vacuum distillation of the sample base, sorption concentration) and such methods of introducing of the samples and trace elements concentrates into the ICP and TJP as electrothermal vaporization and laser ablation for the analysis of high-purity acids, germanium, cadmium, tellurium, zinc, etc. The advantages and limitations of these techniques will be considered. Using of these techniques allows reduce the LODs of analytes by 1-2 orders of magnitude and significantly improve the analytical capabilities of QCA methods in relation to the analysis of high-purity substances and precursors of functional materials.

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BIOMONITORING OF THE IMPACT OF VOLATILE AROMATIC HYDROCARBONS ON HUMANS BY CHROMATO-MASS SPECTROMETRY METHODS

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Benzene, toluene, ethylbenzene and isomeric xylenes (BTEX) are priority toxicants with harmful effects on human health. Exposure to these compounds can be assessed by measuring their concentrations in whole blood, the gold standard for which is gas chromatography-mass spectrometry coupled with headspace solid-phase microextraction. Limitations of the method include difficulties in storing and transporting refrigerated whole blood and the emission of target substances from vacuum blood collection tubes. The detection limits of the analytes were limited by the maximum level of their emission from the caps of vacuum tubes and amounted to 20 μM for toluene and 2 μM for other substances.

When determining urinary BTEX metabolites using HPLC-MS/MS with electrospray ionization in negative ion detection mode, blank samples did not contain extraneous signals. Detection limits of 0.003 μM were achieved for hippuric acid, isomeric methyl hippuric acids, N-acetyl-S-benzyl-L-cysteine and N-acetyl-S-phenyl-L-cysteine. When analyzing urine samples from chemical synthesis operators ($n = 36$), the content of biomarkers of exposure to toluene (hippuric acid), o-xylene (2-methylhippuric acid) and m-xylene (3-methylhippuric acid) was $3.2 \pm 1.1 \text{ mM}$; $4.0 \pm 2.1 \mu\text{M}$ and $1.9 \pm 0.9 \mu\text{M}$, respectively. For the comparison group (persons without occupational hazards, $n = 22$) these indicators were: $2.2 \pm 0.7 \text{ mM}$; $0.4 \pm 0.2 \mu\text{M}$ and $0.6 \pm 0.3 \mu\text{M}$.

The content of amino acids and their metabolites was determined using the high-resolution HPLC-MS/MS method as part of a targeted metabolomic analysis in the blood plasma of chemical synthesis operators and the comparison group. For the operator group, a strong correlation ($r > 0.8$) was established between the content of BTEX metabolites in the urine and the content of indoleacetic acid in the blood plasma, which was recently found to be an activator of aryl hydrocarbon receptors (AhR) and can be considered as a candidate biomarker of effect of aromatic hydrocarbons.

TWO TYPES OF SENSOR DEVICES THAT ALLOW SELECTIVE ANALYSIS

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Compact, low-cost automated sensor analyzers for gases or liquids can provide continuous monitoring in hard-to-reach areas. These devices can be roughly divided into two types. The first of them uses highly selective sensors. In this case, each of the possible analytes is determined by its sensor. The advantage of this type of device is ease of calibration, the ability to simply replace one of the sensors in the case of a malfunction without the need to recalibrate the entire system. The disadvantage of this approach is a very limited choice of highly selective sensors. Our group has developed highly selective gas sensors for hydrogen sulfide¹ and hydrogen².

The second type of devices (“electronic nose”, “electronic tongue”) is based on the use of low-selective sensors. In this case, analyzers must include several sensors combined into a single system (sensor array) or even one low-selective sensor operating in a non-stationary mode, and thereby providing additional information about the analyzed environment. The advantage of this type of device is the possibility of selective analysis of the environment due to a single sensor, which reduces energy consumption and increases the battery life of the device. However, to implement this approach it is necessary to solve a number of scientific and technical problems. In particular, it is necessary to develop special algorithms for processing multidimensional data. In the work of our group, gas mixtures were analyzed using a single low-selective semiconductor sensor³, existing data processing algorithms were improved and fundamentally new ones were developed.

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AUTOMATION AND MINIATURIZATION OF CHEMICAL ANALYSIS USING DEEP EUTECTIC SOLVENTS

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Deep eutectic solvents (DES) are a new type of promising solvents formed from two or more low-fusibility components that together form a liquid with a lower melting point than each of the individual components. These solutions have unique physicochemical properties and attract the attention of researchers in various fields. DESs are widely used in chemistry, biology, pharmaceuticals and other fields due to their ability to replace traditional organic solvents. Environmental safety, ease of preparation in the laboratory, and low cost of DESs have led to the fact that they have recently found increasing use in analytical chemistry. DESs are most widely used in analytical chemistry in microextraction methods for isolating analytes of various natures from various and complex objects of analysis.

The report will discuss both the analytical capabilities and limitations of DESs in microextraction approaches such as classical liquid-liquid microextraction, droplet, dispersion, homogeneous, membrane and other types of microextraction, as well as issues of formation, destruction and stability of DESs. The report will also present the latest trends in the field of automation of chemical analysis using deep eutectic solvents, as well as problems and limitations associated with the use of SER in chemical analysis and the main prospects for the development of this area.

The work was carried out with financial support from a grant from the Russian Science Foundation (project No. 22-73-10039). Scientific research was carried out in the resource centers of the St. Petersburg State University Science Park "Methods for analyzing the composition of matter, Thermogravimetric and calorimetric research methods, Center for diagnostics of functional materials for medicine, pharmacology and nanoelectronics, Prototyping Department."

NEW DERIVATIZATION REAGENTS FOR HPLC-MS – ISSUES AND PERSPECTIVITIES

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The development of modern analytical instrumentation already provides analysts with often redundant opportunities for identification and quantitative analysis; the question of the analysis of many biogenic compounds present at the nanogram level still remains open due to the need for preliminary purification and concentration of samples to get rid of matrix components and increase sensitivity, efficiency and selectivity in determining biogenic components using HPLC-MS.

This report discusses the main approaches to the derivatization of biogenic amines and steroid hormones for their further HPLC-MS determination. The main difficulties, prospects and expected vectors for the development of analytical derivatization are shown both for the purposes of targeted analysis and during non-targeted screening.

Using the considered examples of new derivatizing agents, it is shown that despite the increased sensitivity, the sample preparation procedure can often become significantly more complicated, which will be a negative factor that increases the risk of obtaining unreliable data. In addition, it has been shown that even for a number of promising derivatizing reagents, great difficulties arise with the stability of both derivatives and parent compounds.

The presented work notes the ambiguous influence of the derivatizing reagent on the retention parameters of analytes. Particularly challenging are potentially promising reagents that can form not only several variants of derivatives (mono-, di- and tri), but also form isomers (for example, syn- and anti-forms), which is clearly demonstrated by the previously described steroid hormone oximes and hydrozine derivatives.

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CURRENT STATE OF SPORTS METABOLOMICS

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The use of high-intensity and oftentimes extreme training loads is one of the required conditions for achieving the highest sports results among top qualified athletes. To date, the investigation of adaptation processes to training loads in athletes has been built upon studying the reaction of one or several main metabolites¹. Obviously, this approach is possible to detect only limited changes in the leading body systems, which may hamper the understanding fundamental links between physical exercise and metabolism. For this purpose, it is relevant to incorporate metabolomic approaches into the biochemical monitoring practice of elite athletes, making it possible to systematically identify the response of metabolic pathways to physical stress by detection, identification and quantification broad range of endogenous metabolites in high complexity matrices using chromatography and mass-spectrometry methods. Despite the great capabilities offered by metabolomics approaches their implementation and application in sports sciences began as few as several years ago².

The presentation provides a detailed description of contemporary metabolomic approaches for the quantification of endogenous bioindicators using chromatography-mass spectrometry methods as well as a comparative analysis of described techniques, their advantages and limitations. Metabolomic approaches aimed at validation of biomarkers assays associated with negative consequences of excessive training loads are specified with the demonstration of their high prognostic potential.

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METABOLIC PROFILING OF BIOACTIVE SUBSTANCES IN NATURAL OBJECTS BY CHROMATOGRAPHY AND CAPILLARY ELECTROPHORESIS USING IONIC LIQUIDS

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An important task of modern analytical chemistry is the acquisition and chemometric processing of chromatographic and/or electrophoretic profiles of bioactive substances (BAS) and the identification of potential dominant biomarkers in natural objects that are complex matrices. In this regard, it is important to develop both effective methods for extracting and concentrating trace amounts of BAS, as well as to search conditions for the selective and highly sensitive determination of analytes. Interesting prospects are opening up with the use of materials with adjustable properties, the so-called “*smart materials*”. These include ionic liquids (ILs). The aim of this study was to evaluate the analytical capabilities of imidazolium-based ILs for the selective extraction, concentration and separation of bioactive substances in natural objects.

A unique feature of imidazolium-based ILs is the ability to undergo various types of interactions with analytes, which provides them with great potential as multifunctional modifiers of chromatographic and electrophoretic systems. The use of hydrophilic ILs as a dynamic modifier of the C18 stationary phase allows the realization of the hydrophilic chromatography mode for the determination of highly polar analytes in biological objects. Interesting prospects for the use of imidazolium ILs as components of micelles and microemulsions have been revealed in MEKC and MEEKC for the separation of analytes of hydrophilic (*catechins*) and hydrophobic nature (*steroid hormones, polyphenols*). The introduction of ILs into the composition of pseudostationary phases contributed to a significant increase in the efficiency and selectivity of the separation of analytes, providing the possibility of *on-line* preconcentration to reduce LOD. Due to their low boiling points and the ability to change the physicochemical properties of ILs over a wide range based on the nature of the cation and anion, ILs are in some cases an excellent alternative to volatile toxic organic solvents in extraction techniques for bioactive substances. We have revealed the potential of ILs as effective extractants of bioactive substances (*steroid hormones, pesticides, polyphenols*) in dispersive liquid-liquid and magnetic solid-phase microextraction (the recovery of the analytes were 64–99%).

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MONITORING OF CATALYTIC REACTIONS BY DESORPTION/IONIZATION MASS SPECTROMETRY

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Online monitoring of reactions by mass spectrometry is a rapidly developing field of organic chemistry, allowing the study of chemical process pathways through the detection of their intermediates. The main tool for such monitoring is electrospray ionization mass spectrometry (ESI-MS). The design of the ESI source enables the direct introduction of the reaction medium into it and the direct detection of reaction intermediates. However, the use of this approach may also involve oxidative-reductive processes during the spraying of analyte solutions, which can significantly distort the observed picture. An alternative to this monitoring method can be mass spectrometry with desorption ionization methods - matrix-assisted laser desorption/ionization (MALDI) and "direct analysis in real time" (DART). In this work, these ionization methods were applied for the detection of intermediates of two reactions –telomerization of isoprene with methanol and additive polymerization of norbornene derivatives catalyzed by palladium complexes. The obtained results were compared with data acquired by ESI-MS.

Comparative analysis of the obtained mass spectral data arrays showed their noticeable difference. MALDI and DART mass spectra of the reaction media of both processes contained ion peaks corresponding to the coordination by the catalyst of various numbers of substrate molecules. The relative intensity of these signals changed over time, indicating the progress of the reaction and the formation of its final products. In the case of ESI-MS, the appearance of mass spectra did not depend on time, detecting only ions that are the main intermediates of the reactions, which apparently is a consequence of their formation directly in the ion source during spraying of the reaction medium.

DEVELOPMENT OF A METHODOLOGY FOR EXTRACTION NANOPARTICLES FROM VOLCANIC ASH

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Volcanic ash nanoparticles are tiny particles ranging in size from 1 to 100 nm, they can travel long distances and penetrate biological systems, which underscores the importance of studying them. One of the most important features of volcanic ash nanoparticles is their high specific surface area, which allows them to sorb various substances, including toxic ones. The study showed that the concentrations of a number of elements (Ni, Zn, Cd, Ag, Sn, Se, Te, Hg, Tl, Pb, Bi) in volcanic ash particles are 10-500 times higher than the total content of these elements in the bulk samples.

The study of volcanic ash nanoparticles is an interdisciplinary task that requires the use of a complex of sample preparation and analysis methods. The main problem in studying volcanic ash nanoparticles is the low efficiency of extracting nanoparticles from samples. In this work, the method of field-flow fractionation in a rotating coiled column (RCC) for the extracting of nanoparticles from polydisperse samples, proposed at the Vernadsky Institute of Geochemistry and Analytical Chemistry of the Russian Academy of Sciences, was developed.

The effectiveness of six different techniques and two eluents: deionized water and sodium pyrophosphate for the extraction of nanoparticles from an ash sample from the Karymsky volcano (2013 eruption) using RCC was assessed.

Extracting with pyrophosphate was researched in three different variants: with soaking in 2 mM $\text{Na}_4\text{P}_2\text{O}_7$ for 24 hours and preliminary soaking in 0.1 M NaCl, without soaking in NaCl and without preliminary soaking in pyrophosphate. To compare the effectiveness of the methods, the extraction of nanoparticles of volcanic ash in deionized water was also represented. As a result, preliminary soaking in sodium pyrophosphate and its use as an eluent turned out to be optimal and provide the greatest isolation efficiency.

Using this technique, became possible to increase the mass of the extracting nanofraction by 10 times (from 0.1 mg to 1 mg) compared to the extracting process in deionized water. It has been shown that with elemental analysis of nanoparticles it has become possible to determine a number of elements below the limit of detection (Ag, Cs, Tu, etc.).

Expanding the possibilities for studying the elemental composition of volcanic ash nanoparticles is promising for solving fundamental problems in geochemistry.

DETERMINATION OF IMPURITIES IN COBALT AND NICKEL BY ICP-AES AFTER COPRECIPITATION WITH SCANDIUM HYDROXIDE

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The existing standardized atomic spectrometry applications designed for the cobalt and nickel analysis: GOST 13047, GOST 6012, GOST 8776, GOST R ISO 7523, do not correspond to modern and near future specifications to provide the impurities determination in these metals.

In order to improve the quantity determination limits of impurities presenting in cobalt and nickel, their coprecipitation on scandium hydroxide was investigated.

2.5 g Cobalt and Nickel weights were dissolved in solutions of nitric acid diluted 1:1. 4 ml of Scandium nitrate solution (500 mg/L) was introduced and an ammonia solution was added so that Cobalt and Nickel ammonia complexes were formed in the solution, on the one hand, and a precipitate of scandium hydroxide, on the other. The precipitate was filtered, rinsed several times with 1% ammonia solution and then dissolved in hydrochloric acid.

Impurities coprecipitation on scandium hydroxide allowed the following impurities to be separated from Cobalt and Nickel: Fe, Cr, Mn, P, As, Bi, Sb, Sn, Te.

The analysis of the obtained solutions was performed by inductively coupled plasma atomic emission spectrometry (ICP-AES) on iCAP 6500 Duo and iCAP PRO XP Duo spectrometers manufactured by Thermo Fisher Scientific (USA).

The limits of the quantity determinations of impurities in Cobalt and Nickel by ICP-AES after coprecipitation with scandium hydroxide are at the level of 0.01-0.1 ppm.

These impurities concentrating way was used to develop a method for determining impurities in Cobalt and Nickel. It can complement the existing GOST 6012 and GOST 8776, as well as replace GOST R ISO 7523, which uses the method of atomic absorption spectrometry with electrothermal atomization, which is characterized by insufficient sensitivity, low productivity, the use and rapid deterioration of expensive consumables.

SORBENTS SPECIFIC TO ZEARALENONE BASED ON SILICA MODIFIED WITH MOLECULAR IMPRINTING POLYMERS

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Sorption materials based on organosilanes are widely used to solve various problems in science and technology.^{1,2} High performance qualities, environmental safety, unique structural characteristics (stable structure, high specific surface area, adjustable porous structure) and the possibility of targeted modification, due to the presence of surface silanol groups, are the undeniable advantages of silica based materials. An effective solution for the creation highly selective sorption materials is the modification of the silica surface with molecular imprinted polymers (MIP).

The work considers the conditions for obtaining a sorption material for the selective extraction of the mycotoxin zearalenone (ZEA), which is a contaminant of grain crops. The influence of various factors was studied and optimal conditions for the synthesis of ZEA-selective MIP on the surface of commercially available Aerosil® 200 microparticles by the core-shell method using (3-aminopropyl 3-ethoxysilane) were selected. The possibilities of selective to ZEA MIP synthesis and their sorption properties obtained by using ZEA templates and its structural analogues as molecules have been studied. The sorption properties of materials based on them were compared.

The quantum chemical study of the possibility of association between functionalized monomers of organosilanes with various structure and template molecules has been carried out. It was shown that the formation of associates between template molecules and organosilanes can be caused by hydrogen bonds between carbonyl and hydroxyl groups of template molecules with hydrogen atoms in organosilane molecules.

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DIFFICULTIES IN INTERPRETING THE RESULTS OF STUDIES ON HIGHLY GLYCOSYLED PROTEINS

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Researches on biotherapeutic proteins are becoming more complex every year, as are their role as drugs for the treatment of cancer, orphan and autoimmune diseases. The increasing complexity of such studies is associated both with increasing regulatory requirements^{1,2} and with the increasing complexity of the structures of molecules used for therapy. Many of the modern drugs are no longer classical proteins, such as immunoglobulins or insulin, but hybrid structures. Often, in these types of protein structures, the main contribution to their heterogeneity is determined by oligosaccharides (N- and O-glycans). Considering the possibility of attaching hundreds of different glycans to one protein region, the overall heterogeneity of, for example, erythropoietin can be estimated at a four-digit number of variants.

The combination of modern methods for studying the profile of protein glycosylation (HPLC-MS with various types of chromatography, CE-MS, HPLC with fluorescent detection, etc.) allows one to obtain heterogeneous results, which often leads to serious difficulties in their interpretation. For example, when studying the sites of protein glycosylation and assessing the population of oligosaccharides at the peptide level, gained information does not correlate with the obtained spectra of the native protein, as well as the results of studying its subunits. Thus, interpreting a body of data for even a single drug often requires weeks of work by several highly trained analytical chemists.

In recent years, the scientific team of the Laboratory Complex of the Sirius University has developed several methods for quality control of biotherapeutic proteins and also conducts experimental and theoretical studies to overcome these difficulties.

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INVESTIGATION OF THE ELEMENTAL COMPOSITION OF MINERALS AND FUNCTIONAL MATERIALS (LA-ICP-MS)

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The laser ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS) is a unique method for local analysis that allows studying mineral grains *in situ*. However, the domestic practice of using the LA-ICP-MS aircraft to solve these problems is not so wide. In geochemistry, the accessory minerals zircon and baddeleyite are of interest for studying the distributions of rare and rare earth elements (REE), as well as ore minerals - sulfides for determining elements of the platinum group (PGE), REE, etc. We have proposed a method for spectrometric analysis of solid material [1] based on the use of a single standard for constructing a calibration, for example, NIST SRM 612 (with control of the parameters of the obtained craters), which allows to expand the range of concentrations of the elements to be determined. This approach made it possible to develop methods for the determination of rare and REE in zircon, baddeleyite, as well as PGE, REE, Cu, Co, Ni, As, Sb, Se, Te, Bi, Sn, etc. in ore minerals - pyrite, pentlandite, pyrrhotite and other sulfides and minerals of PGE. The ELAN 9000 DRC-e quadrupole mass spectrometer was used for measurements, and ablation was performed using the UP-266 MACRO unit. Information on the concentrations and distribution of elements in minerals is important for studying the sources of the origin of metals and understanding the conditions for the formation of complex deposits.

A method has been developed for determining the isotope ratios of $^{231}\text{Pa}/^{235}\text{U}$ in baddeleyite samples by the method of LA-ICP-MS. The international BCR-2 sample was used as a reference material.

The LA-ICP-MS is used to study functional materials – germanium glasses with a gradient change in composition, insoluble titanium anodes, tantalum powder, doped lithium niobates and lithium tantalates, etc.

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METAL IONS EXTRACTION BY IONIC LIQUIDS BASED ON IONIC SURFACTANTS

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Ionic surfactants are widely used for concentrating metal ions, however, traditional methods for producing stratified systems (heating aqueous solutions, salting out with inorganic salts and acids) are of limited applicability for them, therefore it is advisable to obtain ionic associates of ionic surfactants with counterions having low hydration energy.

The possibility of obtaining stratified systems by salting out alkylbenzyltrimethylammonium chloride (Catamine AB, $[C_nH_{2n+1}N^+(CH_3)_3CH_2C_6H_5]Cl^-$, $n=10-18$) by the action of bromides, iodides, perchlorates and thiocyanates of alkali metals has been studied. The possibility of quantitative extraction of thallium (III) has been demonstrated. In a system with sodium iodide, as well as zinc, copper and cobalt in a system with potassium thiocyanate according to the anion exchange mechanism.

Salicylate or sulfosalicylate ions can also be used as an anion in the formation of ionic liquids. The maximum degree of extraction of iron (III), zinc, cobalt and copper (II) in a system with sulfosalicylic acid is observed at a pH of more than 10; for a system with sodium salicylate, the optimal pH is in the range of 3–9. Extraction of metal ions is accompanied by the formation of salicylate and sulfosalicylate complexes of metal ions.

The possibility of developing extraction systems based on sodium dodecylsulfate or sulfonol ($C_nH_{2n+1}C_6H_4SO_3Na$, $n = 12-18$) and antipyrine (1,2-dihydro-1,5-dimethyl-2-furyl-3H-pyrazol-3-one) has been studied. In the presence of inorganic acids. A study of the distribution of thallium (III), indium, gallium, iron (III) and lanthanum ions showed that at low acidity the studied metal ions are extracted in the form of complex compounds with antipyrine by a coordination mechanism, and at acidity more than 2 mol/l by an anion exchange mechanism in the form of halide acid complexes with the antipyrinium cation.

CONTINUOUS-FLOW SEPARATION AND PRECONCENTRATION OF MICROPLASTICS FROM FRESH AND SEA WATERS USING “WATER-OIL” SYSTEMS

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Aquatic ecosystems are the main sink for microplastics entering the environment. Estimation of the content of microplastics in natural waters is necessary for the assessment of pollution of water bodies and potential risks for their inhabitants. As a rule, the content of microplastics in natural waters is low, so the development of new effective particle separation methods that allow one to selectively separate and preconcentrate microplastics from natural waters is an urgent task. This study is devoted to the development of a new method of separation and preconcentration of microplastics from natural waters in a rotating coiled column (RCC) in countercurrent chromatography mode using two-phase water-oil liquid systems. The retention parameters of systems based on vegetable (castor, tung, linseed, soybean, sunflower, sesame, rapeseed, olive, turpentine, limonene) and synthetic (vaseline and motor) oils in RCC are determined and the applicability of these systems for separation of microplastics from water samples is assessed. The effect of RCC rotation speed and mobile phase flow rate on the retention of the oil phase in the column is studied. By the example of polyethylene microparticles of various sizes (40–63, 63–100, and 100–250 μm), the high efficiency of microplastics separation (about 100%) in RCC using water-oil systems is shown. It is found that the efficiency of separation does not depend on the salinity of water samples, so the proposed method can be used for separation of microplastics from both fresh and sea waters. The developed method of separation and preconcentration of microplastics is promising for treatment of wastewater from microplastics.

The work was carried out with the financial support of the Russian Science Foundation, project 23-13-00263.

APPROACHES TO THE CREATION OF MIXED-MODE STATIONARY PHASES FOR LIQUID CHROMATOGRAPHY BASED ON POLYMERIC SUBSTRATES

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A relatively new direction in the design of stationary phases for liquid chromatography is the development of mixed-mode adsorbents, which retain analytes by several mechanisms simultaneously. They allow to solve a wider range of problems than classical phases, which are suitable for operation in only one chromatographic mode. However, the disadvantage of the vast majority of mixed-mode adsorbents is the limited range of pH stability associated with the use of silica as their substrate. The use of substrates that are stable over the entire pH range in the synthesis of such adsorbents will lead to the possibility of their use with strongly acidic and strongly alkaline eluents. In addition, adsorbents with strong ion exchange groups can be used in ion chromatography with suppression of background electrical conductivity — a sensitive method for the determination of ions.

This work evaluates the prospects of approaches for the covalent fixation of different functional layers to create mixed-mode adsorbents based on polystyrene-divinylbenzene. Among the approaches studied are hyperbranching as well as grafting of polyethyleneimine followed by quaternization of its amino groups with glycidol or anchoring of polyelectrolyte chains formed *in situ* from a secondary amine and diepoxide. The developed mixed-mode phases allow operation in reversed-phase and hydrophilic modes, as well as in ion-exchange chromatography, including suppression ion chromatography.

The possibility of separating multicomponent mixtures of anions was demonstrated in the ion chromatography mode with suppression, including inorganic, highly polarizable anions, and organic acids anions; in the hydrophilic mode — mixtures of nitrogenous bases and nucleosides, water-soluble vitamins, amino acids, sugars and sweeteners; and in the reversed-phase mode — alkylbenzenes, phenols and fat-soluble vitamins. The work demonstrates the applicability of the resulting adsorbents for the analysis of a wide range of real objects.

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DEVELOPMENT OF A COLORIMETRIC SCALE FOR GRADATION-FREE ANALYSIS OF BIOLOGICAL FLUIDS USING OPTICAL SENSORS

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One trend in analytical chemistry is the miniaturization of measurement devices. This trend is reflected in the active development and application of bulk ion-selective colorimetric sensors (optodes) that are only a few millimeters in size. Optodes offer the advantage of non-invasive analysis using remote recording of the analytical signal.

One significant disadvantage of these sensors is the need for frequent recalibration due to photodegradation of components in the sensor phase and their leaching into aqueous solution.

To address this issue, this work proposes the creation of a colorimetric scale consisting of optodes with a fixed value of the analytical signal included in the sensor array (see Fig. 1A). The study investigated arrays of polymer optodes that are selective to Na^+ and Cl^- and can be used for sweat fluid analysis. Using the results of the initial one-time calibration, we established an internal correspondence between the analytical signal and analyte content. This allowed us to perform further calibration-free analyses (see Fig. 1B).

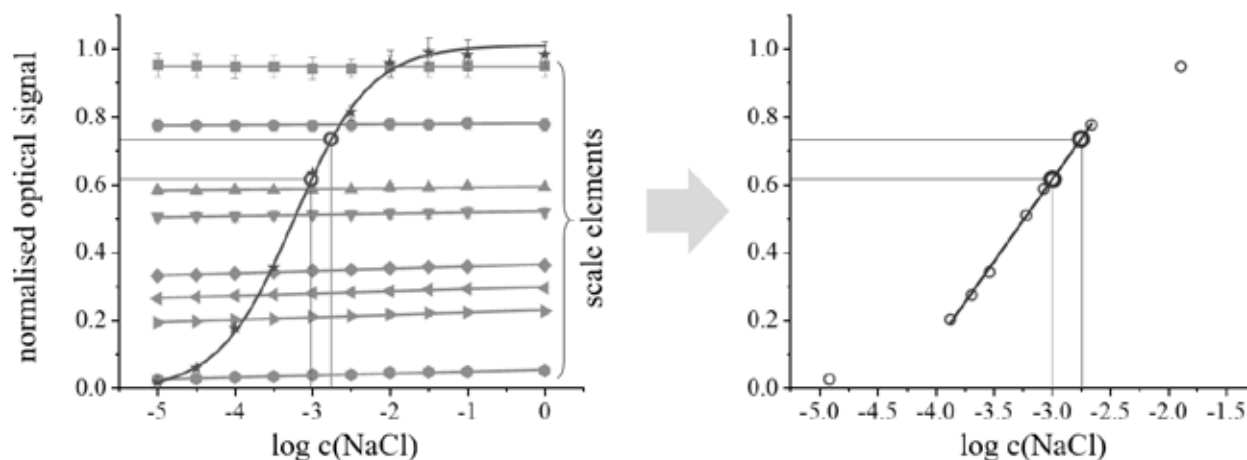


Figure 1. Obtaining a colorimetric scale for analysis.

The study demonstrated the applicability of the colorimetric scale for analyzing unknown samples and as part of a sensor device.

The work received financial support from RNF, project No. 20-73-10033P.

ULTRAFAST TRANSMISSION ELECTRON MICROSCOPY AND DIFFRACTION: PROGRESS FROM SUBNANOSECOND LASER—INDUCED STRUCTURAL DYNAMICS TO FEMTOSECOND QUANTUM TOMOGRAPHY

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Throughout its centuries-long history, transmission electron microscopy has been a powerful tool for studying matter that is predominantly in equilibrium or in states close to it. However, to obtain a complete picture of the subject being studied, it is also important to study its dynamic properties. Time-resolved laser spectroscopy has opened up the possibility of observing a wide range of fast processes related to “ultra-fast science”, but the diffraction limit in optics seriously limits the possibilities of a purely optical experiment.

Under the influence of powerful laser pulses, a “restructuring” of the substance occurs. Structural dynamics manifests itself in an atomic-molecular film, the detection of which is of great interest both from the point of view of fundamental science and in the applied aspect. To directly observe laser-induced structural changes, it is first necessary to ensure high spatiotemporal resolution. The use of very short electron (or X-ray) pulses synchronized with the “optical triggering” of fast processes in the sample is the key to solving this problem.

The use of short photoelectron pulses, pioneered in the 1980s, opened up the possibility of studying structural dynamics with high spatiotemporal resolution. The combination of nano-pico-femtosecond lasers with electron-based technology has become extremely fruitful for observing the behavior of atoms and molecules at their natural scales. In imaging mode, this concept soon led to the creation of 4D transmission electron microscopy. In the electron diffraction mode, a unique opportunity has appeared to shoot atomic-molecular movies. The method has high sensitivity combined with a low level of radiation damage to the test sample. In contrast to the X-ray free electron laser, this made it possible to conduct experiments on very thin films of promising materials in small-scale facilities in standard laboratories. Quantum tomography has opened a new page in the study of matter using short electron bunches. Here we review the development of ultrafast transmission electron microscopy and diffraction techniques that enable detection of structural dynamics on ultrashort time scales.

THE EXTRACTION/GC-MS DETERMINATION OF ALKYLATED DIPHENYLAMINES IN ENGINE OILS

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Alkylated diphenylamines are important antioxidant additives for engine oils that are subjected to weathering degradation. The GC-MS chromatograms of engine mineral oils contain one large unresolved complex hump that is due the presence of hundreds compounds as well as alkylated diphenylamines which cannot be resolved by conventional chromatography^{1,2}. Thus, an identification of alkylated diphenylamines and then environmental forensic investigations in terms of determining the source of weathered engine oils, differentiating oils and monitoring the weathering state of oils under the influence of various conditions are quite a difficult tasks.

In this study it was found, that the use of preliminary selective liquid extraction of alkylated diphenylamines from oils by acidified acetonitrile allows to obtain pure extracts of amines. GC-MS chromatograms of such extracts (Fig. 1) can be easier interpreted by the visually comparing obtained distribution pattern with published data^{3,4}. Therefore, characterization and distribution of studied additives generate important information for environmental forensic investigations.

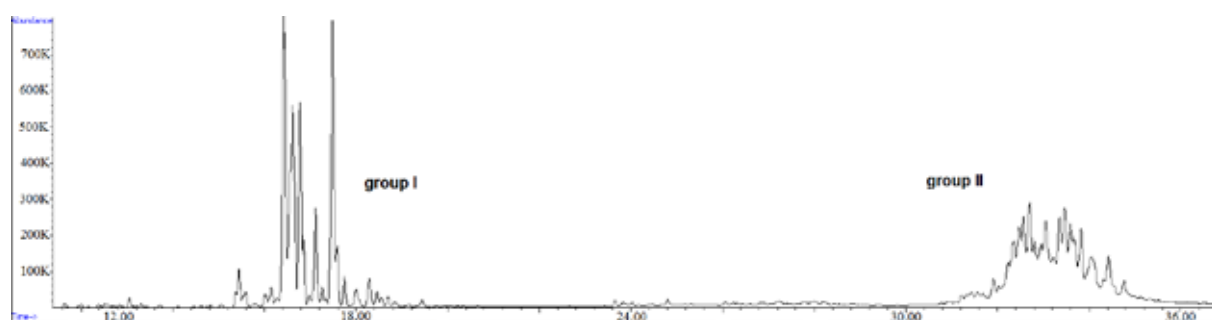


Fig. 1 GC-MS distribution pattern of alkylated diphenylamines.

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ADAPTATION OF A LOW GAS LOAD MASS SPECTROMETRIC ION SOURCE FOR CONNECTION WITH MULTICAPILLARY COLUMNS

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A small-sized analytical platform based on a miniature mass filter and ion source with atmospheric pressure chemical ionization (APCI) with a low gas load is being developed in the Laboratory of Field Analytical and Measurement Technologies of IPGG SB RAS for use in field conditions¹.

Previously, when determining sulfur-containing compounds in the air at the MPC level, the detector was interfaced with a thermal desorption input system and a gas chromatographic (GC) separation system with short capillary columns². Due to the high gas dynamic resistance, the use of capillary columns has limitations in technical implementation. It seems promising to use multicapillary columns (MCC) in this scheme for GC separation. Technical solutions have been proposed and an adaptation of a low gas load APCI mass spectrometric ion source has been performed for connection to a MCC, which ensures operation with wide range of carrier gas flow. Theoretically and experimentally, the effect of the carrier gas flow on the ionization efficiency of analytes has been shown. The improvement of the characteristics of the adapted analytical platform with a MCC, such as speed, sensitivity, aftereffect and detection limit, is shown.

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The work was carried out within the framework of the Program of Fundamental Scientific Research of the Russian Federation: the project “New technologies for extracellular chemical analysis and control, precision measurements of physical fields of natural and man-made objects” FWZZ-2022-0027.

PHYSICAL FIELDS IN SAMPE PREPARATION METHODS

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Physical fields (gravitational, magnetic, ultrasonic, electric and others) can be used for intensification of sample pretreatments in analysis of different samples.

Modern trends in the application of physical fields in sample preparation methods are noted in this work. Application of physical fields in sample preparation methods leads to increase the efficiency of traditional extraction and sorption methods of concentration and separation of elements in analysis of different objects. The prospect of their application is confirmed by many publications in various fields of science.

Along with a literature review, the report will present the results of recent work by employees of the Laboratory of concentration methods in Vernadsky Institute of Geochemistry and Analytical Chemistry Russian Academy of Science.

For the first time the application of ultrasonic fields in the standing wave mode (suspension columns) for the analysis of environmental objects (soils, solutions and others) and medical samples was proposed. It has been shown that the combined use of ultrasonic and magnetic fields makes it possible to isolate DNA from soils with a high content of organic substances.

It has been demonstrated using many examples of testing real field samples (oil, petroleum products, field layers) that the application of wave fields (magnetic and ultrasonic) at the sample preparation stage changes the physicochemical properties of the analyzed objects. They make it possible to separate water-oil emulsions into separate fractions and change the viscosity of petroleum samples.

Particular attention was paid to the application of wave effects to intensify extraction (including using microchannels) and sorption processes. Magnetic particles (including those modified with ionic liquids and deep eutectic solvents) in constant and alternating fields are used to intensify the sorption process. The behavior of particles is controlled in flow cells of a special design.

The presented results can be the basis for the development of new high efficient industrial processes.

This work was performed within the framework of the government assignment for the GEOKHI RAS.

ULTRA-SENSITIVE RAPID METHODS FOR REGISTRATION OF TOXINS IN FOOD AND MARKERS OF CARDIO DISEASES IN SALIVA

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Through the use of magnetic nanoparticles (MPs) as markers of biochemical reactions in combination with electronic methods for detecting such nanolabels by nonlinear magnetization reversal ^{1,2}, rapid immunochromatographic methods for measuring ultra-low concentrations of mycotoxins in food products have been developed. In particular, detection limits of 2.3 pg/ml zearalenone (ZEA) and 11 pg/ml ochratoxin A (OTA) have been achieved with a 20-minute analysis time. The experiments confirmed the high specificity and reproducibility of the analyzes, as well as high accuracy for low and high concentrations of toxins in grain flour samples. This is due to the extremely high slope of the calibration plots: within a concentration range of 5 orders of magnitude, the electronic detection signal for MPs changes 1000 times.

To further increase sensitivity and reduce analysis time, original biosensor systems have been developed based on field-effect transistors with graphene coating and aptamers that selectively interact with mycotoxins or biomarkers of a number of diseases. This approach made it possible to measure ultra-low concentrations of ochratoxin A in wine with a record detection limit of 1.4 pM and a response time of 10 s. On this basis, miniature immersible aptasensors have been developed for the invasive analysis of natural human fluids, such as saliva, where the concentration of markers is 1000 times less than in blood. The developed aptasensors have high selectivity and sensitivity for the rapid detection of disease markers, in particular, cardiac markers for diagnosing heart failure at a record concentration level of 41 fg/ml (~ 4.6 fM) ⁴. Besides, in the presence of corresponding aptamers at surface ⁵ such aptasensor can serve as an effective tool for monitoring a variety of chemical active compounds and biomarkers of a wide range of diseases.

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TWO IN ONE: ELEMENTAL ANALYSIS ON A MÖSSBAUER SPECTROMETER

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Mössbauer spectroscopy is a popular method for analyzing the chemical states of resonant atoms in solids. This method has found wide application in materials science, medicinal chemistry, geology, biology, and mineralogy. Generally, the information obtained by Mössbauer spectroscopy is enhanced by data about the elemental composition of samples. To solve this problem, additional methods such as, for example, X-ray fluorescence analysis (XRF) are employed, because it allows direct quantitative determination of elements in solids. Noteworthy, both Mössbauer and XRF spectrometers employ similar experimental schemes, including similar components for generating and registering electromagnetic radiation. However, commercial Mössbauer spectrometers usually use scintillation or proportional counters as detectors, which have higher registration efficiency than semiconductor detectors, while XRF spectrometers use semiconductor detectors with higher resolution. This similarity of spectrometer units allowed us to hypothesize that a Mössbauer spectrometer could be used for direct registration of XRF spectra, but with worse resolution compared to spectra obtained from commercial XRF spectrometers. This problem can be solved by using chemometric data processing algorithms. In the present study the possibility of elemental analysis with the help of Mössbauer spectrometer was tested on the example of quantification of Ti, Cr, Mn, Ni, Nb and Mo in real steel samples. The obtained results were compared with the results of quantitative analysis obtained on a traditional XRF spectrometer.

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QUERCETIN-IMPRINTED PHLOROGLUCINOL-MELAMINE-FORMALDEHYDE RESINS

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Molecularly imprinted polymers, including hydrophilic molecularly imprinted resins (MIR), have been actively used to increase the selectivity and sensitivity of the extraction of biologically active compounds from natural objects. In this work, we investigated quercetin (Qu) imprinted phloroglucinol-melamine-formaldehyde resins (PMF), obtained by bulk polymerization (MIR) and on the surface of supports such as nanostructured silica or silanized magnetic nanoparticles (SMIR). The PMF resins were obtained by polycondensation of phloroglucinol and melamine (monomer ratio 3:1) with formaldehyde in the presence of porogen – polyethylene glycol (PEG) of various average molecular weight (M_w 6000-10000 Da).

An increase in the recovery and sorption capacity of PMF MIR was observed with an increase in the average molecular weight of PEG. Thus, for the PMF-10K MIR sample synthesized in the presence of PEG 10000 the maximum values of the recovery and sorption capacity reached ~43% and 4.7 $\mu\text{mol Qu/g}$, respectively. The maximum imprinting factor was 1.4. The maximum values of the recovery and sorption capacity of PMF SMIR were observed for PMF@SiO₂ MIR obtained on the surface of nanostructured silica: ~37% and 3.8 $\mu\text{mol Qu/g}$, respectively. The maximum imprinting factor increased to ~1.6.

The kinetics of quercetin adsorption by PMF MIR and SMIR are better described by a pseudo-second order kinetic model. Analysis of kinetic data within the framework of diffusion adsorption models indicates a mixed-diffusion sorption mechanism. Quercetin adsorption isotherms of PMF MIR are better described by the Dubinin-Radushkevich equation in which adsorption follows a micropore filling mechanism that may be attributed to the small pore size of PMF resins (≤ 2 nm).

The work was carried out with the financial support of the Government of the Khanty-Mansiysk Autonomous Okrug - Ugra (project 2023-227-28) and the world-class West Siberian Interregional Scientific and Educational Center.

LIQUID-LIQUID EXTRACTION INTO SUPRAMOLECULAR SOLVENTS

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Supramolecular solvents are nanostructured liquids formed from molecules or ions of amphiphilic surfactants as a result of two consecutive processes: aggregation of amphiphiles, when the critical micelle concentration is exceeded, and coacervation. These extractants allow to separate both polar and non-polar substances due to electrostatic, van der Waals and π - π interactions, formation of hydrogen bonds, which causes their active application in analytical chemistry. Such diversity is provided due to a variety of functional groups and different polarity regions within the supramolecular aggregates. In addition, solvents of this class can be referred to designer extractants, since their properties can be controlled within a wide range by varying the nature of the amphiphile, dispersion medium, coacervation agent, and the concentrations of reagents introduced into the extraction system.

Liquid-liquid extraction into supramolecular solvents is also referred to as micelle-mediated extraction. Its first step usually consists of introducing an amphiphile into the sample, resulting in an isotropic solution containing micelles or vesicles. The second step involves the introduction of a certain substance (coacervation agent) or a change in the temperature of the system, which causes the formation of an extractant phase with the release of analytes into it. It is worth noting that the obtained phase can also be used in the extraction of target substances from solid samples, which expands the field of application of such solvents. The considered method of separation and preconcentration is also widely used in miniaturized and automated modes. The main aspects of formation of supramolecular solvents, peculiarities of extraction processes with their participation and the latest trends in this field of research will be presented during the talk.

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ION-SELECTIVE OPTICAL SENSORS: PRINCIPLES OF RESPONSE GENERATION AND TUNING RESPONSE CHARACTERISTICS

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Ion-selective optical sensors (ISOs), also known as optodes, are highly selective, sensitive, and easily miniaturized tools used in various fields of science and technology. The signal of an ISO is generated by the ionic transport between the optode phase and the solution, and depends on either the ratio or the product of the activities of two ions: the analyte and the paired ion. In [1], a potential solution to this problem was proposed. The study showed that to create an optode sensitive to individual ionic activity, it was necessary to stabilize the Galvanic potential difference at the optode-solution phase boundary. This was achieved by adding a moderately lipophilic organic electrolyte (MLOE) to the membrane, which stabilizes the interfacial potential by distributing between the phases. However, the classical theory cannot be used to describe or predict the response of such optodes [2].

In this contribution, we propose a theoretical model capable of predicting and characterization of the response of both MLOE-containing sensors and conventional optodes [3]. This model allowed us to predict the influence of the nature of the active components of the optode (ion-selective ligand, lipophilic pH indicator, and MLOE) and their content on the range of the optical response. This opens an avenue for creating optical sensors with predetermined characteristics. Additionally, ionic distribution coefficients of MLOE ions between the aqueous and membrane phases could also be estimated.

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VOLATILE ORGANIC COMPOUNDS AND PROTEINS AS MARKERS OF IRRADIATION OF BIOLOGICAL OBJECTS

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Irradiation can solve a wide range of problems ranging from inhibition of root germination during storage to suppression of pathogenic microflora of food products. Exposure in doses corresponding to the optimal dose range allows inactivation of pathogenic microorganisms while preserving the surrounding molecular structures. Studies conducted by scientists from the Chemistry Department and Physics Department of Moscow State University are aimed at establishing biochemical markers of irradiation of biological objects, the concentrations of which can be used as criteria for choosing the optimal dose range of irradiation treatment.

Based on the results of a set of studies, clear dose and time dependences of the concentration of aldehydes formed during oxidation of lipids and proteins, alkanes, as well as the concentration of ethyl alcohol in bio-objects after radiation treatment were found. It was found that with increasing irradiation dose in the range from 250 to 10000 Gy, the peak concentration of lipid and protein oxidation derivatives, aldehydes, in bio-objects is found on day 1 and day 2 after irradiation. At the same time, the higher the irradiation dose, the lower the ethanol content in biological objects during 4 days of storage after irradiation. Thus, the concentration of aldehydes, which are derivatives of lipid and protein oxidation, can serve as a marker of lipid and protein peroxidation, and the concentration of ethanol can serve as a marker of the efficiency of bacterial suppression as a result of irradiation. The spectrophotometric method of calculating the concentration of metmyoglobin in bio-objects containing myoglobin and the method of enzymatic hydrolysis by trypsin of the native protein structure (bovine serum albumin) allow quantitative assessment of the degree of radiation exposure at doses up to 10000 Gy on proteins depending on the physical parameters of radiation (dose, dose rate, type of radiation).

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CHEMOMETRIC METHODS IN SPECTROPHOTOMETRIC ANALYSIS

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A widely used pharmacopoeial method is spectrophotometry, the use of which is usually limited to single-component analysis. Therefore, the development of new approaches to the multicomponent analysis of complex mixtures using the spectrophotometric method is one of the urgent problems of modern analytical chemistry. The report will discuss approaches proposed by the authors for combining spectrophotometry with chemometric methods.

Methods have been proposed for the separate spectrophotometric determination of synthetic food dyes (SFD) in drinks and medicines, as well as active pharmaceutical ingredients (API) in eye drops, using targeted chemometric algorithms for multidimensional data processing (regression on principal components and projections on latent structures).

Modeling of spectral data was carried out using the example of the absorption spectra of SFD and API using the methods of regression to principal components (RPC) and projection to latent structures (PLS). It is shown that the modeling parameters are influenced by the choice of chemometric method, the degree of overlap of the absorption spectra of the analytes, photometric conditions, and the ratio of the concentrations of components in the mixture. Using the example of the E110 and E124 SFD systems, a comparison was made of the Vierordt methods, the first derivative at “zero crossing”, RPC and PLS. It has been shown that chemometric algorithms are characterized by the smallest determination error.

Methods for the spectrophotometric determination of E110 and E122 in the preparation “Travisil”; dyes E110 and E124 – in the preparation “Strepsils”; E110, E122 and E102 – in the “YUPI” drink; E102 and E110 in – opaque drinks in combination with magnetic solid-phase extraction; naphthyzine and diphenhydramine in eye drops from various manufacturers have been developed.

The research was carried out with the financial support of the Russian Science Foundation, project 24-23-00519.

APPLICATION OF PIEZOSENSORS WITH POLYCOMPOSITE COATINGS FOR ASSESSMENT OF MICROBIOLOGICAL SAFETY OF MILK

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Milk is an important food product that provides essential nutrients to humans starting in early childhood. However, today the assessment of the microbiological safety of raw milk is carried out using bacteriological examination according to a schedule, which does not allow quickly identifying changes in the microflora of milk. In addition, research is time consuming, requires qualified personnel, many reagents and, therefore, it is expensive. Consequently, the development of new quick and simple methods for assessing microbiological safety indicators of milk is relevant and significant.

The goal of the work was to develop a method for assessing the microbiological parameters of raw milk based on the composition of its gas phase using piezoelectric sensors with polycomposite coatings.

The study of the gas phase over raw milk samples was carried out using twenty composite coatings of piezoelectric sensors based on chromatographic phases, chitosan, deep eutectic solvents, and micellar casein concentrate. In parallel, physicochemical and microbiological indicators were assessed for milk samples according to GOST (QMAFAnM, yeast and mold content, presence of pathogenic microorganisms using molecular genetic methods).

Based on the results of the correlation analysis, sensors were selected whose parameters are statistically significantly related with microbiological indicators. Wherein the selected sensor parameters did not correlate with the physicochemical indicators of milk samples. Using chemometrics methods, multidimensional patterns of sensor parameters have been proposed to determine QMAFAnM and the presence of pathogenic microorganisms. The proposed approach was tested on mastitis milk samples, the conclusions were confirmed by analyzes according to GOST.

The work was carried out with the financial support of the Russian Science Foundation, project 22-76-10048.

RECOGNITION OF WATER SAMPLES USING THE KINETIC FINGERPRINT METHOD

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More than a billion people in the world do not have access to clean water, which makes it urgent to search for simple and cheap methods for assessing the quality of drinking water. For screening studies of water, it is advisable to use “fingerprint” methods, which provide an integral characteristic of a sample without identifying its individual components. We have proposed¹ a variant of the optical “fingerprint” method, based on carrying out indicator reactions in the presence of a sample. To recognize water, we used substitution reactions for the chlorine atom in carbocyanine dyes (see the figure for an example of the structure), which was accompanied by a gradual change in light absorption and fluorescence in the visible and near-IR regions of the spectrum. The progress of reactions was monitored by periodically photographing a 96-well plate with the reaction mixture; the results were processed by linear discriminant analysis (LDA).

It was shown that the proposed approach makes it possible to distinguish at least 11 samples of natural water from various sources. In addition, they demonstrated the ability to establish the fact of water contamination with motor oils, and it was possible to distinguish between samples containing oils that differ in characteristics and manufacturers. In both cases, the accuracy of discrimination after processing the results using the LDA method was 100%.

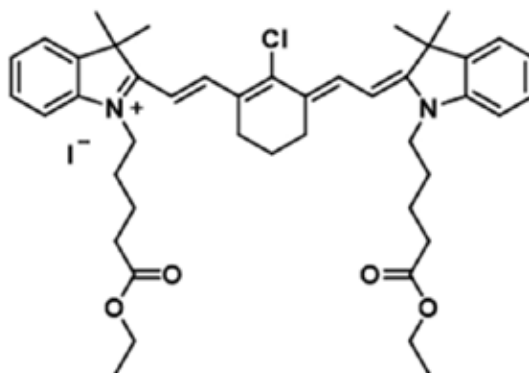


Figure 1. Structure of carbocyanine dye

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COBALT PIGMENTS IN THE PAINTINGS OF C. MONET

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The study of paints in art works is an important task of applied chemistry. It helps researchers to attribute, restore and conserve the objects. Two main problems are the size of the samples and their multicomponent mixture.

Researchers of the National London Gallery¹ have suggested a hypothesis: in the early period of the work Claude Monet used a mixture of cobalt blue and red organic pigment as a violet paint, and later he used ready-made violet pigments: cobalt violet $\text{Co}_3(\text{PO}_4)_2$ and cobalt violet light $\text{Co}_3(\text{AsO}_4)_2$. Analyzed commercial pigments and ready-made oil paints from different companies at the beginning of the 20th century with the trade name “light cobalt violet” in some cases contain the compound $\text{Mg}_x\text{Co}_{3-x}(\text{AsO}_4)_2$ in addition to $\text{Co}_3(\text{AsO}_4)_2$. The exact origin of $\text{Mg}_x\text{Co}_{3-x}(\text{AsO}_4)_2$ in the pigment composition is unknown^{2,3}.

The purpose of this study is to test the hypothesis on C. Monet’s serial works “Rouen Cathedral” from the collection of The Pushkin State Museum of Fine Art. The investigation was carried out using the methods of X-ray fluorescence spectral analysis (XRF), IR-Fourier microspectroscopy, Raman-microspectroscopy, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) and powder diffraction (XRD).

Based on the results of this work, new data have been introduced into the general understanding of C. Monet’s palette and painting technique and also a method for determining the rare magnesium-containing pigment “light cobalt violet” in a multicomponent mixture has been developed.

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STRUCTURAL-ANALYTICAL STUDIES USING RESONANT ELECTRON CAPTURE NEGATIVE ION MASS SPECTROMETRY

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One of the most informative methods for identifying substances and determining the molecular structure is mass spectrometry (MS), especially in combination with gas chromatography (GC). Due to the high sensitivity, good reproducibility and simplicity of the instrumental implementation, in the vast majority of studies, MS is used in the (classical) electronic ionization (EI) mode with the formation of positive ions (PI), but negative ions (NI) are rarely used and only to solve non-trivial tasks not feasible to MS PI. The processes of NI formation are associated with the addition of an excess electron to the vacant molecular orbitals, and the pathways of NI decay are determined by the electronic affinity of the fragment species formed. In contrast, at positive ion formation an electron is removed from occupied orbitals, and fragmentation is dependent on the ionization energy of molecules and their fragments, making the fundamental bases of these two branches of MS drastically different. The specific features of NI MS provide an additional opportunity to recognize compounds by the unique ionic composition of the NI mass spectra and indispensable information about the spatial and electronic structure of molecules and ions, at the same time requiring special theoretical knowledge, methodological approaches and technical tricks in instrumentation to operate with NIs.

The report discusses a low-cost way to complement the standard PI mode capabilities of GC/MS by the resonant electron capture (REC) mode for recording NI mass spectra. Using vivid examples, it is demonstrated that the resonance dependence of the NI yield intensity on the energy of captured electrons, unique for each compound, makes it possible to distinguish isomeric molecules; the presence of strict rules for NI fragmentation leads to relatively few-peaked and easily predictable REC NI mass spectra; high sensitivity to electrophilic molecules cause a number of advantages of the REC NI MS method, and often precisely in those cases where EI PI MS appears to be powerless.

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MICROEXTRACTION IN FOOD QUALITY CONTROL

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Food safety, including food quality control, is one of the strategies of scientific and technological development of the Russian Federation. The increase in food production and the use of a large number of chemicals (antibiotics, pesticides, fertilizers, hormones, preservatives, etc.) during their production are the main reasons for that. The need for the control leads to the development of highly effective and available procedures for the chemical food analysis. Considering the complexity of sample matrices, sample pretreatment is an important and integral stage of chemical analysis. Using various separation and pre-concentration methods, it is possible to eliminate the interfering influence of accompanying components and achieve concentration of the target analytes. Their combination with sensitive instrumental methods allows to determine selectively various substances at the MRL and below. New opportunities for food analysis are opened by microextraction procedures, which provide rapid mass transfer and phase separation with minimal consumption of extraction solvents. Ionic liquids, deep eutectic solvents, surfactants, natural extraction solvents, solvents with “switchable” hydrophilicity can be identified as the latest generation of extractants. In recent years, much attention has been paid to the “green index” of the method, which is calculated using various metrics. The microextraction techniques developed on the basis of St. Petersburg State University including their mechanisms, characteristics and practical application in food analysis will be reported.

The work was supported by a grant from the Russian Science Foundation (project No. 24-23-00052, <https://rscf.ru/project/24-23-00052/>).

SENSING SYSTEMS BASED ON PLASMON NANOCOMPOSITES WITH CONTROLLED SPECTRAL AND RECOGNITION PROPERTIES IN THE ANALYSIS OF BIOLOGICAL SAMPLES AND SAMPLES OF ECOLOGICAL MONITORING

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Surface-enhanced Raman spectroscopy (SERS) is today a highly sensitive and selective method of chemical analysis for the determination (including multiplex) of a wide range of biologically active substances and ecotoxins.

At the same time, the classical SERS sensors, widely known today, based on the noble metals silver and gold, despite their sometimes unique analytical characteristics, have a number of disadvantages, namely 1) a limited range of analyzed compounds due to the mismatch of the spectral characteristics of the surface and the analyte, 2) lack of affinity of analytes to the surface of metal nanostructures, 3) low reproducibility of the analytical signal, which in some cases leads to the impossibility of performing quantitative analysis, 4) instability during storage of sensor elements in air and during operation, 5) photocatalytic ability to degrade components of indicator systems and themselves determined analytes, 6) toxicity of SERS-active surfaces based on metal nanoparticles in relation to a number of biological objects if it is necessary to perform *ex vivo* or *in vivo* analysis.

According to the authors of the report, a promising approach to solve the problems described above, including significantly expanding the analytical capabilities of the SERS method, is the development of fundamental approaches to the creation of indicator systems and surfaces structured at the nanoscale that have specified (required for actual analytes) optical characteristics.

This report will primarily discuss the prospects of SERS sensing systems in the analysis of biological samples and samples of environmental monitoring.

The work was carried out with the financial support of the Russian Science Foundation 23-13-00276.

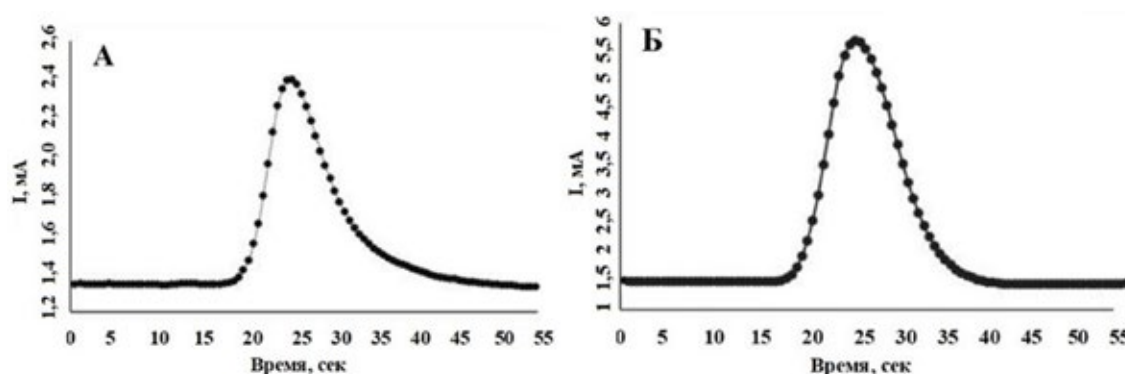
DETERMINATION OF POROUS MATERIALS SORPTION CAPACITY BY OXYTHERMOGRAPHY METHOD

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Nowadays, classical methods for determining the characteristics of porous materials have some limitations for researchers, in particular, the time and cost of analyzing a single sample. We suggest a new approach to investigation the sorption capacity of porous materials (sorbents) based on the oxythermography method¹. At the heart of proposed approach is based on the cyclic processes of sorption of oxygen from air during cooling of the sample and further desorption of oxygen during programmed heating in a flow of inert gas. The analytical signal is the kinetic curve of high-temperature oxygen desorption (Fig. 1). Natural sorbents (zeolites) from two different mineral assets - Honguruu and Kholinsky - were used as porous materials to trialability.



As a result of the research, the mass of oxygen per 1 gram of the porous materials under study was calculated, put it otherwise, their sorption capacity. For zeolite from the Honguruu mineral asset was $(7.70 \pm 0.3) \cdot 10^{-7}$ grams, and for Kholinsky mineral asset was $(31.3 \pm 1.1) \cdot 10^{-7}$ grams.

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CONTACTLESS CHEMICAL SENSOR FOR THE DETERMINATION OF INORGANIC CATIONS AND ANIONS

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A promising direction in analytical chemistry is the development of contactless sensors. This is driven by industries' demand for fast, simple, and inexpensive determination of the chemical composition of various media. One of the most accessible technical methods for obtaining information about the composition of analyzed media without physical contact of sensor elements with the sample are contactless conductivity measurements, which were widely studied in the 1940s and 1950s. At that time, the devices were primarily used for conductometric titration and were not widely used due to difficulties with mathematical processing of the signal, which depends in a complex way on the dielectric constant, conductivity, capacitance and magnetic properties of the sample. The technology has since evolved into several independent methods and devices, including high-frequency contactless conductometry, microwave spectroscopy, dielectric spectroscopy, C4D detectors (capacitively coupled contactless conductivity detection), and microwave sensors. Recently, we have proposed a new type of high-frequency contactless sensors. In contrast to previously described methods, we record the sensor response spectrum over the AC frequency range for each specific sample and, using chemometric methods, obtain qualitative and quantitative information about the samples. To date, the responses of such a sensor have been obtained in individual aqueous solutions of 16 inorganic cations (Li^+ , K^+ , Na^+ , Cs^+ , Rb^+ , Ca^{2+} , Mg^{2+} , Sr^{2+} , Co^{2+} , Ni^{2+} , Zn^{2+} , Cd^{2+} , Cu^{2+} , Pb^{2+} , Nd^{3+} , Gd^{3+}) and 10 anions (F^- , Cl^- , Br^- , I^- , OH^- , NO_3^- , OAc^- , SO_4^{2-} , H_2PO_4^- , HCO_3^-) in the concentration range 10^{-5} - 10^{-1} mol/L. Sensitivity and limits of detection of the sensor have been studied. The presentation will focus on the details of these studies along with a discussion of possible future applications of the sensor to analyze real samples.

This research was funded by the Russian Science Foundation, grant number 23-23-00114.

CARBON NANOMATERIALS: OPPORTUNITIES AND PROSPECTS OF USE FOR THE PRECONCENTRATION OF REE AND NOBLE METALS

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The inclusion of the sorption preconcentration stage into the procedure of analysis of complex geochemical objects is nowadays probably the most effective way to determine ultra-low contents of rare-earth and noble elements. The main disadvantages of the known sorbents include low extraction selectivity and difficulties in desorption of elements. This significantly reduces the effectiveness of their application in analysis.

Carbon nanostructured materials (CNMs) are among the most promising sorbents for elements preconcentration thanks to their sorption activity, well-developed surface, ability to ensure high purity, as well as the constantly expanding assortment of nanomaterials available. The obtaining of composite materials and the covalent or non-covalent immobilization of functional groups make it possible to obtain a wide range of effective sorbents, including those that are effective in acidic solutions. This increases the selectivity and fullness of analyte recovery. The acid decomposition of CNMs in a microwave system allows to eliminate the desorption stage, to simplify the subsequent ICP-MS/AES analysis, providing a relatively high analytes concentration coefficient.

This report presents the results of a study of various CNMs types sorption properties: nanotubes, mesoporous carbon, nanocomposites (Tambov State Technical University), as well as modified forms of CNMs synthesized in Vernadsky Institute of Geochemistry and Analytical Chemistry by mineral acids oxidation, as well as non-covalent and covalent modification with N- and N,S-containing reagents. The report considers the specificity of the CNMs use, the conditions for the preconcentration of a wide range of elements including REE and noble metals, the influence of macro components and other aspects. The examples of CNMs application for rare-earth and noble elements preconcentration from solutions of different compositions are given, including the ones obtained after various types of geological samples decomposition.

A METHODOLOGICAL APPROACH TO DETERMINING THE ELEMENTAL COMPOSITION AND IDENTIFYING THE SOURCES OF SEDIMENTS FORMED DURING TECHNOLOGICAL PROCESSES AT OIL REFINING ENTERPRISES.

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In the technological processes of oil refining, harsh conditions are almost always required – high temperatures and pressure. In combination with aggressive media such as hydrogen chloride, sulfur-containing compounds, free sulfur, etc., lead to corrosion of equipment materials and the formation of sediments of various compositions. The formation of sediments poses a threat of unplanned equipment shutdowns and requires preventive measures to combat them.

In order to develop effective measures aimed at finding the sources of sediment origin, information on their elemental composition is needed. The choice of an approach for determining the elemental composition, first of all, will depend on their component composition - the ratio of organic and inorganic components, as well as the composition of the organic phase. The experience of working with deposits selected from various technological units of installations has shown the expediency of dividing them into the following groups:

- sediments with an organic component of no more than 10% by weight.,
- sediments with an organic component of 90% of the mass. and more,
- sediments with an organic component of 10 - 90% by weight.

In this work, a combination of various methods of sample preparation of sediment samples to determine their elemental composition by atomic spectroscopy (AAS, MS-ICP, AES-ICP) was studied: acid dissolution in open systems, in autoclaves with resistive and microwave heating, fusion with subsequent transfer of melt into solution. Depending on the ratio of the organic and inorganic components of the sediments, optimal options have been selected that allow quantitative conversion of sediment samples into solution

The proposed approaches have been tested on sediments samples taken from various units and assemblies of technological equipment of oil refineries: heat exchangers, refrigerators, pumps, filters, etc. Based on the work performed, a methodological approach to the determination of the elemental composition of sediments is proposed, which made it possible to optimize the analysis and increase the effectiveness of the determination. An approach to the identification of sediment sources based on their composition is formulated.

NOVEL CATIONIC MODIFIER OF ELECTROPHORETIC SYSTEMS BASED ON A POLYMER WITH A CHIRAL TAG

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Currently, capillary electrophoresis (CE) is an actively developing and widely popular analytical method. The development of CE is due, to a significant extent, to the creation and use of new modifiers of the internal surface of a quartz capillary.

The purpose of this study is to synthesize new multifunctional cationic modifiers – poly-11-acryloyloxyundecyl-N-methylpiperidinium bromide (pAUMP-Br) and a copolymer with a chiral tag – acylated quinine (pAUMP-AQin·HCl) and to identify their analytical capabilities in the separation of biologically active substances with various functional groups, including enantiomers.

The synthesis of the micellar polymer was carried out according to scheme¹, the surface-active properties of pAUMP-Br were studied – solubilization capacity and critical concentration of micellization (0.001 M), allowing one to make a prediction regarding the possibility of using the new micellar polymer as a modifier in CE. Copolymerization of AUMP and AQin was carried out to obtain a modifier with a chiral tag. Modification of the walls of a quartz capillary after etching and washing with a 0.001 M polymer solution was confirmed by the generation of an anodic electroosmotic flow when using both polymers. It was found that new micellar polymers make it possible to implement various modes of capillary electrophoresis (micellar electrokinetic chromatography and capillary electrokinetic chromatography), increase the selectivity and efficiency of separation of amino acids, catecholamines, steroid hormones, and they are also capable of participating in the online-concentration of biologically active analytes.

The results of the enantiomeric separation of amino acids and non-steroidal anti-inflammatory drugs using a new polymer with a chiral tag are discussed.

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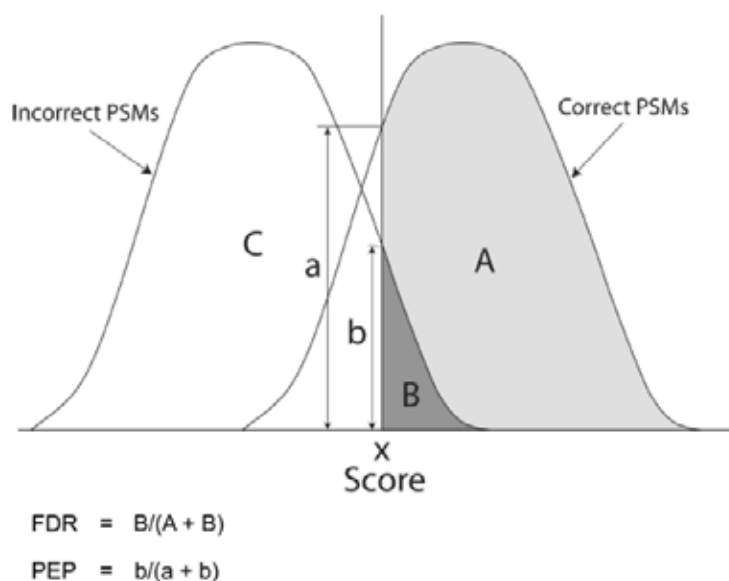
The research was supported by RSF, project 24-13-00378.

THE DEVELOPMENT AND QUALIFICATION OF A HIGH-SPEED METHOD FOR THE VERIFICATION OF PROTEIN AMINO ACID SEQUENCES

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Verification of protein drug amino acid sequences is a crucial quality metric in ensuring the authenticity of pharmaceuticals¹. As is widely recognized, LC-MS/MS is the only method that can directly affirm (and in the case of de novo approaches, establish) the amino acid sequences of protein drugs. A robust verification requires the use of multiple enzymes and their combinations, as well as unconventional methods for separation, purification, and detection. This verification process relies heavily on software, particularly in its approach to assembling peptide signals into amino acid sequences and in analyzing the data.



A versatile and cost-effective analytical platform has been developed by the scientific team at the Laboratory Complex of Sirius University in recent years. This platform enables the confirmation of the amino acid sequences of the majority of therapeutic proteins (including heavily modified ones) through the simultaneous application of multiple enzymes in a single analysis

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THE STUDY OF GRAPHENE QUANTUM DOTS IN DMSO AND DMF SOLUTIONS BY MOLECULAR SPECTROSCOPY METHOD

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In recent years, graphene quantum dots (GQDs) have stood out among other fluorescent nanomaterials due to their biocompatibility, photostability, and low toxicity¹. The optimal method for obtaining GQDs is based on the synthesis of graphene oxide from glucose². In this study, the optical properties of GQDs in DMSO and DMF solutions were investigated using molecular spectroscopy methods (UV and near-IR). Figure 1 shows differences in the UV and near-IR spectra for the studied solutions in the range of 1100-300 nm.

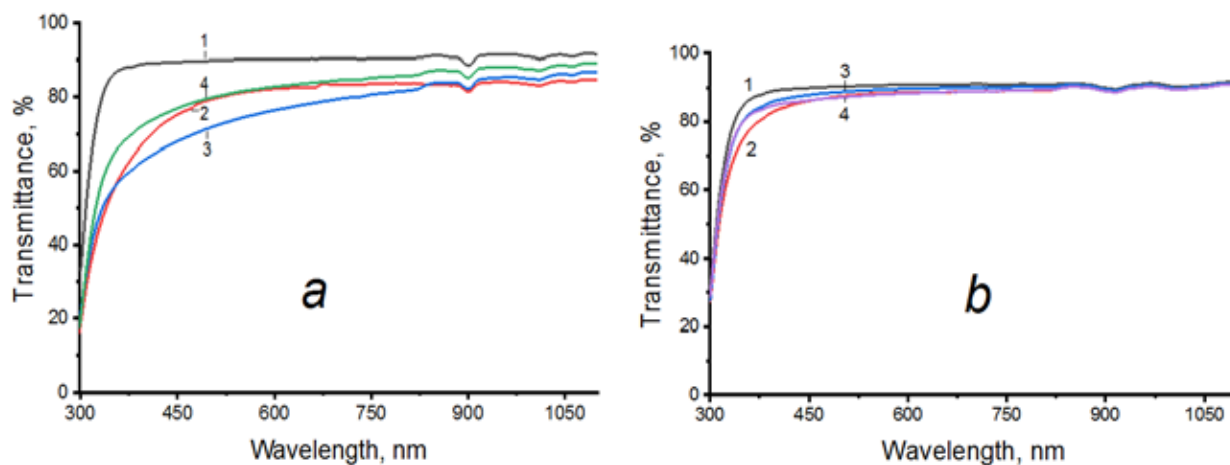


Figure 1. UV and near-IR spectra in solutions a - DMSO-GQDs and b - DMF-GQDs, taken in the pure solvent (1), in the presence of GQDs, after treatment: mechanical (2), ultrasonic for 5 minutes (3), and 10 minutes (4).

As can be seen, when adding GQDs to DMSO, the transmittance (Fig. 1.a) decreases by 40%, while in the case of DMF solutions with GQDs (Fig. 1.b), the decrease is only 10%. Presumably, this is due to the more efficient interaction of GQDs with DMSO molecules compared to DMF molecules. These differences may be caused by the chemical composition or properties of the solvents, as well as the characteristics of their interaction with GQDs. Further research will help to better understand these processes and optimize the use of GQDs in optical materials.

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SEPARATION AND PRECONCENTRATION OF BIOLOGICALLY ACTIVE COMPOUNDS BY CAPILLARY ELECTROPHORESIS METHOD USING MODIFIERS BASED ON IMIDAZOLIUM CATION

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Modifiers based on the imidazolium cation are the most universal in the capillary electrophoresis (CE) and are suitable for the separation of the large number of biologically active compounds, since they have a positive charge in the structure, an aromatic system, are characterized by the ease of introducing various substituents into the imidazolium fragment, and can also participate in processes of intracapillary preconcentration.

Alkylimidazolium ionic liquids have proven themselves in the formation of covalent coatings on the walls of quartz capillaries, promoting intracapillary preconcentration of biogenic amines. The combination of sweeping and field-amplified stacking injection makes it possible to reduce the detection limits (LOD) of neurotransmitters to 0.3-1 ng/ml. The influence of the coating structure on the concentration efficiency was additionally studied.

A variant of the synthesis of covalent coatings based on imidazolium cation and β -CD is proposed. The present of β -CD into the coating structure allows for the simultaneous determination of hydrophobic corticosteroid hormones and hydrophilic biogenic amines, as well as act the role of a chiral stationary phase in the separation of ketoprofen enantiomers.

Modifiers based on alkylimidazole and β -CD (similar to the covalent coating formed on the walls) were synthesized and their analytical possibilities were revealed as a pseudo stationary phase in the separation of corticosteroid hormones and as a chiral selector in the separation of drug enantiomers. The presence of a positively charged imidazolium ring in the selector structure provides an additional contribution to the efficiency and separation selectivity of the ketoprofen and ketorolac enantiomers. In the found conditions, a new version of the hybrid on-line preconcentration of the enantiomers of ketoprofen and ketorolac (dynamic pH-junction + sweeping) was proposed, which made it possible to achieve concentration factors of 325-390 and thus reduce the detection limits to 11-12 ng/ml for ketoprofen and 51-57 ng/ml for ketorolac.

FUNCTIONALIZED MATERIALS BASED ON IONIC LIQUIDS AND MAGNETIC NANOPARTICLES FOR EXTRACTION AND CONCENTRATION OF BIOLOGICALLY ACTIVE COMPOUNDS IN SOLID-PHASE MICROEXTRACTION

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Determination of biologically active compounds in natural objects allows for solving important medical-diagnostic, biochemical and ecological problems. However, the complexity of the sample matrix and low concentrations of the analytes (ng/mL) require the development of highly efficient and selective approaches for the extraction and concentration of analytes. A promising direction is the development of new sorbents based on magnetic nanoparticles (MNPs) and their application in the method of magnetic solid-phase microextraction (MSPME). Ionic liquids (ILs) are of particular interest as modifiers. ILs are ionic non-molecular solvents with adjustable properties that depend on the nature of the cation and anion in their composition. The combination of ILs with MNPs opens up possibilities for development rapid extraction methods due to the high sorption capacity of MNPs and various types of interactions between ILs and analytes.

The aim of the research is the synthesis of sorbents based on magnetite MNPs modified with imidazolium ILs, and the study of their application possibilities for the concentration and extraction of biologically active compounds with different polarities. Polyphenolic antioxidants and steroid hormones were chosen as model systems. Conditions for their separation by HPLC with DAD detection were found. MNPs were synthesized by coprecipitation of Fe(II) and Fe(III) salts and stabilized with oleic acid and SiO₂ coating. The potential of hydrophobic imidazolium ILs as surface modifiers for MNPs in MSPME conditions for analyte extraction was identified. Factors influencing the extraction efficiency were optimized using experimental design. It was established that the use of [C₈MIM][BF₄] IL as a modifier for MNPs in MSPME conditions ensures high extraction efficiencies of the studied analytes (64-98%).

The authors express their gratitude to the Resource Centers "Methods for Substance Analysis," "Nanotechnologies," and "Innovative Technologies of Composite Nanomaterials," as well as to the Scientific Park of Saint Petersburg State University. The work was carried out with financial support from the Russian Science Foundation (grant No. 24-13-00378).

NEW ANALYTICAL SOLUTIONS IN THE ANALYSIS OF CHEMICAL REAGENTS OF OIL PRODUCTION

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Chemical reagents (ChR) of various aggregate states, actively used in the process of extraction, transportation and storage of petroleum raw materials, containing organochlorine compounds (OChC), well clean the associated equipment, oil, etc. from deposits. However, OChC are the strongest poisons that cause corrosion of equipment during oil refining. Therefore, the need to control the OChC in chemical reagents used at different stages of oil production is an urgent task.

Standard methods are absolutely not intended for the study of samples of chemical reagents used in oil production. ChR radically differ in physico-chemical properties from the typical naphtha BP-204⁰C isolated from traditional oils, not only in fractional composition, but also have a different phase form, up to solid conglomerates or crumbly states. Without preliminary sample preparation, it is impossible to determine the OChC in ChR.

To prepare ChR for the determination of OChC, studies were conducted on the use of hydrocarbon solvents as an extractant, in which the presence of OChC was previously determined. As a result, an effective hydrocarbon solvent, nefras C-50/170, was selected, since it is less toxic, affordable, cheap, does not contain OChC and is close to naphtha in fractional composition. Alcohol was added to the nonpolar solvent to regulate the mutual solubility of the phase components, as well as to improve the efficiency and selectivity of the OChC extraction process. The experiment with a binary solvent was carried out on model mixtures with different concentrations of individual OChC, which are most often used in ChR. ChR, which does not contain OChC (according to the passport for this reagent), was used as the basis of the model mixture. In the extraction process, low-boiling OChC pass into nefras. Extraction was carried out in several stages until the complete absence of OChC.

The developed method of sample preparation has been tested on various ChR, both liquid and solid, bulk, with reliable results on the OChC content. A Patent of the Russian Federation has been obtained.

SELECTION OF PARAMETERS IN THE DEVELOPMENT OF METHODS FOR DETERMINING TAURINE USING HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

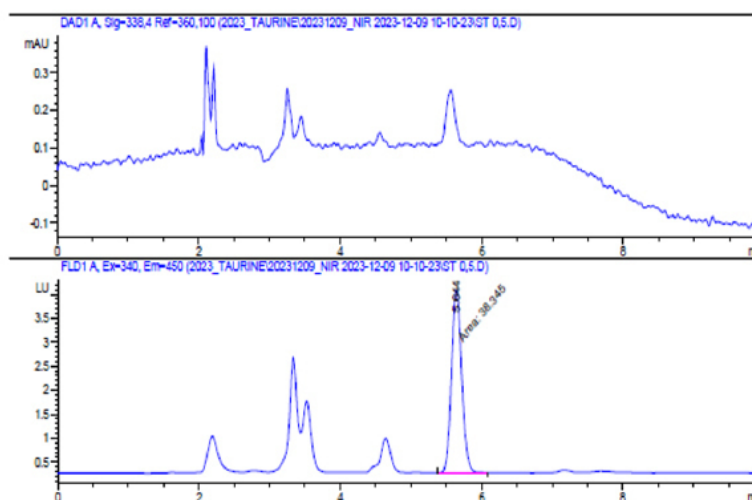
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Taurine is a free, semi-essential, sulfur-containing β -amino acid, formed in a small proportion from methionine, and also when decarboxylation of the amino acid cysteine.

The research work was carried out on an Agilent 1260 high-performance liquid chromatograph with fluorimetric and diode array detectors, the latter recording a higher signal, lower baseline noise and a lower limit of quantitative detection (Pic. 1).

When selecting chromatographic parameters, a Kromasil 100 C18 250*4.6 mm, 5 μ m column was selected, isocratic eluent supply mode (A – 0.02 mol/dm³ sodium acetate trihydrate with pH 6.6, B – acetonitrile), flow rate – 1 cm³/min.



Picture 1. Example of a chromatogram of a standard taurine solution with a concentration of 0.5 mg/dm³ on a diode array detector and a fluorimetric detector

Sample preparation consisted of extraction of the analyte with a 0.1 N solution of hydrochloric acid for 10 minutes, followed by derivatization. The derivatization step consisted of obtaining a complex of the test sample with o-phthalaldehyde in the presence of 3-mercaptopropionic acid. This complex is destroyed within 7-10 minutes, which requires immediate analysis.

DEVELOPMENT OF ENZYME-LINKED IMMUNOSASSAY TECHNIQUE FOR THE DETERMINATION OF GENTAMICIN IN MEAT

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Gentamicin is a broad-spectrum antibiotic of the aminoglycoside group. Excessive and uncontrolled use of this drug in veterinary may further lead to the emergence of pathogenic microorganisms' resistance, and thereby cause potential harm to human and animal health.

The enzyme-linked immunoassay is actively used for rapid quantitative evaluation of the therapeutic drugs residues in food of animal origin.^{1,2} It is highly sensitive, fast and can detect multiple samples simultaneously.

The purpose of this work was to develop a technique for the determination of gentamicin in samples of beef and poultry meat.

The developed method was based on the method of direct competitive enzyme immunoassay. Polyclonal sera were obtained by immunization of rabbits with synthesized conjugates of gentamicin with keyhole limpet hemocyanin and bovine serum albumin. The most specific sera were selected for work. Also, optimal concentrations of reagents and the most suitable conditions for carrying out the immunochemical reaction were selected. When evaluation of the technique's specificity, cross-reaction with other aminoglycoside antibiotics was less than 1%.

The detection limit was 0.3 µg/kg, and the coefficient of variation did not exceed 8%. The analysis time was 15 minutes at room temperature. The recovery range was 70-90%.

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TEST INDICATION OF ROCKET HYDRAZINE FUELS UNDER OPERATING CONDITIONS

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Rocket hydrazine fuels (RHF), which include hydrazine and 1,1-dimethylhydrazine (heptyl), are widely used in rocket and space technology¹, and in terms of toxicity are classified as hazard class 1 as super-ecotoxicants in terms of their effects on the human body². When working with RHF in production, transportation, storage and use conditions environmental monitoring of work areas, permanent personnel premises, and equipment surfaces for leakage indication using rapid tests is of current interest. The maximum permissible concentration of heptyl in the air of the working area is 0.1 mg/m³, in water – 0.02 mg/dm³. A set of indicator rapid tests has been developed to ensure occupational safety and prevent emergency situations at infrastructure facilities operating with RHF (Table 1).

Table 1. Characteristics of test kits

Object of analysis	Test agent	Reagents *	Determination range
Working area air	Indicator tube ³	THMS	0.05–5 mg/m ³
Personnel room air	Passive chemical indicator ⁴	TFT	0.05–0.25 mg/dm ³
Leakage of RGG	Indicator element ⁵	THMS	1 µl/1 cm ² or more
Aqueous medium	Indicator strip	NTFT-C	0.005–5 mg/dm ³
Wash waters	Indicator strips	THMS-C6	0.01–10; 10–500 mg/dm ³

* THMS – Potassium tetrahydro-12-molybdosilicate octahydrate $K_4H_4[Si(Mo_2O_7)_6] \cdot 8H_2O$; TPhT – 2,3,5-triphenyl-2H-tetrazolium chloride; NTPhT-C – 3-(4-nitrophenyl)-2,5-diphenyl-2H-tetrazolium chloride grafted onto cellulose aldehyde; THMS-C – TGMS grafted onto epoxycellulose.

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PROMPT GAS CHROMATOGRAPHIC DETERMINATION OF JET FUEL FLASH POINT

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Flash point (FP) of jet fuels (JF) is one of the indexes of flammability being their important operational property. Low precision of experimental methods of FP determination (for example, according to Russian Standard GOST 6356–75), the need for a fairly significant volume of the sample being analyzed using them, limits of the initial true boiling point (TBP) and TBP of 5 %- and 10 %-fractions of JF established in calculation method ASTM D7215–22, as well as the presence of significant differences between them and the corresponding values for JF produced in Russian Federation, which has been determined by the authors, make necessary the other method for JF FP determination development.

New method for JF FP calculation based on gas chromatographic (GC-) determination of TBP by simulated distillation in optimized conditions and the full array of GC-data processing by the developed algorithm free from special foreign software¹ is presented. The type of TBP values dependence on JF fractions content in the range of 0.5–10 wt. % and the values of its coefficients for different JF have been established. The equation type for FP calculation being general for JF of different component compositions has been offered.

The average absolute deviation between calculated and experimental FP values is 1.5 °C, which corresponds to the repeatability according to GOST 6356 (2 °C) and is lower than the corresponding deviation indicated in ASTM D7215 (4 °C). The repeatability of FP values determination by the developed method is 0.03 °C. The duration of the determinations does not exceed 10 min. The use of the method is perspective for aircraft failures investigations.

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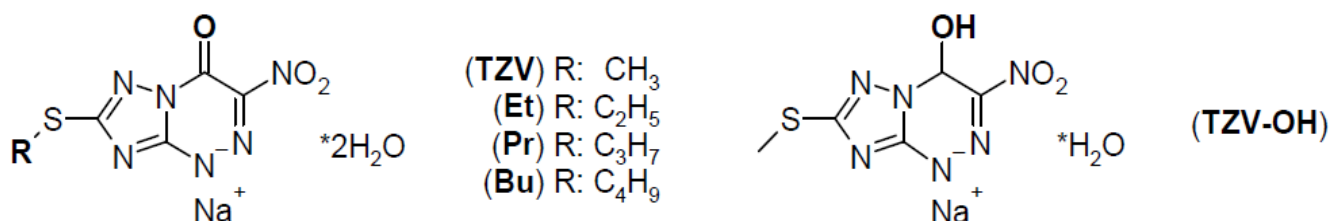
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DIGITAL METHODS IN THE STUDY OF POSSIBLE TRANSFORMATION PATHWAYS OF STRUCTURAL ANALOGUES OF TRIAZAVIRIN®

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The process of developing an original drug is quite complex. Today, in order to speed up this process, in addition to well-known instrumental methods of analysis, digital technologies are actively used. They focus on modeling methods such as quantum chemical calculations (QCC). This work can be the basis for further development of an approach to the study of potential drugs in order to identify the lead compound using a combination of electrochemical analysis, EPR spectroscopy and QCC methods. The purpose of the work is to develop an approach to a comprehensive study of the transformations of the antiviral drug Triazavirin® (TZV) and its structural analogues using the example of a series of TZV, TZV-OH, Et, Pr, Bu.



Using the QCC method, the probable adducts of the DMPO spin trap with the products of electrochemical reduction of all compounds were calculated, and the probable path of electrotransformations was simulated. It has been shown that the process of electrochemical reduction of the studied substances in an aprotic environment occurs through the formation of a dianion radical, and the absence of an additional stage of protonation of the dianion radical **TZV-OH** probably leads to the formation of a larger number of intermediates of a radical nature. As a consequence, this may be one of the reasons for greater biological activity. Thus, data obtained by a combination of digital and instrumental methods are likely to be fundamental for the identification of compounds with biological activity.

The work was carried out under agreement with the Ministry of Science and Higher Education № 075-03-2023-006 dated 01.16.2023 (topic number FEUZ-2023-0021)

EXTRACTION OF CARBOXYLIC ACIDS FROM THE CONTENTS OF AMPHORAE FOR DETERMINATION OF FATTY ACID PROFILE BY GC/MS

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During archaeological excavations, preserved pottery or its shards are found, which were used for transportation and storage of foodstuffs. Analysis of the fatty acid profile of lipid-containing inclusions in the contents of amphorae allows for the identification of their species affiliation. The extraction method is usually used for isolation and subsequent determination of the fatty acid composition. The high carbonate content prevents the efficient extraction of analytes by classical extractants.

In this work, studies were carried out to optimize the extraction method to obtain an undistorted fatty acid profile providing reliable indicator ratios, for example, palmitic and stearic acids. Instrumentally, the proven Soxhlet extraction was chosen as a reference.

The main parameters for optimization were the type of extractant (chloroform:methanol (2:1) and methanol:HCl mixture), time of extraction, temperature, volume ratio of solid and liquid phases.

The object of analysis is a ceramic jug from the elite complex of the Sargat culture in the Middle Priirtysh (Isakovka I burial ground, barrow 3, burial 6). Measurements were carried out on Clarus 600 GC-MS system (Perkin Elmer), on Elite-5MS column (L:30 m; ID 0.25; DF 0.25). Helium flow rate was 1 mL/min.

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FORMS OF NICKEL IN BOTTOM SEDIMENTS OF SMALL RIVERS

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Heavy metals pollution of river systems is interest due to their ability to bioaccumulate and high toxicity, and the degree of toxicity depends on the chemical form of the element¹⁻². The object of the study was the bottom sediments of the small Barsuk River, which were fractionated into sizes using a set of sieves. The forms of nickel in the fractions were determined by five-step extraction³.

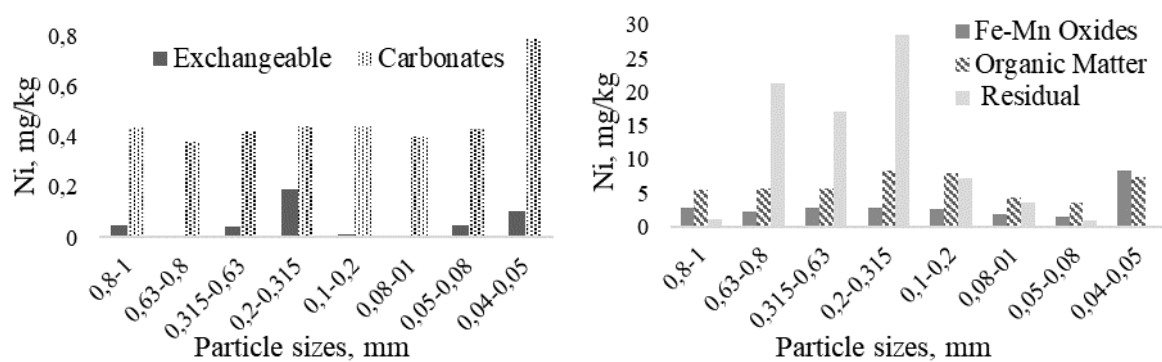


Figure 1. Distribution of nickel's forms (in extractable fractions) in various particle size fractions of bottom sediments of the Barsuk river

Content of Ni in exchangeable and bound to carbonates fractions is insignificantly (fig.1) and equal to 0.3 and 2.4% of the total content respectively. Despite the high mobility of these forms, they cannot have a significant impact on the river ecosystem. The fraction Bound to Fe-Mn oxides amounted to 15.9% of the total content and is mainly represented in the 0.05-0.04 mm fraction. The form of Ni bound to organic matter is presented in all size fractions and averaged 30.9%. The mobility of these forms of metal can increase significantly when environmental conditions change. The share of the residual form of Ni from the total content was 50.6%, while migration of this form into the water is unlikely. The maximum content of residual and exchangeable forms of Ni was observed in the fraction 0.2-0.315 mm, organic - in fractions 0.2-0.315, 0.1-0.2 and 0.04-0.05 mm.

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DEVELOPMENT OF ICP-AES METHOD FOR DETERMINING GALLIUM IN METALLURGICAL MATERIALS

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Inductively coupled plasma atomic emission spectrometry (ICP-AES) is an informative and widespread method for multi-element analysis of objects of various nature due to its high sensitivity, rapidity, and the availability of a new generation of analytical equipment. It is widely used to solve various problems in the analysis of metallurgical products and ores, including the determination of gallium¹.

However, ICP-AES determination of gallium content in ore and finished metallurgical products is a challenging task due to its low content and the influence of matrix components on the analytical signal².

The purpose of this work is to investigate the possibility of ICP-AES for determining the gallium content in metallurgical materials, with preliminary separation of the analyte from the matrix components.

For the first time, studies were carried out on the separation of gallium and macrocomponents (Fe, Cr, Mo, W, Ni, Co) using sodium fluoride as a precipitant. Analytical lines Ga I 294.364, Ga I 403.298, Ga I 417.206 nm were established, which have the least spectral overlap from macrocomponents when determining the gallium content by the ICP-AES method in metallurgical materials. It has been established that the use of sodium fluoride leads to the almost complete removal of macrocomponents from the analyzed solutions. In this case, gallium is completely coprecipitated on the sediment of the composition $\text{Na}_3\text{FeF}_6\text{--NaF--NaHF}_2$. Optimal conditions have been established for the coprecipitation of gallium on the precipitate and the dissolution of the $\text{Na}_3\text{FeF}_6\text{--NaF--NaHF}_2$ precipitate and the transition of gallium into the analyzed solution for its further ICP-AES determination.

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POLYVINYLFORMAL SORBENTS WITH THIOUREA GROUPS FOR THE CONCENTRATION OF PRECIOUS METALS

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From a whole list of sorbents of various natures proposed for the concentration of precious metals, sorbents with sulfur-containing functional groups are characterized by the best selectivity. Polymer sorbents synthesized as a result of condensation or polymerization reactions involving sulfur-containing reagents are of particular interest.

In this paper, the properties of a sorbent based on a copolymer of polyvinyl alcohol, formalin and thiourea (PVF-TM) with respect to noble metals are investigated.

The PVF-TM sorbent has been characterized by thermogravimetry and differential scanning calorimetry, IR spectroscopy and electron microscopy with microanalysis. The hydrolytic stability of sorbents in hydrochloric acid solutions of different concentrations and at different temperatures has been determined. It has been shown that the PVF-TM sorbent is resistant to 1-4 M HCl at room temperature.

The PVF-TM sorbent quantitatively ($\geq 95\%$) extracts Pt(IV), Pd(II), Au(III), Ag(I) at room temperature in the acidity range of 2 M HCl – pH 3. The degree of extraction of Rh(IV), Ru(IV) and Ir(IV) in under these conditions, it does not exceed 30%. The time to establish sorption equilibrium is 5 minutes for Ag(I), Au(III), Pd(II) and 10 minutes for Pt(IV). An increase in temperature to 95 °C leads to an increase in the degree of extraction of Rh(IV), Ru(IV) and Ir(III) up to 60%. However, with an increase in the contact time of the phases at an elevated temperature, partial dissolution of the sorbent is observed during the extraction of Rh(IV), Ru(IV) and Ir(III). The sorption capacity determined from the horizontal sections of the sorption isotherms was 4.5 mmol/g for Ag(I), 1.5 mmol/g for Pd(II), 0.74 mmol/g for Pt(IV). When Pt(IV) and Au(III) interact with the functional groups of the sorbent, they are restored with the formation of surface complexes Pt(II) and Au(I) with thiourea groups possessing intense low-temperature luminescence. Subsequently, as a result of the Au(I) disproportionation reaction, the formation of nanoscale.

INFLUENCE OF HAPTEN STRUCTURE ON THE SPECIFICITY OF IMMUNOANALYSIS OF ACETOCHLOR AND BUTACHLOR HERBICIDES

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Acetochlor (AC, Fig. 1A) and butachlor (BC, Fig. 1B) are chloroacetanilide herbicides widely used to protect rice, corn, soybeans and sunflower crops. Immunoanalytical methods provide effective control of herbicide contamination of agricultural products, but their characteristics largely depend on the properties of the hapten-protein conjugates used for immunization and competitive interactions during the analysis.

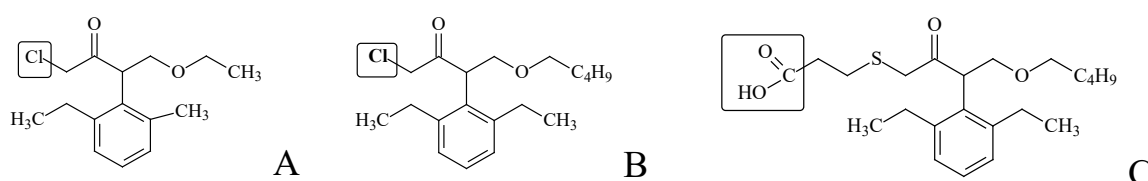


Figure 1. Structures of acetochlor (**A**), butachlor (**B**) and the product of its modification with mercaptopropionic acid (**C**). The frames shows the used functional groups

In herbicide molecules, the Cl atom was replaced by a mercaptopropionic (MPA) (Fig. 1B) or mercaptosuccinic (MSA) acid residue. The carboxyl derivatives synthesized in this way were conjugated by the carbodiimide method with soybean trypsin inhibitor (STI) and bovine serum albumin (BSA).

Testing of polyclonal rabbit antibodies obtained during immunization showed a significant influence of the properties of haptens (in particular, differences in the length of side radicals) on the characteristics of the enzyme-linked immunosorbent assay. For example, the choice of a conjugate immobilized in a microplate that competes with the controlled compound changed the detection limit by more than an order of magnitude. For the conjugates STI-AC-MPA, STI-AC-MSA and BSA-BC-MPA in combination with anti-BC antibodies, the detection limits of BC were 22.3, 3.4 and 1.3 ng/ml, respectively.

The suitability of the developed analytical systems for monitoring different types of agricultural products is shown.

This work was financially supported by the Russian Science Foundation, project 23-46-00018 (<https://www.rscf.ru/project/23-46-00018/>).

BIOLOGICALLY ACTIVE SUBSTANCES EXTRACTION FROM MADDER ROOTS *RUBIA TINCTORUM* L.

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Plants are invaluable reservoir for the discovery of new drugs, which have been used for medicinal purposes across history and cultures. It is known that madder root *Rubia tinctorum* L. contains more than 100 compounds: anthraquinones, iridoids, pectin substances, proteins and organic acids¹. *Rubia tinctorum* L. is known in medicine due to antispasmodic, diuretic and lilolytic properties. *Rubia tinctorum* is used as a diuretic and stone inhibitor. Moreover antifungal activities of *Rubia tinctorum* have been researched².

A series of experiments was conducted to study the effect of various parameters on the degree of BAS extraction. Biologically active substances (BAS) from the root of madder *Rubia tinctorum* L. were extracted using distilled water, methanol, ethanol, acetonitrile and deep eutectic solvents (TBA with ethylene glycol and choline chloride with urea, propylene glycol, ethylene glycol, fructose and glycerin, malonic, lactic and malic acid). Alkaloids, anthraquinones, alizarin and glycosides were found in the extracts by phytochemical screening methods³. HPLC and GC-MS were used to quantify BAS in the extracts.

It was found that the optimal conditions for the extraction of BAS from the roots of *R. tinctorum* are 60°C for 60 minutes with methanol and deep eutectic solvents based on TBA with urea, choline chloride with lactic and malonic acid.

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APPLICATION OF NIR SPECTROSCOPY AND CHEMOMETRICS FOR REAL-TIME DETERMINATION OF COMPOSITION OF URINARY CONCREMENTS

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According to the clinical recommendations of the European Association of Urology and the Ministry of Health of the Russian Federation, the choice of surgical treatment of urolithiasis – one of the most common urologic pathologies – depends largely on the chemical composition of the stone. At the same time, intraoperative methods of determination of stone composition have not been developed so far, which is an urgent task of modern urology.

Near-infrared (NIR) spectroscopy is a promising nondestructive method for determining the composition of urogenital concretions due to the rapidity of acquisition, absence of sample preparation, availability of remote probes compatible with catheterization instruments. Since an NIR spectrum does not have a fine structure, it is necessary to use methods of mathematical processing of NIR spectra, including chemometric methods. Qualitative analysis of a urinary stone presents a classification problem, i.e. correlation of the spectrum of a particular sample and its type: “oxalate”, “urate”, “phosphate” [1].

This paper presents the results of analysis of 172 fragments of urinary stones, surgically removed by laser lithotripsy. Measurements were performed via a portable NIR spectrometer (Avantes, Netherlands) in the range of 939-1799 nm and a fiber-optic probe with a diameter of 1.8 mm (Endoprobe 7NIR + 1NIR, “Optovolokno”, Russia). Each stone was measured at three different points, followed by averaging of the spectra, both in air and under simulated saline flow conditions. The paper presents a comparison of several combinations of data preprocessing and multivariate analysis (linear discriminant analysis, support vector machines with different kernel functions), as well as the results of the intraoperative application of the proposed method.

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DEVELOPMENT OF A UNIVERSAL APPROACH FOR DETERMINING PESTICIDES OF THE PYRAZOLE CARBOXAMIDE GROUP IN VEGETABLES, FRUIT AND GRAIN CROPS AND THEIR PROCESSING PRODUCTS

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Pyrazole carboxamides are highly active substances that have a beneficial effect on the physiological state of plants and a long-term suppressive effect on phytopathogenic fungi. One of the representatives of this group of pesticides is benzovindiflupyr. Benzovindiflupyr is a modern broad-spectrum foliar fungicide and an active ingredient of a number of drugs registered in the Russian Federation.

The purpose of this research was to develop a universal approach for the determination of the pesticide of the pyrazole carboxamide group in vegetable and grain crops and their processed products (oils, juices) on an example of benzovindiflupyr through the identification by high-performance liquid chromatography.

The proposed method for the determining benzovindiflupyr involves isolation of the active substance with a mixture of acetonitrile and water, purification by solid-phase extraction and HPLC detection with ultraviolet on a diode array. In determining residual amounts of benzovindiflupyr in oil of oil-seed crops and in fruit and vegetable juice, use was made of standard procedures for obtaining processed products². The linear range for the determination of benzovindiflupyr was established to be as 0.01-1.00 mg/kg, which is in compliance with the established Russian and international food safety requirements.

The proposed method was tested on a large list of agricultural plants: vegetables (cabbage, potatoes, cucumbers, onions, etc.), fruits (apples, grapes, peaches, etc.) and grain crops (wheat, oats, rice, etc.). The samples under study were purchased from commercial retail chains.

In all the products studied, the content of benzovindiflupyr was found to be at a level lower than the minimum limit of quantitative determination, i.e. less than 0.01 mg/kg.

DEVELOPMENT OF A METHODOLOGY FOR EXTRACTION NANOPARTICLES FROM VOLCANIC ASH

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Volcanic ash nanoparticles are tiny particles ranging in size from 1 to 100 nm, they can travel long distances and penetrate biological systems, which underscores the importance of studying them. One of the most important features of volcanic ash nanoparticles is their high specific surface area, which allows them to sorb various substances, including toxic ones. The study showed that the concentrations of a number of elements (Ni, Zn, Cd, Ag, Sn, Se, Te, Hg, Tl, Pb, Bi) in volcanic ash particles are 10-500 times higher than the total content of these elements in the bulk samples.

The study of volcanic ash nanoparticles is an interdisciplinary task that requires the use of a complex of sample preparation and analysis methods. The main problem in studying volcanic ash nanoparticles is the low efficiency of extracting nanoparticles from samples. In this work, the method of field-flow fractionation in a rotating coiled column (RCC) for the extracting of nanoparticles from polydisperse samples, proposed at the Vernadsky Institute of Geochemistry and Analytical Chemistry of the Russian Academy of Sciences, was developed.

The effectiveness of six different techniques and two eluents: deionized water and sodium pyrophosphate for the extraction of nanoparticles from an ash sample from the Karymsky volcano (2013 eruption) using RCC was assessed.

Extracting with pyrophosphate was researched in three different variants: with soaking in 2 mM $\text{Na}_4\text{P}_2\text{O}_7$ for 24 hours and preliminary soaking in 0.1 M NaCl, without soaking in NaCl and without preliminary soaking in pyrophosphate. To compare the effectiveness of the methods, the extraction of nanoparticles of volcanic ash in deionized water was also represented. As a result, preliminary soaking in sodium pyrophosphate and its use as an eluent turned out to be optimal and provide the greatest isolation efficiency.

Using this technique, became possible to increase the mass of the extracting nanofraction by 10 times (from 0.1 mg to 1 mg) compared to the extracting process in deionized water. It has been shown that with elemental analysis of nanoparticles it has become possible to determine a number of elements below the limit of detection (Ag, Cs, Tu, etc.).

Expanding the possibilities for studying the elemental composition of volcanic ash nanoparticles is promising for solving fundamental problems in geochemistry.

STUDYING THE ANTIGENE-BINDING ABILITY OF ANTIBODIES IMMOBILIZED ON GOLD NANOPARTICLES

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Conjugates of gold nanoparticles (GNPs) and antibodies immobilized on their surface are widely used in a variety of bioanalytical systems. Therefore, information about the composition of these conjugates and the degree to which the antigen-binding activity of antibodies is stored after their immobilization is extremely important. In this work, original approaches based on recording the fluorescence of antibodies (provided primarily by tryptophan residues) and antigen are proposed to evaluate these characteristics. We used the model antigen fluorescein in the forms of carboxy- and amino derivatives and monoclonal antibodies (MAbs) to it. Conjugates of spherical GNPs with average diameters of 14.0; 18.5; 21.0; 31.5 and 35.5 nm, having different degrees of surface coverage with adsorbed antibodies – monolayer, half and quarter of monolayer. With monolayer immobilization, the proportion of antibodies capable of binding carboxyfluorescein varied from 6% to 17%. The charge localized outside the antigenic determinant significantly influenced this parameter: for GNPs with an average diameter of 21.0 nm, the percentage of immobilized antibody sites that bound aminofluorescein reached 27% compared with 13% in the case of carboxyfluorescein. A decrease in the coverage degree of the GNP surface with antibodies did not lead to significant changes in the proportion of active binding sites compared to the monolayer. The results obtained reflect significant inactivation of antibodies during their adsorption immobilization on GNPs and should be taken into account when choosing interaction conditions in analytical systems.

Based on the data obtained, conjugates of GNPs with MAbs to priority food pathogens – *Salmonella typhimurium*, *Escherichia coli* O157:H7 and *Listeria monocytogenes* were synthesized, and immunochromatographic test systems were developed using them to detect the pathogens in milk. Using the example of *S. typhimurium* detection, the modes of immunoreagents interactions were compared. The test systems can detect up to 3×10^4 CFU/mL of microorganisms in 10–20 minutes.

This research was funded by the Russian Science Foundation, grant number 23-46-10011.

SYNTHESIS AND EXTRACTION OF RARE EARTH ELEMENT CARBOXYLATES BY AQUEOUS AND ORGANIC MEDIA

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Carboxylic acid salts are used in REE chemical technology due to their high activity and solubility in both polar and non-polar solvents. Precipitation of REE carboxylates and extraction with aqueous solutions from organic phases are used for their separation and concentration¹.

The aim of the work is to study the extraction processes of neodymium palmitate and lanthanum, yttrium, and gadolinium oxalates by aqueous solutions as well as by oil, including from polymer matrices.

Neodymium palmitate was synthesized from neodymium oxide and palmitic acid in heptane. It was then extracted from the organic solvent with aqueous solutions of hydrochloric acid with pH 1, 2, 4 and 6. The neodymium content in the obtained aqueous solutions was analyzed by spectrophotometric method at a wavelength of 522 nm. Extraction of neodymium palmitate from crude oil was carried out with aqueous solutions with pH=1, 5, 7, 9, 11.

Lanthanum, yttrium, and gadolinium oxalates synthesized from aqueous solutions of REE chlorides by oxalic acid precipitation were placed in polymer matrices. The extraction was carried out with crude oil, a mixture of water and crude oil (1:1) and distilled water at 25°C and 80°C for 6 h. The extracts were analyzed by optical emission spectrometry with inductively coupled plasma.

Studies have shown that Nd is extracted from palmitate solution in heptane according to the molar content of hydrochloric acid in the extractant. Maximum extraction of Nd from crude oil is achieved at pH=1, with increasing pH the degree of extraction practically does not change. The degree of extraction of La, Y and Gd from oxalates in polymer matrix by aqueous solutions is 30 times higher than the degree of extraction by crude oil. REEs can be arranged in a row by increasing degree of extraction: Gd, Y, La. Extraction by both organic and aqueous medium is enhanced by heating.

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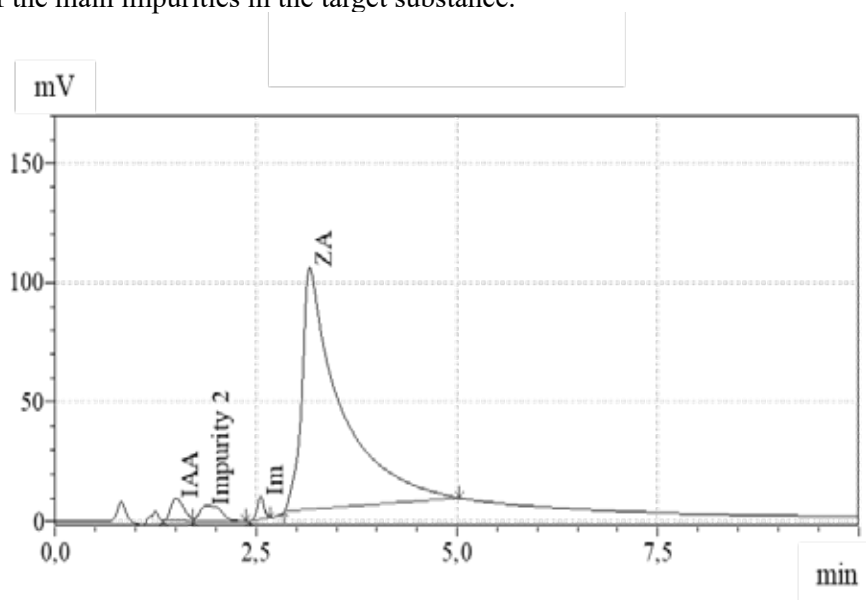
QUANTITATIVE DETERMINATION OF RELATED IMPURITIES IN AMPHOTERIC DRUGS USING MULTIMODAL CHROMATOGRAPHY

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Amphoteric organic substances, as multifunctional compounds with solvent-dependent polarity, are often challenging analytes in drug quality control using reverse phase sorbents. Of particular interest is the study of their chromatographic behavior and mutual separation. To solve this type of problem, multimodal chromatography is a universal and convenient approach, due to the combination of various retention mechanisms¹.

In the presented work, the mechanisms of retention of the main intermediates in the synthesis of zoledronic acid (ZA) were studied on various mixed stationary phases, and a fundamentally new technique was proposed for the quantitative determination of the main impurities in the target substance.



IAA – 1*H*-Imidazol-1-ylacetic acid

Im – Imidazole

ZA – Zoledronic Acid

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SALDI MASS SPECTROMETRY WITH LITHIUM CATIONIZATION – EFFECTIVE TOOL FOR SCREENING POLYPHENOLS IN PLANT EXTRACTS

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Phenolic compounds are the most extensive class of low-molecular-weight plant secondary metabolites, which have an aromatic ring and a hydroxyl group in their structures. The wide range of biological activities of these compounds is important for pharmaceutical cosmetic industries and for other industries. This determines the urgency of developing new methods for searching and identifying phenols, as well as for finding new sources of plants containing these compounds.

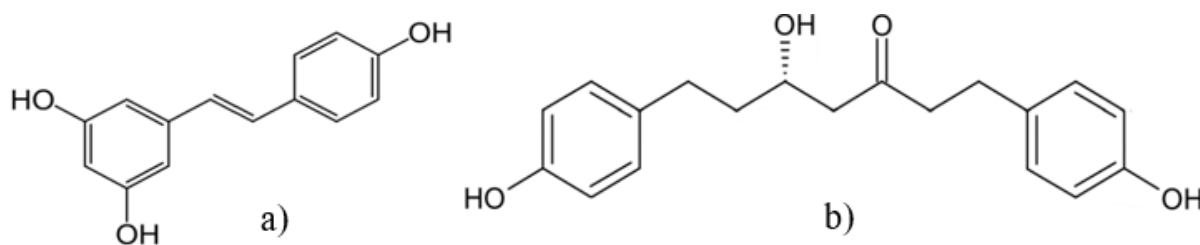


Figure 1. Phenolic compounds: a) stilbenes; b) diarylheptanoids

A promising method for rapid screening of polyphenols in plant extracts is mass spectrometry with surface-assisted laser desorption/ionization (SALDI) with lithium cationization. This approach eliminates interference from the matrix in the lower-mass region, allowing for the acquisition of high-intensity mass spectra, including tandem mass spectra and opens up great possibilities for structural analysis of detectable secondary metabolites.

This approach has demonstrated high efficiency when studying diarylheptanoids and stilbenes, specifically their glycosylated derivatives, in birch and spruce phloem extracts. As a result, 6 diarylheptanoids and their derivatives containing monosaccharides and disaccharides with pentose and hexose sugar residues have been identified in birch phloem extract. Six stilbenes and their glycosylic derivatives with hexose monosaccharide residues were found in spruce phloem extract.

This research was funded by the Ministry of Science and Higher Education of the Russian Federation (grant № FSRU-2024-0004).

DETERMINATION AND SEPARATION OF FOOD ADDITIVES E110 AND E102 IN REAL OBJECTS (BEVERAGES) BY SOLID-PHASE EXTRACTION WITH SPECTROPHOTOMETRIC DETECTION

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One of the methods most suitable for extracting, separating and concentrating food additives is the solid-phase extraction. The purpose of this work was to develop a simple sorption-spectrophotometric method for the determination of food additives E110 and E102 using an adsorbent based on a metal-organic framework (MOF) – copper terephthalate.

Adsorption studies carried out on model compounds (a number of dyes) showed satisfactory adsorption properties of the sorbent, and the adsorption process is best described by the Langmuir isotherm model. The thermodynamic characteristics of the adsorption process were calculated. It has been experimentally established that the synthesized sorbent can function under laboratory conditions without a noticeable loss of efficiency over four operating cycles.

Chromatographic analysis confirmed the presence of food additives in the carbonated drinks analyzed in this study. When a real object was passed through a sorbent, a transparent solution was obtained at the output – the sorbent absorbed both dyes. No food additives were found in this sample; the clear solution contained only sugar. When passing the eluent, food additives were sequentially washed out. Tartrazine (E102) was washed out first, then Sunset yellow FCF (E110). The concentration of each food additive in a separate sample was determined and it was found that their content in the original sample was: E102 – 10.75 mg/L, and E110 – 12.34 mg/L.

The high extraction of the food additives showed that copper terephthalate can be used as an adsorbent to remove food additives in drinks from aqueous solutions and be easily regenerated for reuse. Studies have shown the high efficiency of this sorbent, and with subsequent elution it is possible not only to isolate food additives with good yield, but also to separate them on a chromatographic column.

This work was supported by the Russian Science Foundation, project 22-13-00260.

GC-MS DETERMINATION OF PAHS AND PCBS IN WATER USING EXTRACTIVE FREEZING-OUT

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16 priority polycyclic aromatic hydrocarbons (PAHs) and 7 indicator polychlorinated biphenyls (PCBs) are common pollutants in the environment, regulated for determination in environmental objects by the monitoring programs of the US Environmental Protection Agency (US EPA). PAHs and PCBs enter water bodies through air transport from sources, secondary pollution, with wastewater, etc. The search for effective sample preparation methods for water, ensuring high extraction and concentration of analytes from complex matrices, is relevant. The study of the features of extractive freezing-out as a method of analyte concentration under the influence of centrifugal forces is of interest, as extraction, concentration of analytes, and purification of extracts from major matrix components occur in one step.

This paper presents the results of studies of extraction mixtures with varying contents of the extractant (acetonitrile) for simultaneous extraction of PAHs and PCBs from water. The influence of chloroorganic pesticides on the extraction rates of analytes in the concentration range corresponding to the content of interfering components in real objects was evaluated. The absence of influence of *n*-paraffin hydrocarbons on the analysis of water samples contaminated with petroleum products was demonstrated: under optimized conditions, hydrocarbons were not extracted into acetonitrile extracts. The extraction rates of analytes exceeded 90% in the range of 0.1 – 100 µg/l for PCBs and 0.2 – 100 µg/l for PAHs in their GC-MS determination.

The work was supported by Ministry of Science and Higher Education of the Russian Federation [FZEN-2023-0006].

DEVELOPMENT, STUDY AND CLASSIFICATION OF NOVEL ADSORBENTS FOR HYDROPHILIC INTERACTION LIQUID CHROMATOGRAPHY WITH AMIDE GROUPS AND MACROMOLECULES IN THE FUNCTIONAL LAYER

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The development of new and rapid methods for the construction of functional adsorbent layers for hydrophilic interaction liquid chromatography is an urgent task. To increase the hydrophilicity and selectivity of the stationary phases, as well as the degree of substrate shielding, it is promising to use click reactions and modification with ready-made polymers and large molecules. In this work, a series of novel amide adsorbents have been obtained using a multicomponent and variable Ugi reaction, including those with polymer functional layers. A method of modifying substrates based on silica and polystyrene-divinylbenzene with eremomycin — a large molecule with polar groups, has been proposed.

By using the Ugi reaction to create new layers, it was possible to obtain highly effective amide adsorbents and control their selectivity. In turn, the creation of a polymer layer using this reaction led to significant shielding of the silica substrate. The prospects of modification with eremomycin for the purpose of hydrophilization of various substrates were demonstrated.

The efficiency, selectivity, hydrophilicity and stability of the developed stationary phases were compared with each other and with commercially available adsorbents. A classification of the properties of the resulting stationary phases is presented and it is shown that they can complement the range of the main commercially available phases. The dependence of the retention factors of acidic vitamins on the anion-exchange selectivity of 32 synthesised adsorbents was obtained, allowing the retention of these substances to be predicted.

Examples of the application of the developed stationary phases are given in different fields: pharmacology, enzymology and food industry.

The work was supported by Russian Science Foundation through the grant № 20-13-00140.

X-RAY FLUORESCENCE ANALYSIS OF SMALL MASS SAMPLES FOR GEOCHEMISTRY, GEOECOLOGY AND ARCHAEOLOGY

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Quantitative elemental analysis of relatively small (less than 300 mg) samples is required for the solving of specific scientific issues. The developed technique based on the fusion of 110 mg of a sample with lithium metaborate and further X-ray fluorescence determination of Na, Mg, Al, Si, P, K, Ca, Ti, Mn, Fe and some microelements (Sr, Zr), made it possible to expand the possibilities of X-ray fluorescence method for:

- analysis of cores of bottom lake and peat sediments in order to reconstruct environmental conditions and climate in the past¹;
- analysis of archaeological objects in order to study their origin with the possibility of saving unique artifacts²;
- analysis of samples of snow cover solid phase³ and coal ashes for the environmental monitoring of potentially polluted urban areas.

The advantage of proposed technique is the possibility of further analysis of fused samples using inductively coupled plasma mass spectrometry⁴.

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The research was performed by the governmental assignment in terms of project No. 0284-2021-0005 using the equipment of the Center of isotopic and geochemical Research (Vinogradov Institute of Geochemistry, SB RAS, Irkutsk, Russia) and Geodynamics and Geochronology Center (Institute of the Earth's Crust, SB RAS, Irkutsk, Russia)

DETERMINATION OF STRUCTURAL GROUP COMPOSITION AND KEROGEN DISTRIBUTION IN ROCKS BY IR MICROSCOPY

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The organic matter (OM) present in unconventional reservoir rocks, such as the Bazhenov Formation, is predominantly in a dispersed state (as kerogen). A comprehensive understanding of the chemical composition of kerogen and its distribution in low-porosity reservoir rocks is essential for successful field development and hydrocarbon production. The method of infrared microscopy in the attenuated total reflection mode (ATR) allows to study the local distribution of minerals and kerogen on the rock surface. This method is non-destructive and provides the opportunity to reanalyse or use the sample in further studies. The structure-group composition of minerals and kerogen in the rock can be determined by recording IR-ATR spectra at points on the surface of the sample, the mapping method visualises its distribution. It is possible to semi-quantitatively estimate the kerogen content (as the sum of aliphatic and aromatic fragments) on the rock surface by normalizing the IR spectra by the characteristic absorption bands of functional groups of minerals (clayey – 1100-1000 cm⁻¹; carbonate – 1450-1400 cm⁻¹; siliceous – 800-780 cm⁻¹), aliphatic (2930-2920 cm⁻¹) and aromatic (1630-1600 cm⁻¹) OM fragments.

The mapping of the surface of a sample of siliceous clayey rock of the Bazhenov Formation revealed an extremely heterogeneous distribution of aliphatic and aromatic fragments of kerogen. The content of aliphatic fragments did not exceed 2 wt.%, while that of aromatic fragments did not exceed 9 wt.%. This indicates that the kerogen is located in the rock micropores. The mineral distribution was also highly heterogeneous: quartz content varied from 3 to 79 wt.%, clay mineral content – from 21 to 95 wt.%.

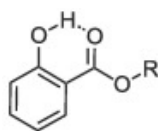
The study was financially supported by Government of Khanty-Mansiysk Autonomous Okrug - Yugra (order from 20.06.2023 № 10-P-1534).

FT-IR AND RAMAN SPECTROSCOPY FOR PREDICTION AND CONTROL OF ALKALI METAL EXTRACTION BY SALICYLIC ACID ESTERS.

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Salicylic acid esters were used as objects of the study:



R= CH(CH₃)₂ (**I**), C₅H₁₁ (**II**), C₈H₁₇ (**III**).

The IR and Raman spectra of salicylic acid esters and Li, Na, K complexes with them were investigated. As a result of the analysis, spectroscopic criteria were proposed, based on which it was determined that salicylic acid esters form complexes with alkali metals in the extracts. The investigations were carried out for crystalline samples and for solutions in chloroform. Since the compound **I** studied earlier showed promise as an extractant¹, **II** and **III** were investigated in terms of translating these possibilities along the homologous series.

Analysis of the spectra of the organic and aqueous phases provides information on the form (free or as part of complexes) of the extractant. On the basis of spectral data recommendations on reduction of extractant losses were proposed. Thus, for the system with **I** the hydrolysis of ester was recorded with formation of alkali metal salicylates, well soluble in water. It was suggested to use more lipophilic extractants **II** and **III** to prevent extractant losses. The study of extraction characteristics of systems with **II** and **III** have proved their stability, which allowed us to propose these esters as effective extractants for the extraction of alkali metals².

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AMPEROMETRIC DETERMINATION OF PYRUVIC ACID ON AN ELECTRODE MODIFIED BY COBALT-PALLADIUM BINARY SYSTEM IN BATCH INJECTION ANALYSIS

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Pyruvic acid (PA) is an important organic biomolecule that plays a fundamental role in the metabolism of fats, proteins and carbohydrates. PA can serve as an important biomarker for various diseases in clinical analysis. Determination of PA levels in human biological fluids can help in the early detection of certain types of cancer, diagnosis and treatment of vitamin B₁ deficiency, type II diabetes and cardiovascular diseases.

In this work, the catalytic activity of a cobalt-palladium binary system (Co-Pd) immobilized on the surface of carbon electrodes (glassy carbon electrode (GCE) and screen-printed electrode (SPE)) during the oxidation of PA under the conditions of cyclic voltammetry and batch injection analysis (BIA) was studied.

PA is not oxidized on unmodified carbon electrodes in the working potential range. On the anodic curve of the cyclic voltammogram of PA oxidation on the Co-Pd-GCE, one peak at E 1.25 V is observed in the region of modifier oxidation. The height of the oxidation peak depends on the concentration of the organic compound. In this case, a multiple increase in current is recorded compared to the oxidation current of the modifier.

A method of the amperometric detection of PA on a Co-Pd-SPE in BIA has been developed. The operating conditions for recording the analytical signal under flow conditions were selected. A linear dependence of the peak current on the analyte concentration is observed in the range from 5×10^{-6} to 5×10^{-2} M. In comparison with static conditions, carrying out analysis in the BI-system allows reducing the lower limit of the determined PA contents by two orders of magnitude.

The developed batch-injection amperometric method of determination is characterized by simplicity and high sensitivity, reproducibility, as well as productivity and rapidity of the analysis method.

The work was supported by the Kazan Federal University Strategic Academic Leadership Program (PRIORITY–2030).

PREPARATION FOR ELEMENTAL ANALYSIS BY ISP-AES/MS METHODS OF PLANT SAMPLES WITH A HIGH SILICON CONTENT

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The studies of plant elemental composition are widely used as a tool for investigation of their growth conditions specific features, as well as the environmental state, especially in places of technogenic pollution. The most convenient methods for multi-element analysis are inductively coupled plasma mass spectrometry and atomic emission spectroscopy (ISP-MS and ISP-AES, respectively), so the transfer of a sample into solution is required. The dissolution of the samples can be carried out in an open system, for example, with a mixture of nitric and perchloric acids. Acid mineralization also successfully occurs in microwave oven with the help of nitric acid or its mixture with hydrogen peroxide. However, when more than 0.1% of silicon is present in plants, one can often observe the formation of silica acid precipitate, which may involve some other elements that need to be measured. Also, knowledge of the silicon content in plant samples provides important information about plant resistance to external influences. This problem can be strongly reduced by adding of a small amount of hydrofluoric acid to the reaction mixture, which, nevertheless, is sufficient to retain silicon in the solution¹.

In this work, for dissolution of dried plant samples (250-300 mg) collected near the Sadono-Unal hollow of North Ossetia by acid mineralization in the MARS-5 microwave system, we used a mixture of hydrogen peroxide (1.5 ml) with nitric (4 ml), hydrochloric (0.5 ml) and hydrofluoric (0.03-0.05 ml) acids. Comparison of the results obtained for a number of reference materials, such as TR-1 (meadow grass mixture), INCT-MPH-2 (grass mixture), etc., showed good agreement with the certified values of the element content.

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WEARABLE GAS SENSOR SYSTEM TO ASSESS THE STATE OF HEALTH BY SKIN ODOR

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Gas sensors are actively being introduced into many areas of human activity, including medical diagnostics. The skin, by releasing volatile compounds, products of metabolic reactions, makes it possible to make the diagnostic method using sensors completely non-invasive and painless. The era of worldwide digitalization dictates the rules for the development of “smart” devices capable of evaluating important parameters of the human body by transmitting information to a personal mobile device.

The work is aimed at developing a bisensory smart bracelet with software that processes a multidimensional analytical signal of the system to assess the state of the body by skin odor. During the research, a mobile device was developed and manufactured, which is a bracelet with a detection cell, in which two piezosensors with a nanostructured solid-state sorbent are located. The principle of operation of the device consists in alternating loading of volatile compounds of each sensitive element of the bisensory system for 30 seconds. Regeneration of the sensitive layer occurs spontaneously in the absence of a load of volatile compounds. The presence of two piezosensors makes it possible to increase the reliability of the information received. Recording of changes in the frequency of oscillation of sensors occurs in the form of double continuous chronochastograms forming a five-dimensional analytical signal. Normalization of sensor output curves makes it possible to remove noise and smooth out the non-identity of sensors, while increasing the reliability of the results. The system was trained on a set of volatile compounds – markers of pathological and natural processes of the body. A set of highly reliable kinetic sorption parameters has been established, which are determined by the nature and concentration of volatile substances. An algorithm for assessing the state of the body by skin odor using a bisensory system has been developed. It was possible to identify the following conditions: hunger, stress, disorders in the gastrointestinal tract and cardiovascular system, the presence of inflammatory processes in the body, a virus, which is consistent with the literature data and established volatile compounds in the bioassays of volunteers.

The work was carried out within the framework of the grant “Start-1 2022” g/k 4838GS1/80291 dated 12/13/2022

APPLICATION OF CAPILLARY ELECTROPHORESIS METHOD FOR QUANTITATIVE ANALYSIS OF MICELLAR DOSAGE FORMS BASED ON HYDROPHOBIZED HYALORNIC ACID

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The capillary electrophoresis method is often used to analyze biopolymers, including hyaluronic acid. The aim of the study is to develop simple and reliable capillary electrophoresis techniques for quantitative analysis of micellar dosage forms based on low molecular weight oleylhyaluronan and its naphthaleimide derivatives. Such dosage forms are polymeric micelles formed by hydrophobized low molecular weight hyaluronan. They are loaded with hydrophobic drug substances (DS). When analyzing such forms, it is important to provide sample preparation to ensure complete release of the drug substance from the micellar medium, which is often difficult. This problem is not present when capillary zone electrophoresis is used.

For quantitative determination of hyaluronan, capillary zone electrophoresis ("Kapel -105M", Lumex Ltd.) was used. Analysis conditions: capillary inner diameter 50 μm , $L_{\text{eff}}/L_{\text{comm}} = 40/50$ cm, $\lambda=190$ nm, 35°C , $U = -20$ kV. The leading electrolyte composition was 0.2M Tris-buffer (pH 3). The migration time of hyaluronan peak ranged from 12 and up to 14 minutes depending on the molecular weight and nature of the hydrophobic molecule in the conjugate. For oleylhyaluronan (15 kDa), the equation of the calibration function is: $y=0.7314 \cdot x$, correlation coefficient 0.9994. The detection efficiency of the polymer was 14072 theoretical plates. The limit of detection was 0.44 $\mu\text{g/mL}$. Thus, taking into account the polymeric nature of the analyzed object, the analytical and metrological characteristics of the presented technique are satisfactory. It is shown that the developed methods allow correct and reproducible determination of various conjugates of low molecular weight HA as well as native HA with different molecular weights. At the same time, the possibility of quantitative determination of drugs loaded in micelles (rifabutin, etc.) for a number of micellar dosage forms under development has been shown.

This work was financially supported by RSCF, project 23-25-00194.

IDENTIFICATION OF LIGNIN STRUCTURAL FRAGMENTS BASED ON TWO-DIMENSIONAL NMR SPECTROSCOPY DATA: MAIN PROBLEMS AND SOLUTIONS

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To date, NMR spectroscopy is the only analytical method that allows the unambiguous determination of lignin fragments. But despite the full potential of this method, the research methodology has not been sufficiently developed. In particular, most researchers carry out identification only based on literary data, without taking into account any criteria for assessing the accuracy of identification, which can lead to significant error.¹

Within the framework of this research, an analytical approach for determining the chemical features of lignin preparations using 2D NMR spectroscopy will be presented. Additionally, a number of methodology techniques will be discussed that allow for accurate identification of known and novel lignin fragments.

Our systematic studies of lignins of various origins have led to the conclusion that following two simple rules avoids serious mistakes. First, it is the presence of all target signals characterizing the atoms in the side chain of lignin fragments; the absence of at least one target signal should cast doubt on the correctness of identification. Secondly, it is the coincidence of the values of the chemical shifts of the target cross peaks with the published data in the range of ± 0.05 and 0.5 ppm for ^1H and ^{13}C , respectively.

Using the example of studying softwood lignin, it is demonstrated how a detailed analysis of the NMR spectra using an expert system including structure search tools can help solve the problem of identifying unknown cross-peaks.

Significant progress in the identification of lignin fragments has also been achieved using chemometric analysis methods.

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IDENTIFICATION OF IRIDOIDS IN A COMPLEX MIXTURE OF PLANT EXTRACTS ACCORDING TO 2D NMR AND HPLC-HRMS DATA

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Iridoids are a group of secondary plant metabolites that belong to the monoterpene class. These compounds have a wide range of biological activities and are often at the heart of the pharmacological properties of many medicinal plants.

The aim of this research is to identify iridoid compounds in members of the *Ericaceae* family, which is one of the major sources of these compounds in the Arkhangelsk region.

The objects of the study were water-methanol extracts obtained through ultrasonic extraction from the stems of *Vaccinium vitis-idaea*, *Vaccinium oxycoccos*, *Vaccinium myrtillus*, *Vaccinium uliginosum* and *Andromeda polifolia*.

To achieve this aim, an approach based on a combination of 2D NMR and HPLC-HRMS methods is proposed, where the analysis of NMR spectra is the first step for the targeted search for the specified type of compounds.

At the first stage, according to the combination of ^1H - ^{13}C HSQC-HMBC spectra, it was found that all five extracts are characterized by a different composition of iridoids. In particular, the spectrum of cranberry extract contains signals characteristic of the structure of the deoxyloganin type, whereas in other extracts there are only compounds of the monotropein type. Based on the obtained NMR data, a targeted search was carried out for compounds containing these fragments using tandem mass spectrometry. As a result, it was possible to detect not only the vaccinoside characteristic of this plant species, but also several previously undiscovered structures.

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CROWN-CONTAINING ORGANIC AND ORGANIC-INORGANIC HYBRID SYSTEMS AS CHEMICAL SENSORS IN LIQUIDS AND GASES

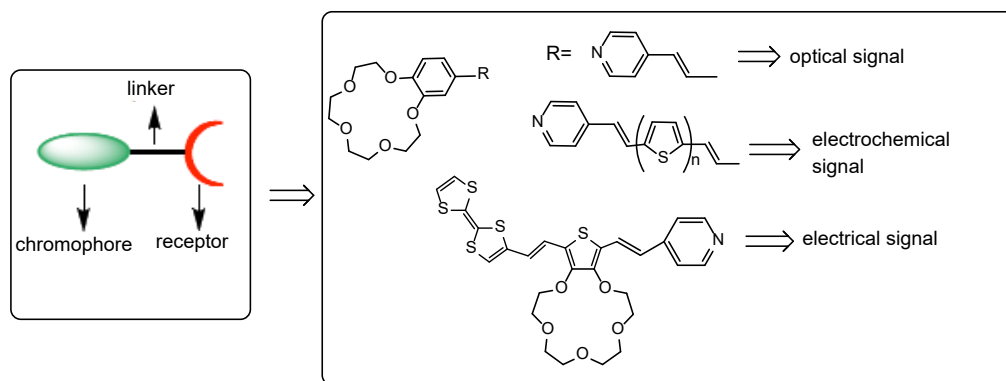
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Sensors are used to continuously monitor the concentration of chemicals for the purposes of environmental safety, optimization of technological processes in production, and medical diagnostics. The main component of analytical instruments is the chemosensory element. Its receptor part ensures binding to a specific type of analyte, while the chromophore part is responsible for the type of analytical signal generated (optical, electrochemical, electrical).

The report presents approaches to obtaining sensors that have crown ether as a receptor part, and also shows possible ways of modifying the chromophore part in order to obtain sensors that demonstrate various types of analytical signals.



The dye molecules presented in the diagram are constructed in such a way that the binding of a metal cation at the receptor part leads to the appearance of an analytical signal. The system is built in a modular manner. Replacing the crown ether fragment leads to a change in the selectivity of complexation; replacing the chromophore part leads to a change in the type of analytical signal observed. In this way, systems were built that exhibited optical, electrochemical, and electrical responses to the analyte.

The work was supported by the Russian Science Foundation No. 23-13-00424.

APPLICATION OF THE PDA-OES METHOD ON A GRAND-EXPERT SPARK ATOMIC EMISSION SPECTROMETER FOR OPERATIONAL MONITORING OF NONMETALLIC INCLUSIONS

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Traditional methods for evaluating non-metallic inclusions (NI) are the analysis of samples under an optical microscope, fractional gas analysis, and surface scanning using a scanning electron microscope with the function of X-ray microanalysis. The main advantage of the PDA-OES method is the high speed of analysis, which allows for prompt quality control of the composition and non-metallic inclusions directly during manufacturing. The aim of this work was to compare the number of intensity peaks of the spectral lines of the elements corresponding to the NI during PDA-OES analysis with the qualitative and quantitative assessment of these NI by scanning electron and optical microscopy. A standard sample of pure iron CRM 10525-93 was used as a material for research. PDA-OES analyses were performed on a Grand Expert atomic emission spectrometer manufactured by: VMK-Optoelectronics LLC with subsequent processing of the results in the original software. The optical microscope study was carried out according to the ASTM E1245-03 method, the dependence of the number of inclusions on their size was constructed and compared with a similar dependence based on the results of PDA analysis. Surface analysis using a scanning electron microscope (SEM) with X-ray microanalyzer showed that the main groups of inclusions in the sample are nitrides. The results of sample analysis using the SEM, ASTM, and PDA-OES methods showed a good correlation in the amount and types of NI in the iron sample, which in turn indicates the potential effectiveness of the method as a tool for operational control.

SORPTION CONCENTRATION OF ANALYTES USING GRAPHENE OXIDE FOR ICP-MS, ICP-OES AND TJP-OES ANALYSIS OF SOLUTIONS

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Determination of concentrations of heavy metals (HMs) in drinking, natural, waste waters and biological fluids is an important task. At the same time, the capabilities of existing methods for quantitative chemical analysis of waters and biological fluids are not always sufficient to control HMs concentrations at the required level. Preliminary sorption concentration of traces allows significantly improve the limits of detection (LODs) of analytes. A current area of research is the development of methods for quantitative chemical analysis using carbon sorbents, in particular graphene oxide, for preliminary concentration of trace elements. This material is obtained by exfoliation of the graphite oxide by microwave irradiation. It is also possible to reduce the LODs of analytes by using an electrothermal vaporization (ETV) – alternative high efficient method of samples introduction into the excitation and ionization sources.

Methods of quantitative chemical analysis of natural, drinking, waste waters and biological fluids have been developed using inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES) and two-jet arc plasma optical emission spectrometry (TJP-OES) with sorption preconcentration of HMs and other analytes, including using of ETV for samples introduction. The LODs of analytes from $n \cdot 10^{-11}$ to $n \cdot 10^{-8}$ % wt have been obtained. Sorption concentration procedures and instrumental parameters of quantitative chemical analysis have been optimized. The influence of sorption conditions and the presence of matrix components on degree of sorption of analytes has been studied. The possibility of increasing the preconcentration factor and improving the LODs of analytes during ETV-ICP-OES and ETV-TJP-OES analysis of suspensions of trace elements concentrates has been shown.

The study was funded by the Russian Science Foundation, project № 23-23-00058, <https://rscf.ru/en/project/23-23-00058/>

STUDY OF METABOLOMIC PROFILES OF PATIENTS WITH GENITOURINARY ONCOPATHOLOGIES USING UNTARGETED LIQUID CHROMATOGRAPHY-MASS-SPECTROMETRY BASED PROFILING

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Malignant tumors of the genitourinary system are among the most common oncopathologies affecting vital internal organs. Bladder cancer (BC) is characterized by a high cancer-specific mortality due to an absence of symptoms or their similarity to those of other illnesses of the urinary tract in the initial stages of disease development.

Traditional diagnostic methods allow to determine the diagnosis, however, have certain disadvantages, among which, depending on the type can be highlighted painfulness and initiation of inflammatory processes – for studies of invasive nature, low sensitivity for some types of tumors – in the case of non-invasive methods.

Obtaining characteristic profiles of biological samples containing a large number of metabolites with different physicochemical properties and a wide range of concentrations poses a major analytical challenge. The chromatography-mass spectrometry method, due to its high sensitivity, is a key approach to perform metabolomic studies, allowing to determine many metabolites from different biochemical pathways and at different concentration levels. The largest metabolomic profile for patients with BC can be obtained by untargeted urinary profiling due to metabolites' accumulation in the immediate surroundings of the tumor.

The work considers sample preparation, selection of conditions of HPLC-MS analysis, and metabolomic profiling of urine samples by liquid chromatography-high-resolution mass spectrometry to obtain the largest possible number of metabolites. Urine samples from BC patients, as well as healthy patients and patients with urinary tract diseases were analyzed.

The results obtained can be used in the detection of complex metabolic abnormalities associated with BC and can complement current methods of diagnostics of such type of disease at different stages in the future.

SELECTIVE VOLTAMMETRIC DETERMINATION OF ASCORBIC ACID, PARACETAMOL AND PHENYLEPHRINE ON AN ELECTRODE MODIFIED BY RHODIUM AND IRIIDIUM OXIDES IN DRUGS

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Ascorbic acid (AA), paracetamol (PA) and phenylephrine (PE) are frequently used as the components of anti-inflammatory and antipyretic drugs with different brands of complex medications.

If the recommended dosage of complex drugs is not observed, serious complications can develop, so it is necessary to control the content of AA, PA and PE in these drugs. One of the simplest and most effective method for solving this problem is the method of voltammetry with chemically modified electrodes.

In this paper, we fabricated on a glassy carbon electrode (GCE) modified by rhodium and iridium oxides, studied electrocatalytic activity of metals particles and a binary system based on them during the oxidation of AA, PC, and PE.

The use of chemically modified electrodes under cyclic voltammetry conditions leads to the registration of signals of the oxidation of analytical compounds at the oxidation potentials of the modifier.

The maximum currents are observed in chemically modified electrodes with the Rh-IrO_x binary system. The height of the signals is linearly related to the concentration of AA, PA and PE in the solution.

The use of a composite electrode compared to clear GCE allows one to increase the sensitivity of determination and reduce the lower limit of the determined content to 1×10^{-6} M.

The results obtained were used to develop a selective voltammetric method for the determination of AA, PA and PE in drugs. The correctness of the methods was assessed using the introduced-found method. The relative standard deviation (S_r) does not exceed 5.0% over the entire range of concentrations studied.

The work was carried out at the expense of the Strategic Academic Leadership Program of the Kazan (Volga Region) Federal University ("Priority-2030").

VOLTAMMETRIC DETERMINATION OF SULPHANAMIDES ON AN ELECTRODE MODIFIED BY COMPOSITE BASED ON GOLD PARTICLES, CARBON NANOTUBES AND IONIC LIQUID

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Sulfonamides are widely used in veterinary practice to combat infectious diseases and as feed additives to stimulate growth and increase the productivity of livestock and poultry. Consumption of food products of animal origin with residual amounts of antibacterial drugs poses a risk to human health. The methods of enzyme immunoassay and HPLC used today for the determination of sulfonamides are characterized by high cost, complexity of analysis and the need for specific reagents. Therefore, the development of alternative highly sensitive methods for the determination of sulfonamides is an actual task.

In this work, we studied the possibility of voltammetric determination of sulfamethazine, sulfacetamide and sulfathiazole on a glassy carbon electrode (GCE) modified with gold particles and a composite based on carbon nanotubes and ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate (Au-MWCNT-IL-GC). Electrochemical impedance spectroscopy was used to evaluate electron exchange conditions at the interface.

During the electrooxidation of the sulfonamides under consideration, poorly defined peaks are observed on the GCE at fairly high anodic potentials ($E_s > 1.20$ V). When scanning the potential in a solution of sulfonamides on Au-MWCNTs-IL-GCE, a peak is recorded at Au potentials, the current of which linearly depends on the concentration of substrates and is controlled by the kinetics of the chemical reaction.

Voltammetric methods have been developed for the determination of sulfamethazine, sulfacetamide, and sulfathiazole on Au-MWCNT-IL-GCE. A linear dependence of the analytical signal on the concentration of analytes is observed in the range from 1×10^{-7} to 5×10^{-3} M. The proposed method for determining sulfonamides was tested in the analysis of drugs and milk.

The work was carried out at the expense of the Strategic Academic Leadership Program of the Kazan (Volga Region) Federal University ("Priority-2030").

FLOW INJECTION AMPEROMETRIC DETERMINATION OF LACTULOSE ON THE ELECTRODE MODIFIED BY GOLD PARTICLES

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Lactulose is a synthetic disaccharide consisting of fructose and galactose. Due to metabolism in the large intestine, lactulose has a prebiotic effect. This is why lactulose is widely used in the food industry. Chromatographic and spectrophotometric methods are used for quantitative detection of lactulose. But these methods are expensive and time-consuming to implement. Therefore, the creation of non-enzymatic sensors with a stable, sensitive and selective response to lactulose is of undoubted interest.

In this work, the feasibility of voltammetric and flow injection (FI) amperometric determination of lactulose on the screen-printed electrode (SPE) modified by gold particles (Au-SPE) was studied.

Lactulose is oxidized irreversibly at E 1.20 V on the SPE. It has been established that during the oxidation of lactulose in an alkaline medium on the Au-SPE, one peak on the anodic curve of the voltammogram at E 0.25 V is observed. The height of the oxidation peak linearly depends on the concentration of the compound. There is a decrease in the overvoltage of lactulose oxidation and a multiple increase in its oxidation current relative to the oxidation current of the modifier.

A method of the amperometric determination of lactulose on the Au-SPE in a FI-system is proposed. A linear dependence of the analytical signal on the lactulose concentration is observed in the range from $5 \cdot 10^{-6}$ до $5 \cdot 10^{-3}$ M. It should be noted that the analysis results are highly consistent in a flow system ($S_r < 2.0\%$). The developed method for determining lactulose is characterized by high sensitivity, repeatability, as well as rapidity and productivity of the analysis method (up to 180 determinations/hour).

The proposed method is used to determine lactulose in commercially available milk samples. The presence of matrix components does not interfere with the determination of lactulose.

SELECTIVE VOLTAMMETRIC DETERMINATION OF PURINE NUCLEOSIDES ON ELECTRODES MODIFIED BY COMPOSITES BASED ON POLY(3,4-ETHYLENEDIOXYOPHENE)

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Structural fragments of DNA - purine nucleosides (guanosine and adenosine) play an important role in the biochemical processes of the human organism. Changes in the concentrations of guanosine and adenosine in biological fluids are used to monitoring and diagnostics of various pathological changes.

In this work electrochemical of purine nucleosides oxidation on glassy carbon electrode (GCE) modified by film of hexacyanoruthenate ruthenium (HCRRu) and composite based on poly-3,4-ethylenedioxythiophene (PEDOT) with incorporated HCRRu (HCRRu-PEDOT) with the aim of developing selective and sensitive method for the voltammetric determination of adenosine and guanosine was studied.

The immobilization of the HCRRu film and the HCRRu-PEDOT composite was carried out by electrochemical deposition. The formation of nanostructured and uniformly distributed HCRRu particles with a diameter of 20 nm to 40 nm on the electrode surface has been proven using atomic force microscopy.

It is established that HCRRu-film shows catalytic activity towards of guanosine and adenosine oxidation. Difference in the oxidation potentials of guanosine and adenosine is 300 mV. The electrocatalytic effect for both nucleosides increases from the HCRRu-GCE to the HCRRu-PEDOT-GCE. The value of this effect correlates well with the calculated values of the electrochemical reaction rate constants. The conditions for the immobilization of modifiers on a GCE with the best catalytic characteristics of guanosine and adenosine oxidation are determined.

The possibility of guanosine and adenosine simultaneous voltammetric determination on the HCRRu-PEDOT-GCE electrode has been established.

The lower bound of the determine amount makes $5 \times 10^{-7} \text{M}$ for both compounds. The developed voltammetric method of determination was tested in the analysis of eye drops containing adenosine.

The work was supported by the Kazan Federal University Strategic Academic Leadership Program (PRIORITY-2030).

COMPARISON OF THE EFFECTIVENESS OF SORBENTS OF DIFFERENT ALLOTROPIC FORMS OF CARBON INCLUDING MODIFIED CARBON NANOTUBES

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Comparative sorption of toxic ions: Be(II), Bi(III), Cd(II), Cr(III), Pb(II) and noble metals Ag(I), Au(III) and Pd(II) from aqueous solutions was studied on carbon nanotubes (CNTs), a magnetic nanosorbent consisting of CNTs and magnetite nanoparticles (CNT@MNPs), and activated carbon (AC). A significant advantage of sorbents with CNTs over AC has been shown. A mechanism has been proposed for the sorption of magnetite nanoparticles (MNPs) on CNTs of various morphologies in the process of additional modification of the latter and the dependence of the sorption capacity of the synthesized magnetic composite on the morphology of CNTs obtained on iron group catalysts: Ni, [CNT (Ni)]; Co, [CNT (Co)] and Fe, [CNT (Fe)]. The advantage of CNT@MNP composites over individual CNTs in relation to all studied sorbates is shown, not only for the separation of solid and liquid phases in magnetic solid-phase extraction (MSPE), but also to demonstrate the additional possibilities of using CNTs of any morphology, regardless of the conditions of their synthesis. It was shown that the optimal CNT:MNP ratio in the composite equal to 10:1 for all sorbates provides maximum sorption capacity and magnetization sufficient for effective phase separation after sorption of all studied elements. Using synthesized sorbents the arc atomic emission methods of analysis have been developed and comparative metrological characteristics are presented for determining the studied elements in aqueous solutions using their preliminary concentration on carbon sorbents.

SORPTION OF SUBSTANCES OF DIFFERENT NATURE BY ALUMINOSILICATE AND ITS APPLICATION IN CHEMICAL ANALYSIS

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Sorption materials are widely used in chemical analysis at the stage of preliminary concentration of substances and leveling the interfering effect of the matrix. Aluminosilicates have proven themselves well as sorbents, characterized by their high sorption capacity, economic availability, and the ability to modify their surface to impart the required properties.

The work systematically studied the sorption properties of natural aluminosilicate from the Khotynets deposit in relation to Pb(II), Zn(II), Hg(II), Cd(II), Ni(II), As(III), Fe(III), U(VI) and Sr(II), as well as organic compounds: 23 pesticides of various classes (organophosphorus and chlorine, synthetic pyrethroids, triazoles), PAHs (benz(a)pyrene), tetracycline antibiotics (tetracycline, chlortetracycline, oxytetracycline, doxycycline , demeclocycline).

Sorption has been studied from the following solvents for pesticides: acetone, acetone-water (1:1), water, hexane, acetonitrile. Sorption was studied from aqueous systems for the remaining compounds. The influence of medium acidity and phase contact time on the degree of analyte recovery was determined. The sorption capacity of the mineral for each of the analytes was established. The distribution coefficients are found. Based on IR spectroscopy data, an assumption was made about the probable chemistry of the processes.

Methods have been developed and tested, including the stage of preliminary concentration or purification of samples: determination of pesticides by GC-MS in grain crops and natural waters; determination of tetracycline antibiotics in natural waters using HPLC-MS/MS and in dairy products using HPLC-UV; colorimetric and test for the determination of tetracycline and doxycycline in milk and dairy products; atomic absorption determination of Hg(II), Cd(II), and As(III) in natural and waste waters; determination of benzo(a)pyrene in some light industry objects using HPLC with fluorimetric detection.

The advantages of the proposed methods are high sensitivity and reproducibility of results, economic accessibility and the possibility of replacing imported sorption materials.

APPLICATION OF PHYSICS INFORMED NEURAL NETWORKS FOR PROCESSING THE RESULTS OF FRACTIONAL GAS ANALYSIS

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Fractional gas analysis (FGA) is a modification of the method of high-temperature extraction of oxygen and nitrogen from a sample in a carrier gas under a given heating mode. This method is based on differences in the thermodynamic stability of oxides and nitrides and the possibility of their fractional separation during monotonous heating in the analyzer furnace. In this case, non-metallic inclusions (NI) are reduced by carbon to form CO. After that, CO is re-oxidized to carbon dioxide CO₂, which is recorded by the IR cell of the LECO TC600 gas analyzer. Thus, at the output, the device provides information about the dependence of the rate of CO₂ extraction from the sample on temperature. One of the difficulties of the FGA is the complexity of interpreting the analysis results, which is associated with the a priori uncertainty of mathematical data processing. As part of this work, a method based on neural networks that takes into account physical information was developed to interpret the results of FGA. The proposed algorithm allows us to estimate the CO₂ extraction curve during the carbothermic reduction of each type of oxide inclusions potentially contained in the sample. To achieve this, the neural network is trained to predict the CO₂ extraction rate for each type of oxide inclusion by temperature. The training is made by minimizing the loss function using Adam gradient-based optimization method. The loss function includes terms responsible for the correspondence of the predicted functions to the differential equation simulating the process of CO₂ extraction rate from the sample during non-isothermal reduction of NI with carbon. The developed algorithm was tested on a standard sample CRM 9110-2008 of steel composition ShKh15 with a certified total oxygen content of 9±1 ppm. The results of testing the proposed algorithm showed that the standard deviation of the determination of oxygen in aluminum oxide is 0.8 ppm, which corresponds to the results obtained by processing in the OXSEP PRO software (0.7 ppm).

INVESTIGATION OF SORPTION PROPERTIES OF THE COMPOSITE MATERIAL GRAPHENE OXIDE/ CARBOXYMETHYLCELLULOSE/IRON NANOPARTICLES WITH RESPECT TO Pb(II) AND Zn(II) IONS BY ICP-AES

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Recently, the issues of water pollution have become increasingly significant due to the rapid pace of urbanization, industrialization and agriculture activities¹. One of the most promising approaches to improving water quality is the development of novel of sorption materials with enhanced characteristics².

This study focuses on a composites or bent composed of grapheneoxide, carboxymethylcellulose, and iron oxide nanoparticles.

The content of Pb(II) and Zn(II) ions in aqueous solutions was determined using inductively coupled plasma atomic emission spectroscopy(ICP-AES) employing the iCAP-6500 Duo spectrometer (Thermo Scientific).

It was found that the maximum values of the sorption capacities of materials with different iron oxides contents, when isolation of Pb(II) and Zn(II) ions from aqueous solutions, were 680 and 387 mg/g, respectively. The obtained kinetic curves of sorption can be described by a pseudo-second-order model (with a highest coefficient of determination). It was shown that the synthesized composite material is efficient for the selective removal of heavy metals from contaminated aqueous media³.

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DETERMINATION OF LEACHING RATE OF SODIUM ALUMINUM IRON PHOSPHATE GLASS COMPONENTS BY ICP-AES METHOD

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The principle of the need to increase the safety level of management of generated and accumulated radioactive waste (RW), consisting in the conversion of all types of waste into solid chemical- and radiation-resistant matrices for subsequent temporary controlled storage and/or final disposal, is currently being realized in Russia. Previously, sodium aluminum iron phosphate (NAFP) glass of optimal composition (mol.%) $40\text{Na}_2\text{O}-10\text{Al}_2\text{O}_3-10\text{Fe}_2\text{O}_3-40\text{P}_2\text{O}_5$ was proposed for immobilization of high-iron RW.¹ At the same time, the composition of the obtained glass may differ from the optimum one at solidification of waste of complex chemical composition containing metals in significant amounts. In this work, NAFP glass samples were obtained by varying the composition of the structure-forming components and their hydrolytic resistance was investigated.

The hydrolytic resistance of NAFP glass samples is the main criterion for the applicability of matrices for RW solidification, which was evaluated by the differential leaching rate of the components. Leaching resistance of samples was determined in dynamic conditions in Soxhlet apparatus according to the standard "Powder" method, as well as in static conditions according to the PCT test. Research conditions: crushed glass sample – particle size 0.01–0.02 mm; leaching agent – bidistilled water; medium temperature – 95°C ("Powder") and 90°C (PCT), total leaching time – 10 days ("Powder") and 7 days (PCT). The solution was taken after leaching the samples and acidified with concentrated nitric acid to pH~1. The content of elements in solutions after leaching was determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) using an iCAP-6500 Duo spectrometer (Thermo Scientific).

The high stability of NAFP glass to leaching of matrix-forming components under various conditions simulating waste disposal in storages was established.

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AUTOMATED ONLINE MAGNETIC SOLID-PHASE MICROEXTRACTION OF PHENOLIC TOXICANTS

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The use of magnetic sorbents for concentration is an effective tool for extraction of substances from complex and contaminated matrices of various compositions. The magnetic properties also make it possible to control the sorbent and create automated systems for online magnetic solid-phase microextraction, including all stages of the process (analyte sorption, desorption and sorbent regeneration).

Various designs of cartridges for concentration have been proposed for automated systems. Sorption concentration can be carried out in a column in which the sorbent is immobilized using two neodymium magnets. Another option is a cartridge with a micro mixer, in which the sorbent is immobilized on the walls of the vessel and is held by magnets located on the outside. The magnets are moved away and brought closer to the column using a manipulator controlled by a servomotor.

A compact programmable relay was used as the digital controller of the automated installation. A personal computer with pre-installed specialized software was used as a source of the setting effects of the digital controller. To output and edit the text and digital parameters of the system, a compact character panel with controlled logic was used in conjunction with controllers or input and output modules connected via the RS-485 network.

Dynamic online concentration techniques have been developed for phenolic toxicants and phenol derivatives (non-ylphenol, octylphenol, bisphenol A, chlorophenols and dichlorophenoxyacetic acids) using magnetic sorbents based on humates, super-crosslinked polystyrene and coals.

RB-SR METHOD AGE DETERMINATION OF GEOLOGICAL SAMPLES USING GAMMA ACTIVATION ANALYSIS

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Gamma-activation analysis (GAA) was applied for determination of the elemental content and the age estimating of geological samples by rubidium-strontium dating method.¹

Advantage of improved Sr/Rb method is to calculate ^{87}Sr content without mass-separation. Isotope $^{87\text{m}}\text{Sr}$ ($E_{\gamma} = 388$ keV, $T_{1/2} = 2.81$ h) can be produced by two ways: $^{87}\text{Sr}(\gamma, \gamma')^{87\text{m}}\text{Sr}$ and $^{88}\text{Sr}(\gamma, n)^{87\text{m}}\text{Sr}$. But to minimize the second side way of $^{87\text{m}}\text{Sr}$ production, the energy of the beam should be near $E_{\gamma} = 10$ MeV.²

Reference sample Siberian Trap (CT-1A) with a known age was used as a standard in this work.³ Samples and the standard were simultaneously activated using bremsstrahlung produced by electron accelerator microtron MT-25, FLNR JINR. For the elemental content determination and age estimation were used energies of beam $E_{\gamma} = 24$ MeV and $E_{\gamma} = 10$ MeV, respectively. Data on the contents of Rb and Sr in the samples were obtained using XRFA to complement GAA. The basic equation of decay and data recommended by the Sub-Commission on the chronology of the International Union of Geological Sciences were used for calculations.¹ Based on the obtained results, a classification of geological samples and an estimate of their age were carried out.

This age estimating method of geological samples using GAA was tested on samples taken in the western part of the Tibetan plateau near the mount Kailash⁴ and on the Gobi Desert samples.

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FIBERS BASED ON SINGLE WALLED CARBON NANOTUBE FILMS – A NEW ELECTRODE MATERIAL FOR ELECTROCHEMICAL ANALYSIS

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Currently, the development of sensors for the rapid detection of various substances, including real-time analysis is a crucial area of research in the field of analytical chemistry. The use of electrochemical response is promising, as it allows for the variation of analytical characteristics through the use of different electrode materials and surface modification techniques.

A new method has been developed to create fiber sensors using single-walled carbon nanotube (SWCNT) films, based on the “wet pulling” technique [1], for the quantitative determination of various analytes, both inorganic (mercury, lead, cadmium, and arsenic) and organic (phenol, dopamine, and ascorbic acid), using voltammetry (VA). SWCNT films were produced using the aerosol CVD method. By assembling SWCNT bundles in the form of a strong free-standing fiber with almost entire surface accessible to an analyte, we are able to avoid the interference of substrate and improve detection efficiency. The influence of physical, chemical and electrochemical pretreatments of the new electrode SWCNT material on its properties was examined. Transmission and scanning electron microscopy, cyclic voltammetry, chronopotentiometry and electrochemical impedance spectrometry were used to characterize the SWCNT fibers.

Methodological approaches to the surface preparation and modification of the electrode material for the selected analyte, using Au and $\text{Fe}_2\text{O}_3/\text{Pd}$ nanoparticles, have been optimized. The optimal conditions for recording an analytical signal on model objects have been experimentally established. The possibility of using sensors in the analysis of real samples was investigated and the results were compared with standard methods. The achieved detection limits make it possible to confidently determine these metals and arsenic in the stripping VA mode at the MPC level for drinking waters; in the case of phenol, dopamine and ascorbic acid in the mode of adsorptive VA and differential pulse VA on unmodified sensors, it is possible to determine these compounds at the level of contents $2\text{--}10\ \mu\text{g L}^{-1}$.

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ION MOBILITY SPECTROMETRY OF HALOGENOALKANES

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Halogenoalkanes, which are commonly used in organic synthesis, pose hazard to human health. The task of express-detection of this class of substances can be successfully solved by using Ion Mobility (IM) Spectrometry method.

Research conducted on automatic stationary gas analyzer «Segment», manufactured by the «Yuzhpolimetal-Holding» group of companies. Characteristic signals (values of coefficient reduced IM (CRIM)) in both the positive and negative ionization regions were determined as a result of analysis IM spectrum for all investigated compounds

Python program (language version 3.3) was used to process «Segment» data, allowing to analysis and research IM spectrum.

In the negative ionization region, the characteristic signal of bromide ion¹ is registered for all investigated alkyl bromides (table 1).

Table 1. Value of CRIM in negative ionization region

Sample	MeBr	EtBr	i-PrBr	n-PrBr	n-BuBr
CRIM cm ² / (V·s)	2,4753	2,4552	2,4711	2,4622	2,4523

Individual registration of each of the halogenoalkanes is possible due existence of different CRIM values in positive ionization region, which makes it feasible to selectively detect them in the air environment (table 2).

Table 2. Value of CRIM in positive ionization region

Sample	MeBr	EtBr	i-PrBr	n-PrBr	n-BuBr
CRIM cm ² / (B·c)	2,4768	1,6424	1,7932	1,7932	2,2987 1,6424

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RAMAN SCATTERING SPECTROSCOPY OF ISOBUTANOL-WATER SYSTEM

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To ensure national security, modern technical solutions are required. In addition to ion mobility spectrometry, rapid detection by Raman scattering spectroscopy¹ is becoming widespread. Russian portable Raman spectrometer (PRS) «ChemExpert-T» was used in the research. The undoubted advantage of the method used for analyzing chemical compounds is its non-destructive and non-contact nature. The sample examination is carried out without sample preparation and without compromising the integrity of the object's packaging.

To expand the registration capabilities of PRS, model isobutanol-water solutions of various concentrations were studied. To analyze a series of Raman spectra and superimpose the results obtained, a computer program was developed in the Python programming language (version 3.11.8). The series includes spectra of distilled water and isobutyl alcohol for comparison of characteristic signals. The results obtained at constant exposure time are presented in Table 1.

Table 1. Value of Raman shift

Concentration, mg/ml \ Raman shift	Intensity, relative units		
	819.343 cm ⁻¹	1463.284 cm ⁻¹	2700.918 cm ⁻¹
38,181	9489,599	5866,848	2312,987
15,722	5108,946	3132,011	1509,888
7,939	5108,946	1845,008	919,417
3,989	3383,354	1363,809	798,761
1,999	2533,591	1210,533	678,571

For isobutyl alcohol, a comparison was made with the signals of the Raman spectrum of distilled water. By changing the values of the characteristic signals, one can judge the change in the concentration of isobutanol in the solution and its threshold value in the system, available for detecting isobutanol using PRS «ChemExpert-T».

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ANALYSIS OF GEOLOGICAL AND TECHNOLOGICAL MATERIALS WITH CLOSED VESSELS DECOMPOSITION IN BOTTOM HEATING RESISTIVE SYSTEMS

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Modern closed vessels decomposition systems with the bottom resistive heating creates possibility to significantly enhance the possibilities of sample preparation for using atomic spectrometry methods.

The dissolution of geological and technical materials in a mixture of acids HNO_3 , HCl , HF at a temperature of 250°C was investigated in this work. Comparing with dissolution in open or closed vessels at $180\text{--}200^\circ\text{C}$, this approach ensures complete decomposition of titanium and chromium minerals presented in geological samples. The dissolution was carried out in 2 stages: HCl , HNO_3 and HF were added in the first stage, and H_3BO_3 solution was added in the second stage to bind excess hydrofluoric acid. In the case of sulfide, sulfate, and phosphate samples, HNO_3 and HF were used in the first stage, and HCl and H_3BO_3 solution were used in the second stage. Compared with standardized methods using dissolution in 4 acids (HCl , HNO_3 , HF , HClO_4), acid consumption is significantly reducing, more complete decomposition of samples is reached, and productivity increases. The total time to prepare a batch of 54 samples is 7 hours. When using several systems with bottom resistive heating, it is possible to increase this number of samples analyzed by 2-4 times.

The decomposition samples of coal and polymer resins for gold sorption from technological solutions of heap leaching was investigated. Usually, it takes about 7 hours of working time, 10 cm^3 of concentrated H_2SO_4 , $25\text{--}50\text{ cm}^3$ of concentrated HNO_3 , and continuous presence of personnel to decompose the $0.1\text{--}0.2\text{ g}$ coal or polymer resin samples on an open plate. Our research has shown that closed vessel dissolution of a coal sample weighing $0.1\text{--}0.125\text{ g}$ and of polymer resins weighing $0.1\text{--}0.2\text{ g}$ at 250°C , it is sufficient to have 5 cm^3 mixture of sulfuric, nitric and hydrofluoric acids. With a 5-10-fold reduction in the volume of acids used and the corresponding emissions, the decomposition time is significantly reduced by 3-4 times.

REDUCING OXYGEN BY SORPTION ON EVAPORATED SALTS MATRIX IN THE CARRIER GAS HOT EXTRACTION

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Determination of oxygen impurities in metal halides is a difficult goal for most modern methods of analytical chemistry. Nowadays, the carrier gas hot extraction method is often used for quick and accurate determination oxygen impurities in solid samples¹. This method is adapted for the determination of oxygen in salts now.

Carrier gas hot extraction is based on the extraction of oxygen by reaction with the crucible graphite at high temperature. The oxygen in the form of CO or CO₂ is transported by the carrier gas to the detector. Despite the large number of publications on metal alloys, there are few articles devoted to salts.

Scientific works dedicated the factors affecting the results of oxygen determination in the analysis of salts aren't enough². CO sorption on sublimated salts is one such important factor. The salts are easily sublimated and precipitated on parts of the system due to low boiling points and become a sorption layer.

The Horiba EMGA 620 W/C system was used to investigate the effect of sorption on salt sublimates.

The research was carried out with salts that are actively used or are prospective for industry (LiF, NaF, KF, LiCl, AlCl₃). Calcined and pressed yttrium oxide was used as a sample with known oxygen content.

The contribution to the error of the effect of sorption on salt substrates in the determination of oxygen under real analytical conditions was estimated.

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ON METHODOLOGICAL ISSUES IN MEASUREMENTS OF THE MELTING TEMPERATURE OF ORGANIC SUBSTANCES

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Most of the operating measuring instruments (MI) provide the ability to measure melting point of organic substances in the range from room temperature to 350 °C - 400 °C. Measurement range of up to 250 °C has the greatest practical significance for the pharmaceutical and medical industry, in which the measurement uncertainty of the used MIs is 0.3 °C – 1.5 °C.

An analysis of metrological tools available for such MIs, as well as the results of interlaboratory comparison¹, showed that current metrological support has a number of limitations that negatively affect the uniformity of measurements in this field. The analysis showed significant differences between the certified values of reference materials (RMs) produced by various manufacturers, exceeding their claimed uncertainties; and lack of a unified approach to interpreting differences between measurement results obtained at various heating rates.

VNIIM calorimetry laboratory started a research project in the field of measuring melting point of organic substances. A new working standard was designed to reproduce the unit of temperature and certify pure organic substances for melting point in the range of 40 °C to 250 °C. Experimental studies of substances – analogues of RMs of various categories were carried out, new approaches to verification and calibration of MIs, as well as certification of melting point RMs, were formulated.

The results of the conducted research were used to develop a set of melting temperature reference standards for high-purity organic substances produced in Russia to replace imports of foreign reference materials and ensure the uniformity and traceability of measurements in this area.

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COMPARATIVE CHARACTERISTICS OF SORPTION AND CONCENTRATION OF FOOD AZO DYES ON MAGNETITE NANOPARTICLES AND NANOFIBERS

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With the advent of nanoobjects and nanotechnologies for the sorption and concentration of analytes, including synthetic food dyes (SFDs), nanosized sorbents began to be widely used, the surface area of which and sorption capacity are significantly greater than those of traditional sorbents. Among nanosorbents, magnetic nanoparticles (MNPs), in particular magnetite, have become widespread. Its advantage is the ability to quickly separate the precipitate from the solution using a permanent magnet. The disadvantage of research in this area is the lack of comparison of the sorption properties of different types of nanosorbents, which does not allow choosing the most convenient method of extraction and concentration, combined with the stage of determining the dye in the object. In this regard, the goal of our work was to compare the sorption and concentration of a number of anionic food azo dyes (tartrazine, sunset yellow, azorubine, charming red and ponceau 4R) on magnetite MNPs (Fe_3O_4) and a new type of nanofiber sorbents - polyamide 6, obtained by electrospinning. MNPs were modified with chitosan (CTS) and polyethylenimine (PEI), which imparted a cationic charge to the MNPs.

The optimal conditions and parameters of sorption and concentration were found, kinetic models were constructed, the patterns of processes were analyzed, and the possibility of sorption-spectrophotometric determination of azo dyes after desorption with MNPs and without desorption on nanofibers was shown. The possibility of regenerating sorbents and their reuse was assessed. Based on the data obtained, a method has been developed for the extraction-spectrophotometric determination of a mixture of azo dyes Ponceau 4R, azorubine and charming red in syrup and dietary supplements using the projections onto latent structures chemometric algorithm. The advantage of the developed method is the rapidity of sample preparation, a 75-fold reduction in the limit of photometric detection of dyes on MNPs, and the possibility of determining azo dyes in a mixture without their separation.

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ACID MINERALIZATION OF BLACK SHALE SAMPLES FOR ANALYSIS BY ICP-MS/AES METHODS

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One of the most difficult tasks of analytical chemistry is the determination of ultra-low (ng/g) concentrations of chemical elements, including noble metals (NM), in geological samples with complex mineral composition, such as black shales. Rocks of this type is the main raw material source of the Russian gold mining industry. Obtaining correct results of the analysis by ICP-AES/MS methods significantly depends on the quality of sample preparation, which implies digestion of the samples and, if necessary, analytes concentration. However, there is still no reliable method for black shales complete digestion, especially for representative sample mass, which is usually at least 0.5 g.

The report presents the method of acid decomposition of black shale samples. The method includes two stages: solid-phase reaction with ammonium bifluoride (2 h, $t = 235^{\circ}\text{C}$) with following dissolution of fusion cake in acids (HCl , HNO_3 , HClO_4). Both stages carried out in the same system for acid decomposition at atmospheric pressure [1]. Comparison of the developed method with methods of acid digestion in open system [1] and in autoclaves [2], as well as with sodium peroxide fusion are given.

For the first time it has been shown that the developed method provides complete digestion of black shales. The decomposition procedure is quite productive and allows to digest of dozens of samples with mass up to 1 g for two days. The results of determination of the 62 elements by ICP-AES/MS methods in reference sample of SLg-1 black shales [3] are presented.

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ASSESSMENT OF TRACE AMOUNTS OF OSMIUM WHILE NOBLE METALS DETERMINATION BY ICP-MS METHOD

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The determination of osmium in comparison with other noble metals (NM) is the most difficult task in the analysis of geological samples. Difficulties are associated with both the mineralization of the samples and the subsequent treatment of the samples with acids at high temperature; as a result, osmium losses due to the formation of volatile OsO_4 compounds are possible. The current sample preparation methods for determining Os are quite complicate. Typically use autoclave digestion followed by diffusion concentration [1], mineralization of samples in steel autoclaves in Carius tubes [2], etc.

Our proposed method for estimating the osmium content is based on the mineralogical similarity of osmium and some NM. The method consists in carrying out the determination of a number of BM (except osmium) after a complete acidic digestion of the samples. At the next stage, NM (including osmium) is determined already in extracts based on aqua regia. For a number of NM, the degree of their extraction from the sample can be found. Further, the value of the degree of extraction is used to introduce the required correction for the concentration of osmium. The key issue is the choice of the reference element(s) for the correction. Osmium mineralogy is usually closely related to iridium, sometimes platinum and gold.

Thus, the following results were obtained for samples of sulfide copper-nickel ore (Table. 1), which corresponds to the estimated range of osmium content ($0.020 \div 0.099$ g/t).

The results of the determination of osmium with correction on various supporting elements

Supporting elements	Os, g/t	%, SD
Au, Ir, Pt	0,056	34
Au	0,051	17

Acid mineralization of the samples (200-300 mg) was carried out in an open system using solid-phase fluorination with ammonium bifluoride and sequential acid treatment (HNO_3 , HClO_4 , HCl). Extracts from the samples (500-1000 mg) were obtained by treating them with 20-40 ml of aqua regia at room temperature for an hour. The elemental composition of solutions after acid mineralization of ore and in “extracts” was determined by the ICP-MS method.

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POSSIBILITIES OF THERMAL LENS SPECTROMETRY IN ANALYSIS OF PHYSICAL AND CHEMICAL PROPERTIES OF AQUEOUS DISPERSIONS OF LATEX NANOPARTICLES

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The morphology of polymer micro- and nanoparticles, the complex dynamics of physicochemical transformations, and low concentrations that can be characteristic of nanoplastics in real systems require sensitive analytical methods that are also capable of measuring physicochemical parameters of aqueous dispersions. To solve such problems, thermo-optical spectroscopy can be used as a group of methods belonging to molecular absorption spectroscopy and, at the same time, to thermal spectroscopy. The dual nature of the measured data provides the information on both light absorption and the thermophysical properties of the objects under study. This ensures both the quantification of light-absorbing particles at levels corresponding to absorbances of up to 10^{-7} , and the measurement of characteristics responsible for the dynamics of establishing a thermal steady state in solution, with an error of less than 1%.

Thermal-lens spectrometry, the most common thermo-optical method, was used to assess the content of polystyrene particles with sizes of 65 and 80 nm in a broad range of concentrations (0.0001–0.1 % w/w). It is shown studying nanoplastics requires taking into account the influence of light scattering and light absorption parameters, but the sensitivity is mainly determined by the thermophysical parameters of the solution. The thermal diffusivity of solutions of polystyrene nanoparticles was assessed depending on the concentration. As the concentration increases from 0.0005 to 0.1% w/w, thermal diffusivity decreases by 4% compared to the pure solvent, while a sharp decrease in thermal diffusivity is observed at 0.001%. This may indicate a change in the mechanism of heat transfer in the solution due to a change in the rate of aggregation of polystyrene particles, which requires additional research.

The work was carried out with financial support from the Ministry of Science and Higher Education of the Russian Federation: AAAA-A21-121011590089-1.

VOLTAMMETRIC DETERMINATION OF CORTISOL AND VITAMIN C ON SCREEN PRINTED ELECTRODES MODIFIED BY GOLD PARTICLES

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Stress is one of the main factors that contribute to the development of pathologies in various organs and systems. Cortisol (hydrocortisone, HC) is a glucocorticoid steroid hormone that is involved in the body's response to stress and can be used as a biomarker for early detection of stress. Vitamin C (ascorbic acid) are of great importance in normalizing cortisol levels. Its antioxidant properties can help eliminate stress and anxiety. Vitamin C prevents an increase in the levels of cortisol, adrenaline and anti-inflammatory polypeptides

The aim of this research is to develop a voltammetric method for the determination of HC and vitamin C on screen-printed electrode (SPE) modified by gold particles.

HC is electrochemically inactive on bare carbon electrodes. Anodic peak of electrooxidation of vitamin C is observed at E 0.75 V in cyclic voltammetry in acidic medium on bare SPE. Gold particles electrodeposited on the surface of the SPE have electrocatalytic activity at the oxidation of organic compounds. This leads to an increase in current compared to the oxidation current of the modifier and a decrease in the overvoltage of oxidation of organic compounds. The peaks of electrooxidation of vitamin C and HC are observed at E 0.4 V and E 1.1 V, respectively.

The possibility of selective determination in the combined presence of HC and vitamin C on SPE modified by gold is considered. The peak potential separation of 0.7 was achieved, allowing the simultaneous detection of two compounds. The linear dynamic ranges of 5 – 500 μM for HC and 0.5 – 500 μM for vitamin C.

The developed method for the selective voltammetric determination of HC and vitamin C was tested in the analysis of a model blood serum system.

The work was supported by the Kazan Federal University Strategic Academic Leadership Program (PRIORITY-2030).

THE STRUCTURE OF NEGATIVE IONS OF SPIROPYRANS AT RESONANT ELECTRON CAPTURE

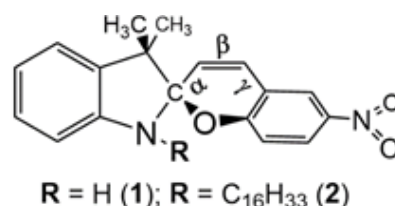
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Spiropyrans are known for their photochromism, which consists in the ability of molecules to photoinduce (i.e., when “capturing” a photon) the transformation of a cyclic structure into an open merocyanine form, accompanied by a break in the C–O bond and a change in absorption spectra and other optical characteristics. In this work, the processes occurring with the molecules of spiropyrans (**1**) and (**2**) during the resonant capture of low-energy electrons (REC) have been studied by mass spectrometry. It has been established that negative ions (NI) are formed in several regions of electron energy (E_e) within the 0–11 eV range, among which molecular (M^-) and fragmented NI [$M-CH_3$]⁻ demonstrate the maximum yield. For **1**, an intense peak of NI [$M-OH$]⁻ was also detected, which is probably due to the H-shift of the R = H atom to the nearest O atom available for this compound. Long-lived M^- with lifetimes of $\tau_a(M^-)$ reaching several hundred ms are observed beginning from ~0 eV to abnormally high E_e 1 eV for **1** and ~4 eV for **2**, indicating a large adiabatic electron affinity (EA_a) for these molecules.

The estimates using $\tau_a(M^-)$ with the aid of RRKM statistical model¹ for **1** showed a value of 1.23 eV, which was confirmed by the results of quantum chemical calculations PBE/3 ζ performed for cyclic forms M and M^- ($EA_a^{calc} = 1.15$ eV). At the same time, calculations of all 8 possible conformations of merocyanine predicted EA_a of at least 2 eV. Nevertheless, in molecular NIs of spiropyran **1**, structural conversions similar to phototransformation of neutral molecules do occur under the REC conditions, which follows from the analysis of dissociative processes in M^- using the mentioned RRKM model.



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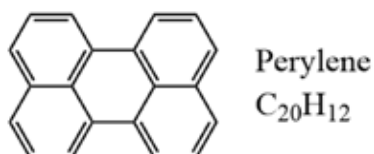
LONG-LIVED NEGATIVE MOLECULAR IONS OF PERYLENE

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Perylene is the representative of polycyclic aromatic hydrocarbons, which have wide practical applications due to its specific physical, optical and electronic properties. In particular, perylene and its derivatives are used in various molecular electronics devices¹.



Long-lived (characterized by the lifetimes with respect to autoneutralization $\tau_a > 10 \mu\text{s}$) negative molecular ions (M^-) of perylene were recorded by means of resonant electron capture mass spectrometry in five resonant states (RS) with maxima observed at electron energies of 0.05, 0.4, 0.7, 1.4 and 2.5 eV. To assign the registered RSs, using TD B3LYP/6-311G approximation, the electronic spectrum of the ground state anion ($^{GS}M^-$) was calculated, based on which a number of features of the RSs were obtained, including electronic configurations, energies and symmetries of the spectroscopic states (Figure 1).

	B_{1g}	B_{3u}	B_{3u}	A_u	B_{1g}
70 a_u	—	—	—	—	↑
69 b_{3u}	—	—	↑	—	—
68 b_{3u}	—	↑	—	—	—
67 b_{1g}	↑	—	—	↑↓	↑
66 a_u	↑↓	↑↓	↑↓	↑↓	↑↓
65 b_{2g}	↑↓	↑↓	↑↓	↑↓	↑↓
64 b_{3u}	↑↓	↑↓	↑↓	↑↓	↑↓
PC $^{GS}M^-$					
$E_{\text{расч}}(\text{эВ})$	0.0	0.5	0.8	1.6	2.5
$E_{\text{эксп}}(\text{эВ})$	0.05	0.4	0.7	1.4	2.5

Figure 1. Calculated electronic spectrum of perylene ground state $^{GS}M^-$.

A comparison of the experimental and calculated RSs revealed that the ions formed in the RS at 2.5 eV has the same symmetry (B_{1g}) as ground-state $^{GS}M^-$ ions, which has a prominent long lifetime due to the significant positive molecular electron affinity. It is hypothesized that the $^{GS}M^-$ ground state contributes to the τ_a value of ions formed at 2.5 eV, providing them with a measurable $\tau_a > 10^{-5}$ s. The question of the possible effect of radiation cooling of M^- on the magnitude of τ_a is also considered.

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A COMPARISON OF SAMPLING METHODS USED IN THE ANALYSIS OF E-LIQUIDS BY GC/MS

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Currently, there are different methods of sample preparation for liquids for electronic nicotine delivery systems (e-liquids), therefore their comparison is an important goal. Glycerin, propylene glycol, flavourings and nicotine in the form of base or salt are the major components of e-liquids. The aim of this work is to compare various methods of sample preparation, followed by processing the results and evaluating them in terms of the relative extraction efficiency of the mixture components.

The analysis was carried out on a Perkin Elmer Clarus 600 gas chromatograph with a Perkin Elmer Clarus 600T mass spectrometer, Elite-5MS capillary column (30 m, 0.25 mm, 0.25 μ m). E-liquids from four different manufacturers were used.

A series of derivatization experiments, liquid-liquid extraction experiments with changing the pH of the aqueous phase were organized. Solvents such as methanol, pyridine, ethyl acetate, hexane were carried out to evaluate different sample preparation options. Analytes have been classified according to their behavior during sampling and relative extraction efficiency was defined.

Table 1. Relative extraction efficiency of sample preparation methods, %

	Methanol dilution	Pyridine dilution, silylation	Ethyl acetate LLE	Hexane LLE, pH > 7	Hexane LLE, pH < 7, silylation
Nicotine	38	87	0	Up to 100	0
Terpenes	30	30	Up to 100	15	2
Esters	4	74	Up to 100	5	1
Carboxylic acids	0	0	0	0	Up to 100

A relevant method can be selected according to Table 1 depending on the component to be analyzed.

SURFACE MODIFICATION OF CARBON NANOMATERIALS FOR SELECTIVE SORPTION OF NOBLE METALS FOR DETERMINATION BY ICP-MS

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The simultaneous determination of Au, Pt, Pd, Rh, Ir, Ru in rocks is a relevant analytical task. The most common detection method is inductively coupled plasma mass spectrometry (ICP-MS), which have 10^{-9} to 10^{-12} g/ml detection limit range. However, after sample decomposition solutions must be diluted to reduce the matrix effect. Noble metal (NM) concentrations are often found to be near or below detection limits. In addition, the determination of noble metals is accompanied by numerous spectral interferences. All these factors shows the need for preconcentration stage to determine NM in most cases.

Carbon nanomaterials (CNMs) are characterized by a large specific surface area and a highly porous structure, which allows them to be considered as base for their surface modifications and for the creation of new highly efficient and selective sorbents.

Two methods of functionalization carbon nanomaterials are considered: impregnation with N, S-containing heterocyclic reagents and chemical (covalent) addition of selective groups. Impregnation in organic solvents is a simple and common method of inclusion reagents to solid porous materials. Such sorbents are characterized by simplicity of preparation and large sorption capacity. Chemical treatment ensures the sorbent's stability and protects the reagent from leaching due to the formation of a strong covalent bond between the reagent molecules and the CNM surface. The method of diazotization of amines in acetic acid with NaNO_2 is used for the chemical modification of reagents.¹

The selectivity to noble metals towards to a wide range of elements was measured for the resulting sorbents; for this purpose, standard solutions for calibration ICP-MS-68A-100 Solution A and C were used. The sorption capacity of the sorbents and the conditions for the group extraction of noble metals from hydrochloric acid solutions were determined.

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COLORIMETRIC DETERMINATION OF POLYDADMAC IN TREATED WASTEWATER

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For wastewater treatment, coagulation and flocculation processes are often used, which consist in combining suspended impurities into larger particles and their precipitation under the action of special reagents – coagulants and flocculants. In practice, the cationic polyelectrolyte polydiallyldimethylammonium chloride (polyDADMAC), which can be used both as a flocculant and as a coagulant, is widely used for these purposes¹. PolyDADMAC is not toxic, but its presence in water disinfected with organochlorine compounds can lead to the formation of carcinogenic nitrosamines². Therefore, the determination of the polyDADMAC content in water is an important analytical task.

A lot of techniques for the determination of polyDADMAC in water are characterized by high limits of detection, which do not allow for the determination of this substance at the maximum permissible concentration (MPC) level. In addition, such techniques are characterized by tedious sample preparation using expensive equipment. In this regard, the urgent task is to develop a simple and rapid technique for the determination of trace concentrations of polyDADMAC in treated wastewater.

In this work, a colorimetric test system for the determination of polyDADMAC in treated wastewater is proposed, based on its interaction with an anionic dye with the formation of an uncolored ionic associate and subsequent extraction of unreacted dye into a deep eutectic solvent. The concentration of polyDADMAC is determined by the color intensity of the extract visually or using a smartphone camera. The technique allows the determination of polyDADMAC concentration in non-laboratory conditions at the MPC level. The developed technique is illustrated on the example of polyDADMAC determination in treated wastewater.

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DEEP EUTECTIC SOLVENTS AS A MEDIUM FOR DERIVATIZATION OF ANTIBACTERIAL DRUGS IN THE ANALYSIS OF BIOLOGICAL FLUIDS

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In modern analytical chemistry, special attention is paid to the development of new methods for the effective, selective, and sensitive determination of drugs in biological fluid samples. The analysis of biological fluids is a complex analytical task due to the complexity of biological fluids matrix. New opportunities for the development of effective methods for the analysis of biological fluids are opened by liquid microextraction using selective and “green” extraction systems, which allow a high rate of interphase equilibration, the possibility of miniaturization, automation, and environmental safety of sample preparation.

Currently, deep eutectic solvents (DES) are widely used in analytical chemistry as effective extractants. At the same time, DES acting as effective extractants can also serve as a medium for the derivatization of target analytes.

In this work, an approach based on the direct participation of DESs components as reagents in the derivatization of polar analytes that are poorly extracted in traditional non-polar organic solvents has been implemented for the first time. The possibility of forming colored non-polar derivatives (Schiff bases) was demonstrated by the interaction of DESs components (aromatic aldehydes) and polar target analytes. It was confirmed that aromatic aldehyde, acting as a precursor to DES, promotes the extraction of polar analytes from the aqueous phase due to the formation of hydrophobic Schiff bases. The developed method formed the basis of the scheme for the determination of sulfonamides and isoniazid in blood plasma and urine samples.

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DEVELOPMENT OF A SAMPLE PREPARATION TECHNIQUE TO RESEACH DISTRIBUTION OF TRACE ELEMENTS IN AUTOPSY MATERIAL OF RED BONE MARROW BY X-RAY FLUORESCENCE METHOD USING SYNCHROTRON RADIATION IN PATIENTS WITH HEMOBLASTOSES

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Sample preparation of autopsy and biopsy samples for X-ray fluorescence analysis with synchrotron radiation (SRXRF) does not have clear protocols for each type of biological tissue. The paper describes a method for sample preparation of red bone marrow (BM) from patients with hemablostoses, which we use for SRXRF. The main advantage of this sample preparation method is non-destructiveness - high temperatures and acids are not used, which reduces the risk of loss of elements or their introduction into the sample. The resulting BM material was placed in the form of 2 μ L drops on a fluoroplastic film and dried at room temperature to constant weight. The dry material was ground in a mortar and tablets weighing 14-15 mg and 8 mm in diameter were formed from it. Tablets with the sample were placed between two Mylar films and exposed to the SR beam.

The prospective study included 17 people (women - 8 (47%), men - 9 (53%)), of which 7 (41%) patients with Hodgkin lymphoma (HL) with laboratory criteria for leukemia and 10 (59%) patients with acute leukemia (L). The median age of patients was 45 (27; 56) years. Patients were examined before the start of treatment and after completion of the course of polychemotherapy (PCT). The analysis of trace elements was carried out by the SRXRF method on the basis of the Siberian Center for Synchrotron and Terahertz Radiation at the Institute of Nuclear Physics SB RAS on the VEPP-3 storage ring. The following chemical elements were determined in the BM: K, Ca, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Mo, La, Th. Statistical analysis of the obtained results was carried out using the Statistica 12 program (Dell, USA). The resulting deviations of the average are within the confidence range of values for the international standard sample A-13 (whole blood).

The data obtained will likely make it possible to identify early predictors of the refractory-recurrent course of hemoblastosis. Further work will be aimed at comparing the results with the control group, identifying prognostic relationships between the identified disorders of metal ligand homeostasis and clinical and therapeutic features in patients with oncohematological diseases.

“COLORISTIC” - A NEW WAY OF CLASSIFICATION FOR ARTIFICIAL SENSE OF SMELL

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The main purpose of methods for processing multidimensional data related to an object is to reduce the analytical data space while preserving distinctive features that allow you to find similarities or differences in samples. From the point of view of bionics, “electronic noses”, as simulation systems, must register as many compounds as possible in the mixture or respond selectively to changes in the concentration of those components that determine its identity.

A new method of multivariate data processing “Coloristic” is proposed to compare a set of different data obtained from piezoquartz micro-weighing of the equilibrium gas phase over any sample. The essence of the new method of chemometrics lies in the fact that within a sample of compared samples and for a certain set of sensors, the calculated selectivity parameters A_{ij} , $a(i/j)$, the normalized quantitative characteristics of the mixture of volatile compounds recorded by the sensor set, depending on their numerical values, are assigned a certain color with standard coordinates in the RGB or other systems (e.g., HEX). For this purpose, an approach is proposed to substantiate the boundaries of parameter values for each color code, which depend on the metrological characteristics of the parameters of paired sensitivity, determined by the nature of the modifier on the sensors, and the value of the parameter. Ways to improve the reliability and recognition properties of such data sets, to unify boundaries for different arrays are proposed. Next, the color labels of the selected set of parameters, which are as different as possible for identical samples, are summed up in any open visual tinting resource and the total color for the entire dataset is obtained.

Color mixer mixes an unlimited number of colors and shades, and the application of the coordinates of the mixed (total) color on the field allows you to unambiguously determine the proximity or distance of the total mark for a particular sample based on the results of detection by a set of sensors. The more parameters included in the sample, the more accurate the differentiation of samples and the more reliable their classification. Ready-made coloristics blocks are also well integrated into personal programs for “electronic noses”.

SEPARATION AND CONCENTRATION OF NOBLE METAL IONS BY SORBENTS BASED ON QUATERNIZED POLYVINYLMIDAZOLES

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One of the most important requirements for sorption materials is their selectivity, which in turn can depend not only on the nature of the polymer matrix, but also on the nature of the crosslinking agent and the degree of crosslinking. In the process of cross-linking polyvinylimidazole by quaternization, the donor ability of imidazole groups, which provide the possibility of complexation with noble metal ions, decreases, however, a network of anion exchange centers appears, which makes it possible to change the sorption mechanism and extract metal ions due to ion exchange.

The objects of the study were sorbents based on polyvinylimidazole (PVI), cross-linked with 1,4-bis(bromomethyl) benzene and epichlorohydrin, with degrees of quaternization of 92 and 71%, respectively. The sorbents were obtained at the Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences, under the guidance of Ph.D. A.V. Pestova.

The selective properties of sorbents with respect to silver (I), gold (III), palladium (II) and platinum (IV) ions were studied from hydrochloric acid solutions of various compositions using the limited volume method.

It has been established that the sorbents under study selectively extract noble metal ions from hydrochloric acid solutions of various compositions. It is worth noting that during the sorption of gold (III) and palladium (II) in the presence of a number of transition metals, sorbents also extract cadmium (II) and zinc (II) ions to a certain extent in strongly acidic media, due to the stability of their chloride complexes. It has been shown that PVI cross-linked with epichlorohydrin also extracts copper (II) ions at pH > 2.0, which may be due to a low degree of quaternization and, as a consequence, extraction of copper (II) ions due to complexation. It was found that the nature of the cross-linking agent has little effect on the selectivity of PVI. The tested sorbents were tested for the extraction of noble ions from standard ore samples.

RESEARCH OF THE COMPOSITION OF ROLLED ELECTRICAL STEEL BY ATOMIC EMISSION SPECTROMETRY OF GLOW DISCHARGE

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Electrical steel is widely used in industry for the manufacture of transformers and electric motors. High magnetic conductivity is provided by the chemical composition in combination with the processing of rolled steel, which affects the distribution of elements in the finished product. The study and control of the distribution of the concentration of elements over the depth of rolled ETS is most promising to be carried out by the method of atomic emission spectrometry of a glow discharge (GDOES).

The work used a GD Profiler-2 spectrometer (HORIBA Jobin Yvon, France) equipped with a radio frequency generator that allows analyzing low-heat conducting layers. The uniformity of spraying of ETS samples was established at a power of 850 Pa, a pressure of 50 W, and a pulse frequency of 400 Hz of the generator.

Calibration graphs for determining concentrations of Al, Si, C, Nb, Cu, Ni, Co, P, S, Ti, Mo, Mn, V, Cr, W, B were constructed using 47 monolithic reference materials of siliceous, carbonaceous and alloy steels, both domestic and foreign production facilities that are close in chemical composition to the analyzed samples. It was found that it is possible to increase the accuracy of the analysis of steel samples by taking into account the interelement influence of Pb, Fe, H, Ca, voltage correction for Fe or using a polynomial calibration dependence for Si. It is shown that the value of the relative error in determining the concentration of elements, for example, sulfur, Si, P, is reduced by 2-6 times.

The absence of a systematic error in the determination of elements in electrical steel samples was confirmed by evaluating the results of the quantitative determination of elements in standard samples according to the Student's criterion and comparing them with the results of the analysis of rolled ETS by atomic emission spectrometry with spark atomization type (ГОСТ Р 54153).

The possibility of conducting a semi-quantitative analysis of the distribution of the concentration of elements in the zone of internal oxidation (including an assessment of the content of silicon oxide IV), the ground layer and the electrical insulation coating of rolled products is investigated. Satisfactory consistency of the results of GDOES and scanning electron microscopy was obtained.

METABOLOMIC PROFILES OF MUSK *MOSCHUS MOSCHIFERUS* AS A PLATFORM FOR ASSESSING THE THERAPEUTIC AND ADAPTOGENIC POTENTIAL OF MUSK MEDICINE PREPARATIONS

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Musk preparations have over 2000-year history of application in traditional Chinese medicine. Several musk preparations are registered in the modern Chinese Pharmacopoeia¹. In Russia, this direction is at the stage of development.

Chemical analysis usually determines lipid components, in particular muscone and androstane group steroids². However, musk also contains other biologically active compounds (BACs) with therapeutic and adaptogenic potential.

Previously, we analyzed the lipid-steroid composition of the Siberian musk deer³ (*Moschus moschiferus*), showing significant differences in the composition compared to species living in China (*Moschus berezovskii*).

In this work, the metabolomic profiles of (1) Siberian musk deer musk (*Moschus moschiferus*), (2) hydrolysis samples of musk and (3) the medicine «Muskuliv» were obtained by HPLC-MS and GC-MS using sialylation. It has been shown that hydrolysis leads to enrichment of metabolomic profiles: the number and quantity of organic acids and nitrogen-containing compounds, including amino acids and dipeptides, increases. Enrichment by these compounds will allow to develop the animal medicine preparations with higher therapeutic and adaptogenic potential.

Also, the applied methods of metabolomic discovery based on HPLC-MS and GC-MS showed effectiveness for searching new BAC and for comparison of samples with hydrophilic and hydrophobic nature of BAC and can be a convenient analytical platform for subsequent study and monitoring of different animal medicine preparations having such a complex composition.

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PRECONCENTRATION OF PLATINUM AND PALLADIUM WITH MAGNETIC SORBENTS IN ANALYSIS OF NATURAL AND TECHNOLOGICAL OBJECT

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The difficulty of instrumental determination of platinum and palladium is concluded in the diversity of their species and extremely low contents (10^{-7} – 10^{-4} %), sample heterogeneity and the influence of matrix components. Decreasing the limit of detection and elimination of matrix effects are achieved at the sample preparation stage.

Sorption by magnetic nanoparticles eliminates filtration and centrifugation steps, reducing sample preparation time.¹ Automation of the process allows to flow large volumes of analyzed solutions and reuse sorbents. The study proposes original flow-through sorption cells of various geometries, which can be placed in the field of action of constant or variable magnetic fields of different configurations.

In the present study, methods of sorption preconcentration under static and dynamic conditions were proposed for the determination of platinum metals in samples of ore, road dust and spent catalysts. Magnetite-based nanoparticles in a polyethylene glycol shell synthesized at the interface of a two-phase polymer-salt system were used as sorbents.² To ensure the necessary selectivity and stability in solutions, the surface of nanoparticles was functionalized with phosphonium, pyridinium, imidazolium ionic liquids and deep eutectic solvents based on quaternary ammonium salts and carboxylic acids, organophosphorus substances and monohydroxy alcohols, terpenoids, including those of natural origin. Optimal conditions were established and methods of preconcentration of ultralow amounts of platinum and palladium by the proposed sorbents were developed. The found contents within confidence intervals agree with the certified values.

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COMPARATIVE EVALUATION OF VARIOUS APPROACHES FOR SUGARS DETERMINATION USING CAPILLARY ELECTROPHORESIS

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Carbohydrates are significant components of metabolism, which makes their determination in biological matrices a relevant problem. Capillary electrophoresis (CE) allows solving it along with reaching high efficiency and selectivity of separation, as well as reducing the limits of detection (LOD) with intracapillary concentration. Indirect detection was used for the determination of sugars with the following background electrolyte (BGE): 2.5 mM acridoneacetic acid, 75 mM KOH, 0.5 mM cetyltrimethylammonium bromide and 5% (vol.) methanol. Other approaches tested were reductive amination with ethyl-p-aminobenzoate (ABEE) and condensation with 1-phenyl-3-methyl-5-pyrazolone (PMP). Indirect detection enables the determination of both reducing and non-reducing mono- and disaccharides. However, the achieved LOD (4 – 10 µg/ml) was higher than the ones reachable after the derivatization of the analytes. For reductive amination, separation was performed in zone (CZE) and micellar (MEKC) modes with the introduction of sodium dodecyl sulfate as a micelle-forming agent. In the latter case, higher efficiency was achieved, and the LOD were reduced by 2 times compared to the CZE. Intracapillary concentration under sweeping conditions helped reduce the LOD to 0.12 – 0.27 µg/ml. For condensation of sugars with PMP, separation was performed in CZE mode, and the use of large-volume sample stacking enabled the lowering of LOD to 0.10 – 0.16 µg/ml. Both derivatization methods and the indirect detection mode for determining sugars were adapted to the analysis of baby food.

The analytical capabilities of intracapillary derivatization of sugars with the introduction of a reagent, BGE, and the analyzed sample into a fused silica capillary with optimization of this mode were tested. During derivatization with PMP, the electrophoretic mobility of the derivatives was conditioned by the pH value of the reaction medium; and for reductive amination, surfactant micelles were introduced into the solution.

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THE NEED TO TAKE INTO ACCOUNT METAL CARBOXYLATES WHEN ANALYZING FATTY ACIDS IN CULTURAL HERITAGE SITES

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In many cases, the analysis of cultural heritage sites uses common approaches for the determination of fatty acids, which are applicable for simple matrices. When it comes to complex inorganic matrices, analysts may encounter the possible influence of carboxylic acid salts formed over time. This may affect the fatty acid profile. Additionally, a feature of the cultural heritage objects provided for analysis is an extremely wide weight range (from mg to tens of g). Also, the value of the object itself indirectly affects the analysis, which may prevent the selection of the necessary sample.

In this regard, the effect of the inorganic matrix on the formation of metal carboxylates was considered in this work. Carbon black, aluminum silicate and zinc whitewash were selected as inorganic matrices. The tasks to achieve this goal were a) analysis of corn and linseed oils before aging; b) creation of model inorganic matrices with different ratios of introduced oils; c) aging of samples at specified humidity and temperature parameters, including oil without a matrix; d) analysis of samples after 5 months. The ratio of the oil to the inorganic matrix is 1:1 and 1:50. After the analytes were extracted by the Folch method with further methylation. The analysis was performed on GC-MS Perkin Elmer Clarus 600T.

According to the results of the work, the formation of carboxylates is noted. A noticeable effect can be traced in the case of the predominance of the inorganic matrix over the oil. This can be solved by using steps in the sample preparation technique that allow the conversion of salts into acids.

The work is supported by RSF grant 22-18-00593.

DETERMINATION OF THE CHEMICAL COMPOSITION OF CRYSTALLINE SILICON BY SPECTRAL METHODS. ADVANTAGES OF ALTERNATIVE ANALYSIS METHODS. POSSIBILITIES FOR DEFINING ADDITIONAL ELEMENTS

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Crystalline silicon is intended for the manufacture of silicon-containing alloys, organosilicon products, semiconductor silicon, as well as for special purposes. The grades and chemical composition of silicon must correspond to those indicated in the table 1.

Silicon grade	Code	Mass fraction, %				
		Silicon not less than	Impurities, no more than			
			Iron	Aluminium	Calcium	Sum of impurities determined
Kp00	17 1131 001	99,0	0,4	0,3	0,4	1,0
Kp0	17 1131 002	98,8	0,5	0,4	0,4	1,2
Kp1	17 1131 003	98,0	0,7	0,7	0,6	2,0
Kp2	17 1131 004	97,0	1,0	1,2	0,8	3,0
Kp3	17 1131 005	96,0	1,5	1,5	1,5	4,0

Table 1. The grades and chemical composition of silicon

The mass fraction of silicon is set as the difference between 100% and the sum of the determined impurities as a percentage. The mass fraction of components in silicon (iron, aluminum, calcium and titanium) is determined according to GOST 19014.0 - GOST 19014.4.

Determining the chemical composition of crystalline silicon by titrimetric and photo colorimetric methods in accordance with the current ND is a very labor-intensive process that does not meet modern requirements for rapidity and cost-effectiveness of analysis. In this regard, the possibility of determining the content of standardized impurities (iron, aluminum, calcium and titanium) and others (phosphorus, chromium, manganese, carbon and sulfur) in crystalline silicon using alternative methods on atomic emission spectrometers with inductively coupled plasma was investigated. and X-ray fluorescence (AES ICP and XRF).

A semi-quantitative analysis was carried out using the X-ray fluorescence method and a quantitative analysis was carried out using the atomic emission spectral method with inductively coupled plasma.

Based on the test results, a set of statistical data is collected to certify methods using ICP and XRF nuclear power plants.

ION MOBILITY SPECTROMETRY OF ALCOHOLS

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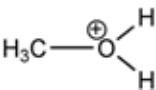
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Ion mobility spectrometry (IM) is a method of rapid detection of substances of various chemistry (chemical nature). However, the possibilities for application of this analysis method are usually limited by the lack of reliable data on ionization processes. Available literature almost exclusively describes general pathways of ion formation, which do not reflect the essence of processes taking place in instruments¹.

In this paper, we have decided to focus on saturated monohydric alcohol series due to the identical nature of the C-O, C-C, O-H of their bonds, which presumably means that ionization processes taking place in the spectrometer are also the same.

According to the literature ion mass is the main factor influencing ion mobility¹. Examination of the chosen objects has shown that the reduced ion mobility coefficient (RIMC) values were identical for alcohols with significantly different initial molecular weights (Table 1). This fact indicates that after a corona discharge ionization molecular ions of this compound series disintegrated into smaller fragments.

Table 1. Methanol vs. Butanol Comparison

Sample	Molecular mass, Da.	RIMC, cm ² /(V·s)	Proposed ion structure	RIMC value difference, %	Reaction enthalpy, kJ/mol
MeOH	32,04	2,1800		0,015	-58,45
BuOH	74,12	2,1803			-24,48

Enthalpy of proposed reactions is calculated using the quantum chemical package ORCA version 5.0.4. According to the literature, the use of the B3LYP method in combination with the def2-TZVPD basis set and the RIJCOSX approximation allows to calculate sufficiently accurate reaction enthalpies and optimal particle geometry^{2,3}.

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EXPANDING THE ANALYTICAL CAPABILITIES OF ENERGY DISPERSION X-RAY FLUORESCENCE ANALYSIS OF REFRACTORY METALS

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The production of electronic components in Russia, which was hopelessly lost in the 1970s, is again becoming an urgent task. So-called passive electronic components (niobium and tantalum capacitors and resistors) are in great demand in various industrial fields.

It has been studied that the specific characteristics of electrophysical quantities (nominal capacitance, voltage, loss tangent, leakage current, absorption coefficient, impedance, etc.) depend on the properties of dielectric materials¹.

Analytical control of raw materials for the manufacture of capacitors is an integral part of the technological processes of their production. The use of X-ray fluorescence analysis (XRF), which differs from atomic emission methods in its rapidity, is promising in the analytical control of refractory metals. The use of secondary targets and filters makes it possible to “monochromatize” the irradiation of the analyzed sample and significantly improve the signal-to-background ratio².

The purpose of this work is to expand the analytical capabilities of XRF as applied to the analysis of refractory metals. The work was performed on a Genius IF Xenometrix energy-dispersive X-ray fluorescence spectrometer using secondary targets and filters. The paper presents the results of a study on the development of a methodology for the analysis of industrially produced niobium and tantalum powders. The possibility of determining impurity elements (Cr, Fe, Ni, Ta, Nb, W, Mn) at a level of 0.001–0.1 wt.% has been shown.

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NEW METHODS OF SAMPLE PREPARATION FOR IDENTIFYING THE HYDROCARBON COMPOSITION OF CONTAMINATED SOILS

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The development of an integrated approach to determining the hydrocarbon composition of oil-contaminated soils is an urgent task, the solution of which allows us to select the optimal technology for soil restoration.

Current standard methods for determining the concentration of petroleum products in oil-contaminated soils do not allow us to estimate the proportion of volatile (light) hydrocarbons lost at the stage of sample preparation of a soil sample when it is dried at room temperature to an air-dry state. The loss of a significant amount of light hydrocarbons can lead to errors in the interpretation of the obtained analysis results. Especially when it comes to monitoring the condition of a contaminated area or choosing technologies for remediation of soils contaminated with light hydrocarbons. Also, when determining many individual substances, benzo(a)pyrene, for example, a lengthy procedure of sample preparation and identification is required. It should be noted that at the moment there are no methods for determining the composition of oil pollution in the real-life conditions.

In this work, the authors have developed and tested new methods for sample preparation of soils contaminated with various petroleum products allowing to determine the content of hydrocarbons (including light ones) in the analyzed sample in a short period of time as well as individual compounds (benzo(a)pyrene, toluene, chloroform, carbon tetrachloride, etc).

Particular attention is paid to the development of combined methods for analyzing oil-contaminated samples (combining the proposed method of sample preparation with various detectors) which makes it possible to determine the composition of hydrocarbon pollution in field conditions.

The work was carried out within the framework of a state assignment of Vernadsky Institute of Geochemistry and Analytical Chemistry, RAS.

DETERMINATION OF CAROTENOIDS IN FEED AND FEED ADDITIVES BY HPLC METHOD WITH SPECTROPHOTOMETRIC DETECTION

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A method has been developed for the simultaneous determination of the content of 12 carotenoids (astaxanthin, β -apo-8-carotenal, capsorubin, lutein, canthaxanthin, capsanthin, β -apo-8-carotenoic acid ethyl ester, zeaxanthin, citranaxanthin, α -cryptoxanthin, β -cryptoxanthin, β -carotene) in feed and feed additives using liquid chromatography with spectrophotometric detection in the visible range (450 and 475 nm).

Chromatographic separation were obtained using a YMC Carotenoid S-5 (250*4.6 mm) column with isocratic elution of the mobile phase acetonitrile - ethyl alcohol (75:25, v/v) at a flow rate of 0.8 cm³/min and a column thermostat temperature of 30 °C. Carotenoids were extracted from feed with acetonitrile- ethyl acetate (50/50,v/v), followed by purification of the extract on cartridges with a balanced hydrophilic-lipophilic reverse-phase sorbent, and from feed additives - with dichloromethane-ethyl alcohol (50/50,v/v).

The range of measurement of mass concentrations of carotenoids is 4-5000 mg/kg in feed and 0.05-20 % in feed additives.

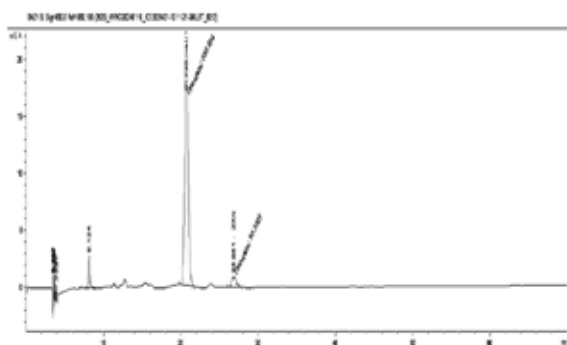


Figure 1. The chromatogram of the feed additive Biofon yellow 40 containing lutein and zeaxanthin

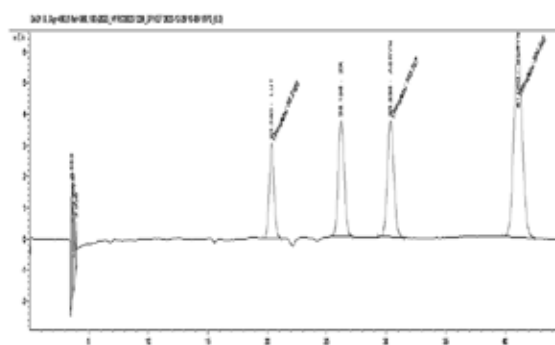


Figure 2. The chromatogram of laying hen feed containing lutein, zeaxanthin, α -cryptoxanthin, β -cryptoxanthin

COMPOSITES BASED ON COBALT TRIMESINATE AND GRAPHENE OXIDE AS EFFECTIVE SORBENTS FOR SOLID-PHASE EXTRACTION OF BRILLIANT GREEN

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Solid-phase extraction is an effective method for extracting, separating and concentrating dyes from the environment, with metal-organic frameworks (MOFs) being popular sorbents. However, they have a number of disadvantages, for example, low moisture resistance, which can be eliminated by introducing MOF composites with graphene oxide (GO) into the system.

Purpose of the work: synthesis of cobalt trimesinate/graphene oxide (Co-BTC/GO) composites with 5, 10 and 20% GO content, and their use as a sorbent for solid-phase extraction of a wide range of dyes. The composite was synthesized in situ in two stages: obtaining GO followed by the introduction of trimesic acid, sodium hydroxide and cobalt salt into the synthesis procedure. The resulting composites were studied by elemental analysis, SEM, TEM, EDS, and IR spectroscopy, the results of which are consistent with the data obtained previously. The ability of the synthesized composites to adsorb the brilliant green (BG) dye from aqueous solutions was studied. The dependence of solid-phase extraction on the weight of the sorbent, the weight fraction of GO, the initial concentration of BG, temperature and contact time was studied. The Co-BTC/GO-10% sample demonstrates the best sorption of BG compared to composites with 5% and 20% GO, which have similar sorption activity. The highest degree of adsorption is observed at the highest initial concentration of BG, while the sorbent is effective even with the introduction of its minimal quantities. The maximum time to establish equilibrium took no more than 180 min, while the bulk of the BG was sorbed during the first 60 min. As the temperature decreases from 308 to 278 K, the required contact time decreases. The values of thermodynamic parameters (ΔG^0 , ΔH^0 , ΔS^0) indicate a possible, spontaneous and exothermic process. Using chromatographic analysis, the presence of dye in the eluent was determined and the adsorption capacity was calculated to be 385.2 mg g⁻¹. The main metrological characteristics of solid-phase extraction have been determined.

This work was supported by the Russian Science Foundation (grant № 22-13-00260).

DETERMINATION OF TRYPTOPHAN ENANTIOMERS USING A VOLTAMPEROMETRIC SENSOR BASED ON MOLECULARLY IMPRINTED POLY(3,4-ETHYLENEDIOXYTHIOPHENE)

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The determination of biologically active substances is an actual issue in the field of analytical chemistry and medicine. One of the effective methods for this purpose is the use of voltammetric sensors based on molecularly imprinted polymers (MIPs)¹. For example, poly(3,4-ethylenedioxythiophene) (PEDOT) is a conductive polymer with unique electrochemical properties such as high specific capacitance, mechanical flexibility, chemical stability, and high conductivity. These features make it an excellent material for creating MIPs. The process of forming MIP sensors involves the interaction of a template molecule with monomers, subsequent polymerization, and removal of the template molecule, leaving unique voids specific to the target molecule.

In this work, voltammetric sensor based on molecularly imprinted poly(3,4-ethylenedioxythiophene) was developed for the determination of tryptophan enantiomers. We electrodeposited PEDOT film on a glassy carbon electrode using cyclic voltammetry in acetonitrile solution with the addition of LiClO₄, 3,4-ethylenedioxythiophene monomer and tryptophan as a template molecule. The morphology, electrochemical and analytical characteristics of the sensor were analyzed. The resulting sensor demonstrated high selectivity to tryptophan enantiomers ($I_D/I_L=1.5$) with a RSD of no more than 7%. In addition, the determination of tryptophan enantiomers in their mixture and in the presence of interfering components such as blood plasma and urine was carried out.

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The work was carried out with the financial support of the Russian Science Foundation, project 23-73-00119.

DETERMINATION OF THE DIETHYLCARBAMAZINE AND BITHIONOL RESIDUES IN LIVESTOCK PRODUCTS

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Because of the export development, including with the Asia-Pacific region countries, and the anthelmintic drugs residues in food health risk assessment, there is a need for the range of controlled compounds of this group enhanced. A method of diethylcarbamazine and bithionol determination in livestock products is needed. The purpose of this work is to develop a methodology for the residues of diethylcarbamazine and bithionol determination in meat and meat products, offal, fish, milk, and dairy products by high-performance liquid chromatography with mass spectrometric detection.

The liquid chromatograph 1290 (Agilent, USA) and the mass spectrometric detector 5500 QTRAP (Sciex, Canada) were used. The separation was carried out on an Agilent Pursuit 5 C18 150 × 2.0 mm column. The initial solutions of anthelmintics and internal standards were prepared by dissolving in methanol.

The optimal mass spectrometric detection conditions for bithionol and diethylcarbamazine were chosen (declustering potential, collision energy, collision exit cell potential). For bithionol in the negative mode MRM (ESI) the precursor ion was detected at m/z 352.8, daughter ions at 161.0 and 191.6; for diethylcarbamazine in the positive mode the precursor ion was detected at m/z 200.0, daughter ions at 100.0 and 127.

The acetonitrile (3 ml) was used in sample preparation of 1.0 g homogenized food samples. The extracts were purified with dispersive solid-phase extraction (150 mg of anhydrous $MgSO_4$ and 50 mg C18). The metrological characteristics of the developed method were validated. The range of determination for bithionol is 1–1000 μ g/kg. The relative standard deviation of the analysis results (%) does not exceed 16.8 and 14.2 (fish, meat, and milk) and 14.7 and 14.1 (offal, eggs, and intestinal raw materials). The developed methodology can be used in laboratory analysis of food as a part of their quality and safety monitoring.

LUMINESCENCE OF THE COMPLEX OF Eu(III) WITH AVOBENZONE

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Avobenzene is an organic substance that is used in cosmetic products as a sunscreen component. Recently, studies have been carried out on the photostability of avobenzene and its negative impact on marine ecosystems, in particular on corals¹. In this regard, the task of determining trace amounts of avobenzene in waters, sand, corals and other environmental objects is relevant. In this work, in order to develop a new method for the determination of avobenzene, the luminescence of its complexes with Eu(III) in micellar media was studied.

Like other aromatic β -diketones, avobenzene can form luminescent complexes with lanthanide(III) ions. The “antenna effect” was observed for the Eu³⁺ complex with avobenzene², but without connection with chemical analysis. We studied the possibility of determining avobenzene in aqueous solutions in the presence of various surfactants.

It has been established that weakly alkaline solutions containing avobenzene and Eu(III) luminesce intensely in the presence of cationic surfactants and nanoanion exchangers. In terms of the glow intensity of complexes with europium, avobenzene is not inferior to the most effective “antennas”, such as thenoyltrifluoroacetone and dibenzoylmethane. To determine avobenzene, an LED with a maximum emission of 370 nm was used. The peak of the most intense transition 5D₀ → 7F₂ corresponds to a wavelength of 613 nm. The best determination conditions are realized when using a nanoanion exchanger obtained from AB-17-8 resin³. The possibility of luminescent determination of avobenzene in water in the range of 10-10000 nM with a detection limit of 3 nM has been demonstrated.

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MEMBRANE IONSELECTIVE ELECTRODES FOR THE ANALYSIS OF ANTIBIOTICS

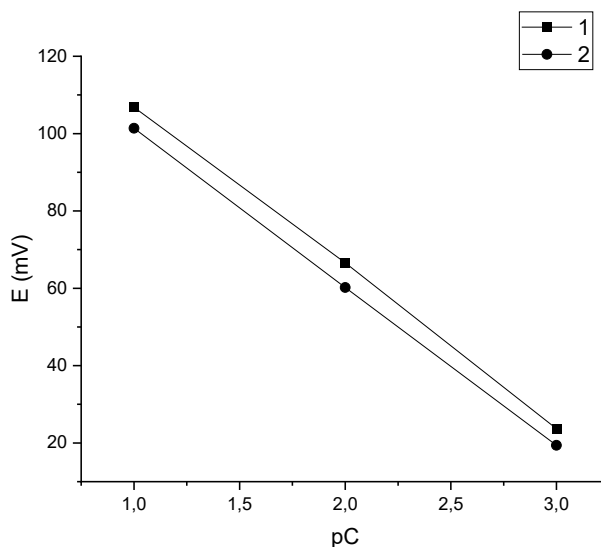
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Metal complexes of rare earth elements (REE) with cefazolin, which have low solubility in water, have been synthesized.¹ This property allows the use of such complexes as electroactive substances (EAS) in the creation of potentiometric sensors with plasticized polymer membranes based on organic ion exchangers with ampicillin, oxacillin, and cefazolin for the determination of antibiotics.²

Metal complexes were obtained by mixing aqueous solutions of neodymium or lutetium chloride with the sodium salt of cefazolin, at a molar ratio of 1:4. To create the membrane polyvinyl chloride (PVC), grade C-70, dioctyl phthalate (DOP) and cyclohexanone were used.

The composition of the created ion-selective membranes included (% by weight): 1% EAS, 1% DOP and 98% PVC.



1-cerium metal complex, 2-lutetium metal complex

In the range $pC=1-3$, experimental points are described by the equation of a straight line. The linear portion of the electrode function indicates the possibility of practical use of electrodes for the determination of concentration of cefazolin.

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DEVELOPMENT AND CERTIFICATION OF THE SET REFERENCE MATERIALS OF COMPOSITION ELECTRICAL ANISOTROPIC STEEL

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Electrical anisotropic steel (EAS) is widely used for the manufacture of electromagnets, electricity transformer, etc. The required electromagnetic indicators (coercive force, magnetic permeability, volume resistivity) depend on the chemical composition of the steel. Currently, there are no solid reference materials (RM) for assessing the correctness of determining the standardized elements and constructing calibration curves for spectral analysis, which significantly increases the cost and complicates the process of quality control of manufactured products.

A set of solid RMs EAS has been developed and certified, consisting of 10 samples, with element content (% mass) in the ranges: C (0,01-0,07), Si (2,5-4,5), Mn (0,07-0,22), S (0,003-0,013), P (0,005-0,04), Al (0,006-0,04), Al_{soul} (0,004-0,03), Cr (0,01-0,13), Ni (0,004-0,05), Cu (0,004-0,10), Ti (0,004-0,013), V (0,001-0,008), Nb (0,002-0,01), Mo (0,002-0,009), N (0,003-0,009), Sn (0,01-0,08), B (0,0003-0,025), Ca (0,0008-0,003). The concentration of components in RM samples was determined using an ARL iSpark 8860 spark atomic emission spectrometer (SAES) (Thermo Fisher Scientific, Switzerland) in accordance with the requirements of GOST R 54153-2010. The feasibility of establishing the homogeneity of the concentration distribution of C, Si, Mn, S, P, Al, Al_{soul}, Cr, Ni, Cu, Ti, V, Nb, Mo, N, Sn, B, Ca within one smelting is statistically substantiated RM samples. The use of the Student's test to process the results obtained by implementing the "accelerated aging method" (RMG 93) showed the absence of a trend in the concentration of C and sulfur, which are prone to diffusion, and the stability of the samples during a storage period of 10 years. Using the SAES method, the concentrations of RM elements were measured, and the limits of the absolute errors of the components were calculated. A set of monolithic EAS RMs is used in the laboratories of NLMK for spectral analysis when monitoring technological processes and can be useful for other enterprises producing EAS.

FLUOROMETRIC ANALYSIS FOR DETERMINING RESIDUAL CONCENTRATIONS OF OXYTETRACYCLINE IN FOOD PRODUCTS

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The large-scale use of tetracycline antibiotics in veterinary practice leads to their accumulation in food products of animal origin and environmental objects, which has a negative impact on human health. The enzyme-linked immunosorbent assay and high-performance liquid chromatography makes it possible to qualitatively detect residual amounts of antibiotics in products, but has a number of disadvantages, such as the complexity and duration of the analysis, and the use of expensive equipment.

It is known that tetracycline antibiotics have a chemical structure capable of luminescence when excited by various light sources. However, the use of fluorimetric analysis of tetracyclines is limited due to their low luminescence intensity.

In this work, we studied the possibility of fluorimetric determination of oxytetracycline after treatment with a hot solution of concentrated alkali, which causes the appearance of a new highly effective emission band ($\lambda_{\text{ex}} = 339 \text{ nm}$, $\lambda_{\text{em}} = 432 \text{ nm}$) and an increase in the intensity of the analytical signal by more than 3 times. The dependence of luminescence intensity on the time of contact of the reagent with the antibiotic was studied under optimal parameters for collecting spectra; it was found that the maximum intensity and stability of emission is observed after 15 minutes of interaction.

Calibration dependences of luminescence intensity on the concentration of oxytetracycline were obtained in a wide concentration range (10^{-7} - 10^{-9} mol/L), which corresponds to the MPC level in meat products. The calibration dependence is linear, $R^2 \geq 0.97$.

The developed method was tested by the "spike/recovery analysis" and tested on a meat extract, the sample preparation of which was carried out using chloroacetic acid as a protein precipitant.

This work was financially supported by the University startup studio within the framework of the federal project "University Technological Entrepreneurship Platform".

INVESTIGATION OF THE IONIZATION PROCESSES OF ANILINE NITRO DERIVATIVES

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The ion mobility spectrometry (IMS) method is based on the ionization of a substance at atmospheric pressure. Reagent ions are formed in the discharge chamber, the concentration of which significantly exceeds the concentration of the substances being determined.¹

The ion mobility spectra were obtained at the Kerber-T IDD at atmospheric pressure. Purified ambient air was used as a drifting gas. A program in the Python programming language version 3.3 was developed for mathematical data processing. During the study, it was found that 4-nitroaniline is detected only in positive ionization.

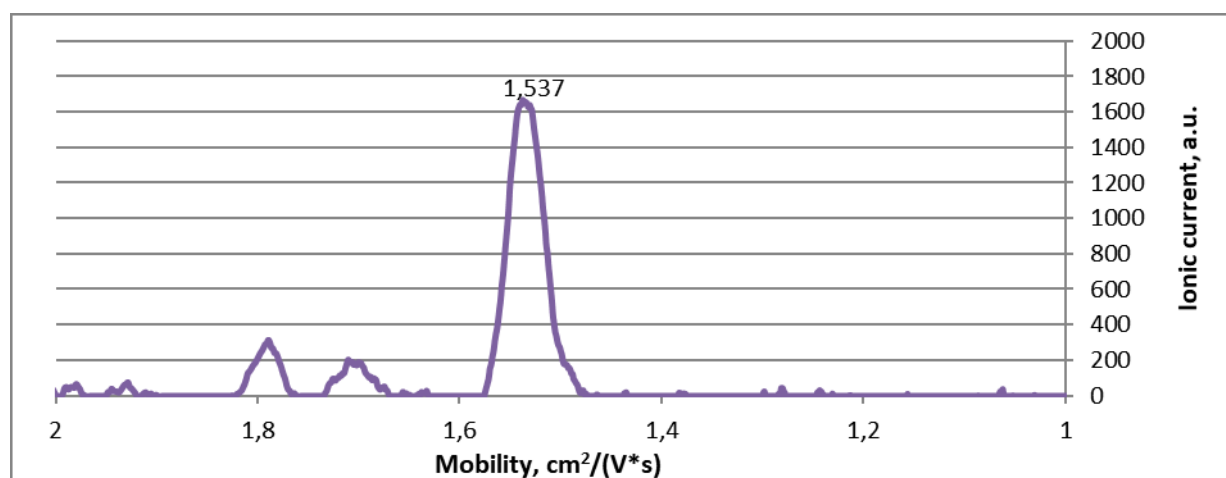


Figure 1. Ionic mobility of 4-nitroaniline in positive ionization

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ORDERED FILMS OF GOLD NANOPARTICLES FOR APPLICATION IN ENHANCED RAMAN SPECTROSCOPY.

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Raman spectroscopy is a method for identifying molecules. Since 1974¹, many studies have been carried out on Raman signal amplification using gold nanoparticles (NPs) due to their ability for localized surface plasmon resonance.

In this work, the problem of enhancing the signal intensity of Raman spectroscopy is solved by a simple approach to the self-assembly of gold NPs without functionalizing the NPs or using covalent linkers. In this case, the synthesis of NP sols was carried out using the classical methods of Frens² and Park³.

Colloidal solutions of nanoparticles were characterized using UV–vis spectroscopy and dynamic light scattering. Both methods showed that the NPs had an average diameter from 14 to 99 nm, while being stable, as the zeta potential was in the range of -25–30 mV.

Self-assembly of gold NPs at the interface was carried out according to the method⁴, then the film was transferred using the “aquaprint” method onto solid substrates (silicon, ITO, etc.), which were then used to amplify a weak Raman scattering signal. Using atomic force and scanning electron microscopy, the morphology of the substrates was studied.

The enhancing properties of the substrates were studied using the dye rhodamine 6G, the addition of which leads to the appearance of characteristic peaks. The calculated enhanced factor (EF) for the lines 1508 cm⁻¹ and 1360 cm⁻¹ ranged from $\sim 1.4 \cdot 10^3$ for nanoparticles with a diameter of 15 nm and $\sim 1.2 \cdot 10^4$ for larger nanoparticles (75 nm).

Substrates with the best EF, coated with gold NPs with a diameter of 75 nm, were selected for the detection of the poly-phenol – chlorogenic acid. They made it possible to determine chlorogenic acid in the concentration range from $1 \cdot 10^{-5}$ M to $3.5 \cdot 10^{-4}$ M.

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The work was carried out under a grant from the Russian Science Foundation (No. 22-73-00206).

ASSESSMENT OF THE POSSIBILITY OF USING STANDARD REFERENCE SAMPLES OF LITHIUM-BORATE ALLOYS IN X-RAY FLUORESCENCE ANALYSIS OF NATURAL AND TECHNICAL OBJECTS

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The great advantage of X-ray fluorescence spectrometry (XRF) is the ability to obtain accurate and reproducible results when analyzing solid monolithic and pressed materials with relatively simple and quick sample preparation.¹

This is achieved by using specialized standard samples and choosing a sample preparation method, that ensures homogeneous distribution of the components of the standard and analyzed samples, small particle size and low surface roughness.

The contribution of these factors to the accuracy of analysis can be reduced and the versatility of the reference samples used can be increased by using the method of high-temperature fusion of samples in flux (HTF). This method ensures a homogeneous distribution of components throughout the flux and makes it possible to significantly reduce the influence of matrix effects on the recorded analytical signal.²

The purpose of this work is to develop a draft methodology for determining 31 elements of the Periodic Table of Elements using the XRF analysis for natural and technical objects that are prepared by the HTF method. To carry out this work, a set of 49 calibration samples was created with the selection of the necessary fusion conditions, parameters for excitation and recording of the analytical signal were selected, considering the background, and calibration curves were constructed for each element determined.

The precision, accuracy, and reproducibility factors of the quantitative chemical analysis were computed. For this purpose, 3 sets of 10 samples of the same composition were created.

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DEVELOPMENT OF A PNEUMATIC FLOW BLURRING NEBULIZER FOR ANALYTICAL SPECTROMETRY

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The type of nebulizer used in ICP-OES, ICP-MS, MIP-OES and flame spectrophotometry significantly affects the basic plasma parameters and, consequently, analytical characteristics such as accuracy, sensitivity and reproducibility.

To solve research problems and import substitution problems of foreign nebulizer, a serial production of pneumatic nebulizer with “flow blurring” technology has been developed and established¹. The appearance of the developed nebulizer is shown in Figure 1 (b). The article compares the analytical characteristics of the created pneumatic nebulizer with the Burgener MiraMist, OneNeb2, Glass Expansion and Mainhard A-type and C-Type nebulizer obtained on the Grand-ICP spectrometer².

The highest plasma temperature and electron concentration were obtained using the created nebulizer and OneNeb2. The MiraMist showed the lowest efficiency of aerosol transportation to plasma. Comparable detection limits, reproducibility and accuracy with the OneNeb2 nebulizer have been obtained and are several times higher than Glass Expansion and MiraMist.

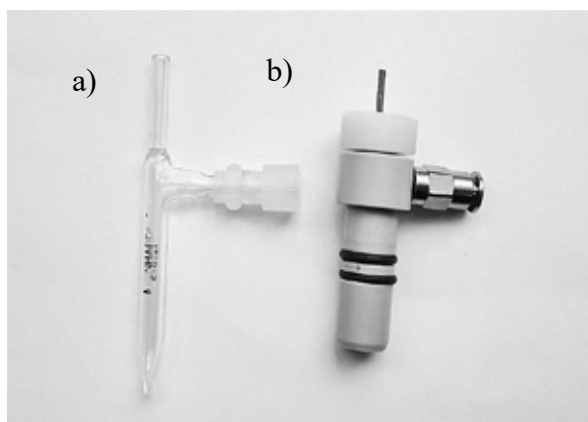


Figure 1. Photos of pneumatic nebulizer: a – Mainhard, b – designed by the authors

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REGULARITIES OF SORPTION OF NOBLE METAL IONS FROM HYDROCHLORIC ACID SOLUTIONS BY POLYVINYLMIDAZOLE DERIVATIVES

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Polyvinylimidazoles are promising materials for the production of sorbents due to the presence of a large number of donor centers in their composition. Quaternization cross-linking of such polymers makes it possible to form anion-exchange materials based on them, including for the separation and concentration of noble metal ions.

The objects of study were materials based on polyvinylimidazoles cross-linked with di-, tri-, tetra- and pentamethylene dibromide. The degree of quaternization of the resulting sorbents was 97, 95, 52 and 45%, respectively. The sorption of gold(III), palladium(II) and platinum(IV) by cross-linked polyvinylimidazoles from hydrochloric acid solutions of various compositions was studied using the limited volume method.

All cross-linked polyvinylimidazoles studied are characterized by similar patterns of sorption of noble metal ions. Thus, the extraction of gold(III) and palladium(II) is maximum in the pH range 2-4 and is more than 90%. Carrying out sorption from 1-2 mol/l hydrochloric acid allows for the selective separation of palladium(II) in the presence of gold(III). When conversion from one- to two-component systems, the nature of the dependence of the sorption of gold(III) and palladium(II) on pH does not change, in contrast to platinum(IV), the sorption of which in the presence of the latter is significantly suppressed. The presence of a 100-fold excess of base metal ions in the system does not affect the sorption of gold(III) and palladium(II) by cross-linked polyvinylimidazoles. Together with the high rate of establishment of sorption, this circumstance determines the prospects for using the studied sorbents in separation and concentration processes.

The research was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation within the framework of the Development Program of the Ural Federal University named after the first President of Russia B.N. Yeltsin in accordance with the strategic academic leadership program "Priority-2030".

STUDY OF THE CHEMICAL COMPOSITION OF TECHNICAL LIGNINS BY MASS SPECTROMETRY

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Technical lignins are an alternative raw material resource to fossil hydrocarbons for obtaining valuable aromatic compounds. However, their wide and rational application is limited by their extremely difficult to understand chemical composition, which is a mixture of several thousand individual compounds and structures.

High-resolution mass spectrometry is a powerful analytical tool for studying the chemical composition of complex mixtures at the molecular level. The development of this area in the study of technical lignins will provide new insights into both the structure of the polymer and its chemical composition.

Our experience in this field allows us to obtain highly informative mass spectra of lignins, covering structures with masses up to 2 kDa and containing several thousand individual components. The application of tandem mass spectrometry allows sequencing of high-molecular-weight lignin oligomers with subsequent modelling of their structures, which, together with two-dimensional NMR spectroscopy, represents an advanced method for studying biopolymers. The obtained data represent a huge amount of information, which requires the application of modern mathematical approaches to their processing. The combined use of mass spectrometry and chemometric methods of analysis opens completely new perspectives in characterisation of lignin and lignin-containing raw materials due to the possibility of visual interpretation of molecular composition or lignin transformation processes in technological processes. The results of implementation of such technologies in lignin mass spectrometry may have important practical significance (development of scientific bases for new processes of deep chemical processing of renewable plant raw materials, including biotechnology, creation of new materials and products based on technical lignins).

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STABILIZATION OF ELECTROCHEMICAL LACTATE BIOSENSORS BASED ON CHITOSAN MEMBRANES

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Detection of lactate is an important analytical task in the fields of biochemistry and medical diagnostics. This compound is the marker of several diseases, e.g. cancer. Suggested sensors are first-generation electrochemical biosensors, which are based on enzymatic oxidation of lactate and consequential electrochemical reduction of hydrogen peroxide on the electrode¹. High selectivity of hydrogen peroxide detection is achieved with modification of electrode surface with electrocatalysts. One of them, Prussian blue, was used in this work.

Immobilization of lactate oxidase was conducted with drop casting of water solution of the enzyme and biopolymer chitosan on working electrode of a Prussian blue modified screen-printed biosensor. In flow injection amperometry mode maximal sensitivity of $346 \pm 71 \text{ mA} \cdot \text{M}^{-1} \cdot \text{cm}^{-2}$ is achieved in a range from 0.05 to 0.1% of chitosan in the casting mixture. Linear range of prepared biosensors is $1 \cdot 10^{-6}$ – $1 \cdot 10^{-4}$ M and they retain 90% of the initial current response on 1 mM lactate solution for 30 minutes. However, stability is insufficient for continuous monitoring of lactate in biological fluids, so additional membranes increasing stability were used. Furthermore, the charge of polyelectrolyte can influence the linear range.

We propose a biosensor with additional sulfonated fluoropolymer coating on the chitosan membrane. Drop casting 0.2% solution of this polymer results in decrease in sensitivity to $97 \pm 22 \text{ mA} \cdot \text{M}^{-1} \cdot \text{cm}^{-2}$ but allows to increase the stability up to 90 minutes. Increasing polymer concentration leads to further decline in sensitivity and improvement of stability of the biosensors. It is important to note that linear calibration range of the biosensor changes too. It was shown that the use of 5% solution leads to the linearity in the range of $1 \cdot 10^{-3}$ – $1 \cdot 10^{-1}$ M of lactate, which is sufficient for undiluted biological fluids analysis.

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SUPRAMOLECULAR SYSTEMS IN ENROFLOXACIN MICROEXTRACTION USING DEEP EUTECTIC SOLVENTS BASED ON TERPENOIDS

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Liquid-liquid microextraction methods open new opportunities for separation and preconcentration of pharmaceutical substances from complex objects (biological samples, food products), allowing to quickly achieve interfacial equilibrium with a minimal amount of extractants. In this case the search for new extraction systems providing effective separation of microquantities of target analytes is a relevant task.

The possibility of formation of supramolecular systems based on primary amines and hydrophobic deep eutectic solvents (DES) is shown. It was found that the introduction of terpenoid and carboxylic acid-based DESs into an isotropic solution of primary amine (volume not exceeding 10 μ L) resulted in the spontaneous separation of a new phase.

The capabilities of the new extraction system are demonstrated on the example of microextraction of veterinary antimicrobial drug of fluoroquinolone series enrofloxacin from aqueous and biological media. The influence of DES composition and nature of primary amines, molar ratio of precursors of supramolecular systems, phase volume ratio, extraction time and sample volume on the extraction efficiency of enrofloxacin was studied. Based on this, the main parameters of microextraction were optimized and a new method of microextraction of enrofloxacin from aqueous media and milk with subsequent determination of the analyte by high-performance liquid chromatography with fluorescence detection was developed. The validation parameters of the developed methodology showed its high efficiency for food and pharmaceutical analysis. The detection limit of enrofloxacin in biological fluids at 1 ng/g per sample was reached.

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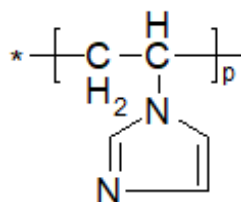
POLY-N-VINYLMIDAZOLE AS AN AMINO ACIDS EXTRACTANT

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Extraction systems based on environmentally friendly water-soluble polymers are currently used to extract amino acids from aqueous media for the purpose of their subsequent determination¹. Poly-N-vinylimidazole has been obtained by radical polymerization for 6 hours (ethyl alcohol is used as a solvent, dinitrile azobisisobutyric acid with a concentration of 10⁻² mol/dm³ is used as a initiator)². The structure of poly-N-vinylimidazole determines its electron-donating properties, in particular, the ability to extract biologically active substances.



Poly-N-vinylimidazole elementary unit structure

Optimal conditions for the extraction of histidine, proline and methionine with a solution of poly-N-vinylimidazole such as salting out agent ammonium sulfate, ratio of aqueous and organic phases 10:4, temperature 20±2 °C and UV spectrophotometric determination of amino acids has been established. The distribution coefficients (D) and the degree of extraction (R, %) of amino acids during extraction with poly-N-vinylimidazole were calculated; the best extraction characteristics are characteristic of histidine.

Extraction characteristics of amino acids

Amino acid	(NH ₄) ₂ SO ₄		NaSO ₃	
	D	R, %	D	R, %
Histidine	107,9	96,3	91,0	89,6
Proline	92,3	91,2	80,4	84,2
Methionine	85,9	88,1	69,5	75,1

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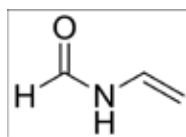
EXTRACTION-ELECTROPHORETIC SEPARATE DETERMINATION OF SORBIC AND BENZOIC ACIDS

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The development of processes for the isolation, separation and determination of sorbic and benzoic acids in aqueous media, food products and drinks is an urgent analytical problem that can be solved using liquid-liquid extraction and capillary electrophoresis. We propose a method for the separate determination of sorbic and benzoic acids, including extraction of their mixture with a solution of poly-N-vinyl-formamide, electrophoretic analysis of the aqueous phase and selective determination of each component of the mixture¹. The rate of formation of the two-phase system water-soluble polymer-water-salt solution of an amino acid is influenced by the molecular weight and intrinsic viscosity of the extractant².



Poly-N-vinylformamide elementary unit structure

The technique is that an aqueous-salt solution of sorbic and benzoic acids with concentrations of 0.045 g/cm³ and 0.01 g/cm³, respectively, is extracted with a solution of poly-N-vinylformamide with a concentration of 0.3 g/cm³, with a phase volume ratio of 10:3. Electrophoretic analysis of the aqueous phase is carried out under the following conditions: voltage +20 kV, temperature 24±1°C, hydrodynamic sample injection under a pressure of 30 mbar for 15 s. The determination of sorbic and benzoic acids is carried out at a wavelength of 254 nm, the leading electrolyte is sodium tetraborate, pH 9.2.

98.2% of sorbic and 97.3% of benzoic acids from their initial content in the analyzed aqueous solution pass into the organic phase. The technique can be used to determine sorbic and benzoic acids in food items.

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PROCEDURES AIMED AT CONTROL OF KR-85 SOURCES MANUFACTURING AT MAYAK RADIOISOTOPE AND RADIOCHEMICAL PLANTS

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Developing metrological methods of conformity assessment is aimed at ensuring the uniformity of measurements and quality control in the course of manufacturing sealed Kr-85 sources at radioisotope and radiochemical plants.

To control the quality of manufacturing the sealed Kr-85 sources, measurement methods have been developed and certified as established in regulatory documents in the field of atomic energy use.

Table 1 below demonstrate metrological characteristics of the methods employed for measurement of ^{85}Kr activity using the Curiementor-4 radiometer and the RKhB-01M radiometric installation, respectively.

Table 1

Range of activity to be measured, Bq (mCi)	Convergence characteristics, σ_{cx}	Residual systematic error, Θ_A	Accuracy, Θ_c	Confidence limits of relative resultant error, δ_A
$3.33 \cdot 10^9$ to $7.40 \cdot 10^{10}$ (90 to 2000)	0.075 %	± 8.9 %	± 7.3 %	± 8.9 %
	2.4 %	± 8.9 %	± 3.8 %	± 10 %

Mayak manufactures sealed Kr-85 sources of various design that allows using them for continuous quality control of products of pulp-and-paper industry, for control of paint coating thickness, for quality control of capacitors, etc. Mayak expands the range of manufactured sources taking into account the needs of the domestic industry and penetrating the market segment previously occupied by foreign manufacturers.

DETERMINATION OF TYLOSIN RESIDUES IN MEAT BY ELISA

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Antibiotics play an important role in modern veterinary and medicine. They are used for treatment various bacterial infections both for animals and people. Failure to comply with the timing of drug use before milking or slaughter can lead to the penetration of antibiotic residues into the human body and cause resistance to treatment with this drug. Tylosin is the macrolide antibiotic using for pneumonia, mastitis, rhinitis and other diseases treatment in poultry, hog-gery and cattle. A number of regulatory documents have established maximum permissible levels to control content of tylosin residues in livestock products.

In modern laboratory practice two methods are used for tylosin detection in samples: high performance liquid chromatography – mass spectrometry and enzyme-linked immunosorbent assay (ELISA). The first one is performed by expensive equipment, plenty of chemicals and also takes many hours. On the other side ELISA allows to analyze 40 samples in two hours with minimal set of reagents.

Previously, ELISA technique for determination of tylosin residues in milk, cream, sour cream, eggs and honey was developed and implemented, so the goal of this research was to select conditions for preparation of meat samples. Solutions with different pH values were tasted as extractants: 5% solution of trichloroacetic acid, acetate buffer, 1 M solution of ammonium sulfate, phosphate buffer, water, borate buffer. Hexane was used for degreasing, also time of extraction and centrifugation was varied.

As the result, it was shown that optimal extraction of tylosin (90-110%) from meat samples is achieved by using phosphate buffer as extractant during 15 minutes and subsequent centrifugation at 4000 rpm. The use of hexane does not affect the result, so the degreasing step can be skipped. In the future it is planned to test this technique on samples of by-products.

MEMBRANE FRACTIONATION AS A BASIS FOR MULTISPECTRAL ANALYSIS OF SOIL ORGANIC MATTER

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Chemical analysis of soil organic matter (SOM) as a complex heterogeneous and heterophase system involving high- and low-molecular compounds as well as colloidal particles is challenging. One of the approaches to its solution is the isolation of characteristic components using membrane filtration, which makes it possible to obtain size fractions from a few nanometers (mass, 10 Da) to several hundred micrometers, and subsequent analysis by spectroscopic methods.

A method for separating water-soluble SOM into narrow fractions by cascade membrane filtration on polyethylene terephthalate and polycarbonate analytical track membranes is proposed. The possibility of fractionation of SOM in the range of particle sizes of 0.01–10 μm is shown, yielding samples with reproducible parameters (total elemental, phase, qualitative and quantitative functional-group composition, and average particle size) for analysis by methods of molecular and atomic spectroscopy. The possibility of trace element analysis of narrow fractions using ICP–AES with direct injection without decomposition is shown. The distribution of element concentrations by fractions is generally independent of the membrane material.

A methodology for IR spectroscopic analysis of SOM fine fractions with drying on the surface of an ATR crystal and fractions of dissolved and suspended SOM using fluorescent excitation–emission matrices is proposed. Ratios of the number of groups of humic-like and tyrosine-like compounds, obtained by volume integration of the intensities of fluorescent matrices, are reproducible and differ from fraction to fraction. The results of using two-dimensional correlation (2D-COS) IR spectroscopy to determine the structural and group composition of SOM, fluorescence 2D-COS, as well as heterospectral 2D-COS (fluorescence–IR, IR–AES and AES–fluorescence) for more accurate identification and assessment of the structural and group composition of SOM in a broad range of size fractions were obtained.

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NEW APPROACHES TO IR SPECTROSCOPY OF SOIL ORGANIC MATTER

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The study and analysis of soil organic matter (SOM), its chemical structure and composition, as well as transformations in land use is a topical problem of chemistry. This problem requires the development of methods for chemical analysis of SOM based on instrumental methods of analysis, primarily spectroscopy. Within the framework of this study, a system for the analysis of SOM based on (1) preparative separation of soils into narrow particle fractions in a broad range of sizes and (2) IR spectroscopic analysis of the selected fractions without their decomposition using complementary methods and techniques of signal registration, as well as specially developed processing of multispectral data, was proposed.

The use of two-dimensional correlation spectroscopy (2D-COS) for micrometer-size soil fractions for IR spectra in various variants provides more accurate identification of almost all bands, as well as establishes the presence of correlations between bands of structures in organomineral complexes of soils. With the help of 2D-COS for samples of similar composition, it is possible to obtain information about differences in the structure-group composition of SOM without additional sample preparation. In particular, the following are manifested bands related to functional groups on the surface of soil particles, aromatic components, a greater number of bands of aliphatic components of SOM, as well as adsorbed forms of water, including in the longwave region (1000 cm^{-1} and below), where the dominance of mineral components does not allow the use of either SOM isolation or destructive sample preparation (annealing or chemolysis).

It is shown that the temperature dependences of the positions of the maxima and intensities of bands in the IR spectrum of soils in the range of low-destructive temperatures ($20\text{--}200^\circ\text{C}$) are analytical signals that are more resistant to changes in experimental conditions than the parameters of these bands at a single temperature. The possibilities of temperature changes in the IR spectra of soil fractions in both the middle and far IR regions as a new parameter characterizing the SOM are shown.

The work was carried out with the financial support of the Russian Science Foundation, project 19-13-00117-P.

FEATURES OF SPECTROPHOTOMETRIC DETERMINATION OF RHODIUM IN SECONDARY RAW MATERIALS

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Platinum metals, their alloys and compounds have unique properties and therefore are widely used in various fields of science and technology. Determination of the content of precious metals (PM) in a variety of industrial products is carried out using classical instrumental methods. Studying the conditions for conducting the analysis of each object is an important stage in the development of analysis methods. The main metrological indicators of the developed methods largely depend on the choice of analysis conditions and instrument parameters that influence the processes.

The method of spectrophotometric determination of rhodium according to GOST 12556.1-82 is very labor-intensive, time-consuming, with a large consumption of reagents and samples, which negatively affects the technological process, the cost of analysis, and irreversible losses of rhodium. Moreover, this GOST can only be used with a palladium content of up to 5.0 wt.%, which makes it impossible to use in alloys with a high palladium content.

Spectrophotometric study of complexes of platinum metals with hydroxylamine, both in individual solutions and in their joint presence, made it possible to establish optimal conditions for determining the rhodium content: the concentration of rhodium in an aliquot, the wavelength and the time to establish equilibrium when heated.

The spectrophotometric determination method is based on comparison of the optical densities of colored complex rhodium chloride in hydrochloric acid solution in the presence of hydroxylamine hydrochloride.

The developed method for determining the mass fraction of rhodium in the range from 10.00 to 40.00 mass. % in materials containing Pt, Pd and Rh allows you to quickly and with the necessary accuracy determine the rhodium content in multicomponent alloys. Tests using this method can be carried out with sample amounts as small as 0.10 g, which makes it possible to reduce irreversible losses of precious metals compared to GOST requirements. Also, using the developed method, it is possible to determine rhodium in objects containing up to 20 wt. % palladium.

DEVELOPMENT OF AN INSTRUMENTAL PLATFORM FOR ASSESSMENT OF GRAPHENE OXIDE PARAMETERS

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Graphene oxide (GO) is a unique two-dimensional graphene-based material consisting of sp^2 clusters surrounded by sp^3 carbon atoms connected to oxygen-containing groups. Aqueous solubility renders graphene oxide the basis for the assembly of nanopharmaceuticals. However, harnessing graphene oxide for this purpose needs robust methodologies for comprehensively evaluating its composition and ensuring control. In this regard, instrumental platforms leveraging molecular and atomic spectroscopies emerge as promising avenues for the simultaneous qualitative and quantitative analysis of graphene-oxide synthesis products derived through diverse chemical and electrochemical methodologies and aqueous GO dispersions.

To date, such advancements still need to be explored. However, every method incorporated into such a platform imposes methodological groundwork. Therefore, comprehending the surface chemistry, particularly the distribution and characteristics of acid sites, is imperative for delineating the chemical properties and reactivity of GO, especially in free-radical processes. There exists a pressing requirement for methodologies to quantitatively assess the distribution of functional groups on GO, akin to the classification of reaction centers observed in materials such as zeolites. These centers primarily consist of heterogeneous strengths including Brønsted acid sites (H^+ donors), Lewis acid sites (most often electron-pair acceptors, metal ions, surface defects in the structure of the material, etc.), and basic centers.

The selected tool for studying acid sites is FTIR-ATR spectroscopy. The initial GO spectra often show broad unresolved bands, which complicate both qualitative and quantitative assessment of acid sites; thus, the method of molecular probes was preferred for this study.

The report will present the results of quantitative assessment of reaction centers using FTIR-ATR and magic-angle spinning NMR spectroscopy. Also, the effect of exhaust gas pretreatment on the number of acid sites will be shown.

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METAL-ORGANIC FRAMEWORKS APPLICATION FOR MERCURY CONCENTRATION AND SPECIATION

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Metal-organic frameworks (MOFs) are a new promising class of materials. These materials have found several applications including storage and separation of gases, sensors, catalysis and functional materials, but in the last decade, they have also attracted interest as stationary phase in separation technologies¹. Their key properties are high porosity, large pore volume, high surface areas, uniform structured cavities and uniform pore size, wide possibilities of direct design of polymers. Due to this properties MOFs can be regarded as promising materials for use in gas chromatography (GC), high performance liquid chromatography (HPLC), and solid phase extraction (SPE) as a stationary phase.

Despite the fact that some MOFs, such as UiO-66, can be synthesized by a method that provides a low cost of the product with a yield of more than 90%, it seems promising to obtain regenerated sorbents, so the possibility of regenerating sorbents will be studied.

From this point of view, Zr-ttdc, a zirconium and thienthiophene dicarboxylic acid based MOF, appears to be a promising compound. Theoretically, this coordination polymer can be used for selective sorption or separation of chemical species of mercury and a number of chalcophile elements. An important difference between this MOF and UiO-66 is the implementation of chemisorption, due to the formation of coordination bonds between active sorption centers and mercury atoms. Moreover, unlike MOFs with sulfhydryl groups, this MOF allows not only to selectively concentrate mercury, but also to ensure regeneration of the sorbent due to weaker interactions with Hg.

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SENSOR PLATFORMS BASED ON PLASMONIC NANOCOMPOSITE MATERIALS FOR DIFFERENTIATION AND DETERMINATION OF ECOTOXICANTS AND BIOLOGICALLY ACTIVE SUBSTANCES BY SURFACE-ENHANCED RAMAN SCATTERING

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Surface-enhanced Raman spectroscopy is currently a highly sensitive and selective method for the determination of a wide range of substances without prior separation and with minimal sample preparation.

However, the currently known sensor surfaces based on noble metals cannot be used for determination of actual analytes due to interfering effects of complex matrix of real objects, poor affinity of analysed compounds to the metal surface; mismatch of spectral characteristics of the sensor surface and analyte. The solving to the above-mentioned problem can be the creation of nanostructured plasmonic composites with controllable spectral and recognition properties and components, allowing reduce/exclude the interfering influence of the matrix of real objects.

We have proposed a sensor system for the determination of ecotoxics (phenols, polyaromatic hydrocarbons, sulfur-containing compounds) in wastewater and fuel. The system is based on the polymer chitosan, which promotes the uniform distribution of silver nanoparticles, extraction and dispersion of the analyte on the sensor surface; the addition of graphene oxide increases the sorption of the analysed compounds and reduces the matrix fluorescence background. To further increase sensitivity, the analysed substances are bound into charge transfer complexes, this makes it possible to realize the effect of resonance Raman scattering.

Also proposed is a sensor system for detecting virus-like particles such as SARS-Cov-2, the system consists of a highly stable silver nanoparticle-based plasmonic surface and an aptasensor that selectively recognises virus-like particles and allows them to firmly attach to the sensor surface.

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DIFFERENTIAL SCANNING CALORIMETRY OF COWS MILK FAT WITH DIFFERENT KEEPING CONDITIONS

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Thermal analysis is applied in the study of fats. It is increasingly used in domestic laboratory practice for fat and oil products quality control. It has been found out that the thermal effects of phase transitions depend on fat chemical composition^{1,2}. The objective of this study was to determine the differences between samples of milk fat obtained from cows of the same breed, the conditions of which are different, with the DSC method. Melting curve profiles (Fig. 1), confirming the difference in the triglyceride composition of milk under different conditions of cows keeping, were obtained as a result of thermal analysis.

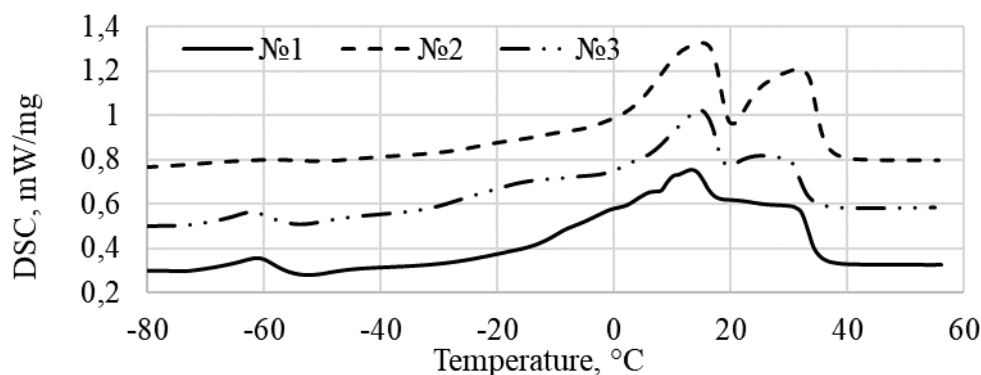


Figure 1. Differential scanning calorimetry in studying milk fat melting of red-motley cows under different keeping conditions:
1 - grazing in meadows; 2- pasture; 3 - stall

Curves 1 and 2 in the thermograms (Fig. 1) have an extremum in the area of -60°C, characteristic of triunsaturated triglycerides. Curve 2 has a depression at 20°C, which is more obvious in curve 3. DSC data indicate that fat polymorphism under meadow and pasture keeping conditions is more obvious. Thus it means that cows receive more valuable nutrition under these conditions.

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APPLICATION OF SOLID PHASE EXTRACTION FOR INVESTIGATION OF VOLATILE AND LEACHED DECAYING LEAF LITTER ORGANIC COMPOUNDS

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Decomposition of leaf litter is one of the key elements of carbon and nutrient circulation in the biosphere. Under the action of various biotic and abiotic factors, litter releases both volatile organic compounds (VOCs) and dissolved organic carbon (DOC) as leachables. Biogenic VOCs are involved in processes occurring in the atmospheric boundary layer, and DOC, being the most active and mobile form of soil carbon, ensures the transport of metals and nutrients in the soil, and also participates in the formation and activity of microbial communities.

In the present study, the composition of organic compounds released into the gas phase and washed out by rainwater from the leaves of four species of deciduous trees – silver birch (*Betula pendula*), European hornbeam (*Carpinus betulus*), black alder (*Alnus glutinosa*) and aspen (*Populus tremula*) – was studied.

Headspace solid phase microextraction (HS-SPME) method using DVB/CAR/PDMS fiber was applied to isolate VOCs from fresh and decomposing litter. Isolation of dissolved organic compounds from rainwater extracts of leaf litter was carried out by solid phase extraction (SPE) using BAKR7020-06 cartridges. The qualitative composition of the isolated organic compounds was determined by gas chromatography with mass spectrometric detection (GC-MS).

In the composition of VOCs of all four types of litter, both fresh and exposed during a six-month field experiment, 213 C1-C16 compounds of various classes, including 83 photochemically active terpenes, were identified. Litter derived VOCs are a mixture of secondary plant metabolites and compounds of microbiological origin. In the composition of DOC leached by rainwater during the field experiment, the presence of 231 organic components was registered, 154 of which were identified. Most of them were carbohydrates, aliphatic acids and alcohols. The results could be applied in the study of global environmental processes.

DETERMINATION OF SHORT-CHAIN FATTY ACIDS IN BLOOD SERUM BY GAS CHROMATOGRAPHY – TANDEM MASS SPECTROMETRY

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The content of short-chain fatty acids (SCFA) in blood serum is an important indicator for the diagnosis and treatment of a number of diseases associated with impaired human immune status. When using a monoquadrupole mass spectrometer, the analytical sensitivity required in clinical practice is not achieved. When using tandem mass spectrometry (GC-MS/MS) for multiple reaction monitoring (MRM) mode for 7 SCFAs, satisfactory accuracy of determinations was achieved in physiologically determined concentration ranges.

Table 1. Conditions for determining SCFA in blood serum by GC-MS/MS

Acid	MMR transition (collision energy, eV)	RT, min	Measuring range, µg/ ml	Coefficient of variation (n=5), %
Acetic D4	63 → 46 (6) 63 → 45 (6)	9.2	Internal standard	
Vinegar	60 → 43 (6) 60 → 45 (9)	9.3	0.5-30	7
Propionic	73 → 55 (9) 74 → 73 (6) 74 → 56 (6)	10.4	0.01-2	9
Iso-oil	73 → 55 (9) 88 → 73 (6)	10.8	0.01-5	7
Oily	60 → 42 (9) 73 → 55 (9)	11.6	0.005-1	7
Isovaleric	60 → 42 (9) 60 → 45 (9)	12.1	0.005-7	7
Valerian	60 → 42 (9) 73 → 55 (9)	12.9	0.005-0.05	10
Nylon	60 → 42 (9) 73 → 55 (9)	14.2	0.005-1	8

Serum (200 µl) with the added internal standard was acidified with HCl and extracted with methyl tert-butyl ether. Chromatographic separation was carried out on a Stabilwax column, 30 m × 0.25 mm × 0.25 µm, in temperature programming mode from 40 to 230 °C at a rate of 10 °C/min. The technique has been successfully tested in the analysis of clinical samples.

This work was supported by the Russian Science Foundation (project No. 23-15-00510).

ICP-OES DETERMINATION OF ELEMENTS IN HOOKAH TOBACCO WITH PRELIMINARY MINERALIZATION OF SAMPLES

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Smoking tobacco poses a significant risk to human health due to the presence of toxic metals such as Cd, Cr, Ni, and Pb, in addition to nicotine. The method of optical emission spectrometry with inductively coupled plasma (ICP-OES) shows promise for quantitative multi-element analysis. However, due to the complicated matrix of hookah tobacco, sample preparation is necessary for analysis. The objective of this study is to experimentally determine the most efficient method for sample digestion.

The decomposition process has been carried out for the following brands of hookah tobacco: Darside, Overdose, Spectrum, and Black Burn. The optimal scheme for sample preparation involved acid digestion of a pre-crushed tobacco sample according to the schemes proposed in GOST and scientific literature (Table 1). The analysis was performed using the Shimadzu ICPE-9000.

Schemes 1 and 2 extracted the maximum number of elements from the sample matrix, produced the most accurate results, had the lowest signal value in the blank experiment, and were also the most efficient and least time-consuming. The schemes were optimized by varying the sample:reagent ratio, the order of reagent application, the possibility of storing mineralizates, and microwave decomposition. The correctness of the obtained results was confirmed on real samples using the ‘injected-found’ method.

Table 1. Schemes of acid digestion (sample weight 0.5 g)

Scheme no.	Reagent mixture
1	3.6 ml HNO ₃ + 0.8 ml HClO ₄
2	3.6 ml HNO ₃ + 0.8 ml HClO ₄ + 0.8 ml H ₂ O ₂
3	4 ml HNO ₃ + 2 ml H ₂ SO ₄ + 1.6 ml HClO ₄
4	4 ml HNO ₃ + 2 ml H ₂ SO ₄ + 1.6 ml H ₂ O ₂
5	3.6 ml HNO ₃ + 0.8 ml H ₂ O ₂

The authors are grateful to the Research Park of St. Petersburg State University “Center for Chemical Analysis and Materials Research”, whose equipment was used in the study.

SPECTROSCOPIC STUDY OF BIOLOGICALLY ACTIVE COPPER (II) COMPLEXES BASED ON *BIS*-HETARYLHYDRAZONES OF 2,6-DIACETILPYRIDINE

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Currently, a large number of papers have been published on copper (II) complexes with hydrazones of mono- and dicarbonyl compounds exhibiting antitumor activity^{1,2}. We synthesized bis-hetarylhydrazones of 2,6-diacetylpyridine, which upon reaction with copper (II) perchlorate and nitrate form a variety of pentacoordinated complexes of the composition $[Cu(HL)C_2H_5OH]X$ and $[Cu_2(HL')_2]X_2$, where HL – *bis*-benzothiazolyhydrazone and *bis*-phthalazinyhydrazone, HL' – *bis*- benzimidazolyhydrazone, $X = NO_3^-, ClO_4^-$). Since the studied complexes have shown significant cytotoxicity against the HepG2 cancer cell line, it is of great interest to further study their parameters of binding to the carrier protein and to develop accessible and sensitive determination methods that can be used both in medical research and drug quality control. A study of the interaction between copper bis-hetarylhydrazone complexes and bovine serum albumin (BSA), which is a structural homolog of human serum albumin, the main carrier protein in the human circulatory system, using fluorescence spectroscopy, showed that all the tested compounds are strongly bound to BSA (the order of binding constants is in the range 4.91–7.03), and no more than two binding site in BSA are involved in associations. The studied copper (II) complexes exhibit a characteristic intense absorption band in the range of 410–460 nm. This property has made it possible to develop spectrophotometric methods for their determination. These methods have demonstrated high sensitivity, with a detection limit as low as $1.1 \cdot 10^{-6}$ M. The accuracy of the analyte measurement was verified using the «input-output» method. These methods have been validated for determining the studied substances in the biological fluid of healthy individuals.

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STUDY OF CHANGES IN THE FRACTIONAL COMPOSITION OF ORGANIC MATTER IN BOTTOM SEDIMENTS USING ISOTOPE RATIO MASS SPECTROMETRY (IRMS)

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The study of organic matter (OM) of bottom sediments and sedimentary rocks of the World Ocean provides insights into the global carbon cycle and climatic changes. The biological signature of OM is significantly transformed during diagenesis depending on the source of OM and the environment. Isotope ratio mass spectrometry is a powerful analytical technique that can detect small changes in the structural and molecular composition of OM based on carbon and nitrogen isotope measurements. The aim of this work was to investigate by IRMS the patterns of changes in the fractional composition of OM in marine sediments during OM biodegradation.

OM extracted from marine sediment was separated into 5 fractions (hexane, hexane-benzene, benzene, benzene-methanol and asphaltene) using solvents of increasing polarity. It is shown that for the gas seep region two clusters can be distinguished based on $\delta^{13}\text{C}$ values of benzene fractions of OM in marine sediments. The center of the first cluster was located in the surface layer (about 10 cm) of marine sediments. The center of the second cluster was located in a deeper layer of sediments. The difference between the cluster centers was 2-3‰. Significantly lower $\delta^{13}\text{C}$ values of the benzene-methanol fractions of OM compared to the $\delta^{13}\text{C}$ values of asphaltenes are associated with the accumulation of bacterial biomass in sediments. Transformation of OM in sediment leads to rapid loss of labile molecules such as carotenoids, decarboxylation of fatty acids to form hydrocarbons, and, subsequently, polymerization of fatty acids and synthesis of geopolymers¹.

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NEW RAPID APPROACHES FOR DETERMINING THE ENZYMATIC ACTIVITY OF LACTOBACTERIAL ENZYMES USING HILIC

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The study of enzymatic kinetics is an urgent task, but usually not for all enzymes the methods already described in the literature are convenient for the researcher. They often take a long time to prepare samples or are not express. One of the promising methods for studying the properties of enzymes is hydrophilic interactions liquid chromatography (HILIC). Approaches for studying enzymatic activity based on this method are currently few, but their development is promising, since this method allows rapid and efficient separation of polar substances, which are often substrates or products of an enzymatic reaction (for example, nucleosides or amino acids), making the analysis more convenient for researcher.

In this work, two approaches were developed for determining the activity of recombinant enzymes RihC and CysK from *L. reuteri* LR1 using HILIC. It was shown that the stationary phases used in the work effectively separate nucleosides and corresponding nitrogenous bases, as well as a number of amino acids, which are substrates and products of enzymatic reactions for said enzymes, and demonstrate better chromatographic characteristics compared to commercially available stationary phases for HPLC, and due to fast sample preparation, they are the most convenient and express ones for studying enzymatic activity.

The work was supported by the Russian Science Foundation grant No. 23-64-10029.

OCCURRENCE OF ORGANIC POLLUTANTS IN ARCTIC ATMOSPHERIC AIR USING THERMAL DESORPTION GAS CHROMATOGRAPHY – HIGH-RESOLUTION MASS SPECTROMETRY

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The study of the chemical composition of the Arctic air and its temporal trends is an important task. Its solution contributes to a deeper understanding of chemical processes in the atmosphere of the polar regions, their impact on climate change and the state of the ozone layer, as well as the assessment of risks for Arctic ecosystems and human health. Nevertheless, the currently available literature data on the chemical composition and levels of atmospheric pollutants at high latitudes are notably incomplete. Most of the earlier studies were focused mainly on the determination of the major gaseous pollutants (nitrogen and sulfur oxides, carbon monoxide and dioxide, ozone, methane), particulate matter, and black carbon, present in relatively high levels and being of paramount importance from the climatology point of view. The range of detected atmospheric pollutants in the Arctic is constantly expanding as new data are being accumulated and more advanced analytical methods are being introduced. Considering the large volumes of production, the long-range transport of pollutants and the increase in anthropogenic impact in the Arctic, new pollutants can become a serious threat to the Arctic ecosystem.

The purpose of this study is to determine the concentration levels of a wide range of highly volatile and semi-volatile priority pollutants, as well as to search for new pollutants in air samples collected during an Arctic expedition on the research vessel “Akademik Keldysh” in the fall of 2022 in the Barents, Pechora, and Kara seas. To achieve this goal, the method of thermal desorption gas chromatography - high-resolution mass spectrometry with a mass analyzer based on an orbitrap ion trap (Orbitrap) was used. The number of quantified compounds varied from 20 to 38 per sample. Benzoic acid was the major constituent, followed by BTEX, phenol, chloroform, bis(2-ethylhexyl) phthalate. As a result of untargeted screening, more than 150 compounds were identified. These include halogenated (Cl, F, I) compounds, compounds with sulfur and nitrogen, aromatic hydrocarbons, alkylphenols, PAHs and their derivatives, phthalates, etc.

This research was performed using instrumentation at the Core Facility Center “Arktika” of the Northern (Arctic) Federal University and was supported by the Ministry of Science and Higher Education of the Russian Federation (No. 0793-2020-0007).

KINETIC-BASED METHOD FOR THE ESTIMATION OF INCREASED Fe^{2+} CONTENT IN NATURAL WATER

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Iron(2+) ions are one of the natural components of groundwater. In small quantities, iron ions are necessary for biological processes. On the other hand, excess iron content leads to negative consequences for humans and worsens the taste and smell of water. In Russia, the maximum allowable concentration of iron in waters is set at 0.3 mg/L (SanPiN 1.2.3685-21). Therefore, simple and rapid methods for Fe^{2+} monitoring are required.

To identify samples of natural waters with high Fe^{2+} content, we used our previously proposed¹ version of the optical “fingerprinting” method, based on carrying out oxidation reactions of carbocyanine dyes in the presence of an object. Natural water samples containing additionally introduced Fe^{2+} ions in the form of Mohr’s salt were introduced into a reaction mixture consisting of a dye, an oxidant and a buffer. Changes in light absorption and fluorescence in the visible and near-IR regions of the spectrum were measured using visualizers every few minutes. The intensities of the received signals were processed by linear discriminant analysis (LDA). The results showed that the method can be used to recognize clean and artificially contaminated with iron(2+) natural waters (fig.1). It is also possible to estimate the Fe^{2+} content in unpolluted waters in the range of 0.003 – 0.3 mg/L.

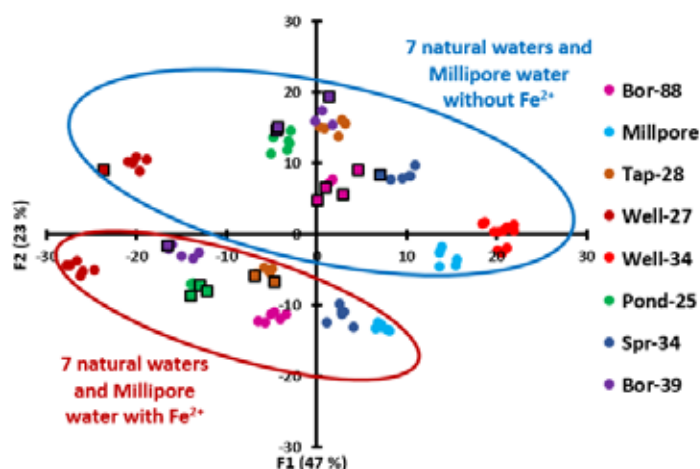


Figure 1. LDA score plot for 7 water samples with and without added Fe^{2+}

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APPLICATION OF THE DYNAMIC LIGHT SCATTERING TO STUDY THE PARTICLE SIZE DISTRIBUTION IN THE POLYDISPERSE SYSTEMS OF NATURAL WATERS USING SYNTHESIZED PMMA PARTICLES WITH KNOWN SIZES

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The chemical composition of natural waters is a key characteristic that determines their quality as the most important natural and technological resource. At the same time, the forms of existence of chemical elements: dissolved (<1nm), colloidal (1nm-1μm), or in the particles (>1μm) are no less important than the total concentration. Determining the size of colloidal particles by dynamic light scattering (DLS) in the case of their polydispersity, which is typical for natural waters, is a non-trivial analytical problem¹. The possibility of using aqueous dispersions containing a mixture of polymethyl methacrylate (PMMA) particles with known size of 80-2000 nm was tested for the correct interpretation of the size distribution spectra of DLS intensity of natural river water samples with different content of colloidal and suspended particles. In addition to the intensity of DLS, the elemental composition of river waters and their fractions obtained after filtration was determined by the ICP-MS method.

For the analysis of DLS, the domestic Photocor Compact device and the imported Zetasizer Nano-ZS analyzer (Malvern, Great Britain) were used.

Analysis of the DLS intensity distribution functions showed that the diameters of PMMA particles in the range from 80 to 600 nm are correctly determined in the mixture only if the mass of large particles is 8-10 times higher than the mass of smaller ones. The addition of 2000 nm PMMA particles to the mixture does not differentiate them from 600 nm particles, although the position of the peak of 80 nm particles is reproduced confidently.

The magnitude of the peaks of the DLS intensity distribution function by size cannot be used to characterize the mass concentration of colloidal particles in contrast to the total DLS intensity of solutions.

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STUDY OF THERMODYNAMICS SORPTION OF COPPER IONS (II) BY A SORBENT OF PLANT ORIGIN

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There are many methods for removing heavy toxic metal ions from water. One promising cleaning method is sorption, the efficiency of which is determined by the quality of the sorbent [1].

An experimental study of the thermodynamics of the sorption process of Cu ions (II) from aqueous solutions on the sorbent, obtained by heat treatment of rice husks, has been conducted.

The resulting adsorption isotherms were analyzed at appropriate linearization coordinates of the classical Langmuir, Temkin and Freundlich adsorption equations. The Freundlich model [2] has been found to be the preferred process model. The experiment at three temperatures allowed to calculate the main thermodynamic characteristics of the process (table. 2)

Table 2. Thermodynamic function parameters for copper cation (II) adsorption process at different temperatures

T, K	ΔH , kJ/mol	$-\Delta G$, kJ/mol	ΔS , J/(mol·K)
284	-12,15	6,2	21,7
297		3,9	13,4
313		5,6	17,7

In conclusion, it may be noted that the results of the experiment in the future will be the basis for developing methods of cleaning water bodies of heavy toxic metals.

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NEW AQUEOUS TWO-PHASE SYSTEMS BASED ON BENZETHONIUM CHLORIDE AND ANIONIC SURFACTANTS FOR EXTRACTION AND PRECONCENTRATION

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The main trend in the development of separation and preconcentration technique is in accordance with the concept of “green” analytical chemistry. Special attention is paid to extraction systems that do not contain organic solvents. Aqueous two-phase systems (ATPS) based on two or more water-soluble components - polymers, alcohols, salts, surfactants, ionic liquids – can be obtained from affordable and cheap components and contain at least 50 mol. % of water in each phase. Aqueous mixtures of some cationic–anionic surfactants can separate spontaneously into two immiscible aqueous phases. One of them is rich, and the other one is poor in surfactants. The phase behavior of cationic/anionic surfactant mixtures strongly depends on the nature of surfactants, the polar surfactant groups, the length of the hydrocarbon chain, and its branching.

New ATPS based on cationic surfactant benzethonium chloride (BztCl) and anionic surfactants sodium dihexylsulfosuccinate (NaDHSS) and sodium N-lauroylsarcosinate (NaLS) are formed at the total surfactant concentration $c_{\text{BztCl}+\text{NaLS}/\text{NaDHSS}} = 0,01\text{--}0,20 \text{ mol L}^{-1}$ and at the molar ratio close to equimolar: $0,36 \leq \chi_{\text{BztCl}} \leq 0,71$ (BztCl/NaLS) и $0,45 \leq \chi_{\text{BztCl}} \leq 0,55$ (BztCl/NaDHSS). For both systems the phase diagrams have been constructed. The liquid-liquid phase separation boundaries in mixtures of anionic and cationic surfactants have been established. The density, viscosity, polarity and water content in surfactants-rich phases have been determined.

New ATPS BztCl–NaDHSS–H₂O and BztCl–NaLS–H₂O were investigated and applied for the microextraction preconcentration of heavy metal ions in natural waters, followed by the ICP-OES determination; the limits of detection for Cd, Co, Cu, Mn, Ni и Pb were 0,04–1,0 µg L⁻¹, the preconcentration factor was 120.

ATPS-based microextraction coupled with subsequent the spectrophotometric and smartphone-based colorimetric determination (directly in the extract, without its separation) of food dyes was proposed. The limits of detection for food dyes were from 0.004 to 0.05 mg L⁻¹. The method was applied to the determination of dyes in food samples and food-processing industrial wastewater.

The work was supported by the Ministry of Science and Higher Education of the Russian Federation, project No. 23-075-67362-1-0409-000195.

DETERMINATION OF IMPURITIES IN ISOTOPICALLY-ENRICHED SILICON TETRAFLUORIDE BY CHROMATO-MASS-SPECTROMETRY

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The use of isotopically-enriched silicon tetrafluoride $^{28}\text{SiF}_4$ as the initial substance for preparation of single-crystal silicon-28 limits the content of molecular impurities in it at the level of up to 10^{-5} mol. %. The molecular composition of impurities in $^{28}\text{SiF}_4$ was studied by the methods of IR-Fourier spectroscopy and gas chromatography¹. To expand the data on impurity composition, it is expedient to use the chromato-mass spectrometry method which makes it possible to reliably identify with high sensitivity the various groups of impurities in one sample.

To increase the efficiency of chromatographic separation and reliability in determination of impurities, we propose:

- the removal of the main substance from the analyzed sample;
- the subsequent cryogenic concentration of the impurity components.

To separate the impurities, the silicon gas-adsorption capillary columns were used. Impurities with low molecular masses and boiling temperatures (atmospheric gases; the oxides of carbon and nitrogen; hydrocarbons $\text{C}_1\text{-C}_4$; freons; silane; sulfur hexafluoride; sulfuryl fluoride) were separated in the 60 m x 0.32 mm GS-GasPro column with modified silica gel. The time of chromatographic determination of these substances did not exceed 18 minutes. To determine the less volatile substances, the 25 m x 0.26 mm x 0.25 μm column was used with the sorbent of poly trimethylsilyl-propyne (PTMSP). Its use made it possible to efficiently separate the hydrocarbons of $\text{C}_3\text{-C}_8$ of a linear and branched structure, freons, aromatic hydrocarbons.

The use of the chromato-mass-spectrometry method made it possible to identify 35 impurities in the samples of $^{28}\text{SiF}_4$ with 28 of these impurities determined for the first time.

The content of impurities in the samples of $^{28}\text{SiF}_4$ is in the interval of $2 \cdot 10^{-3}$ - $6 \cdot 10^{-7}$ vol. %. The limits of the detection of impurities at the level of $10^{-5}\text{-}10^{-7}$ vol. % were attained.

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IDENTIFICATION OF VOLATILE ORGANIC COMPOUNDS EMITTED BY PLANTS OF THE HERBACEOUS-SHRUB LAYER OF THE PERM REGION FORESTS

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Worldwide, plants emit about 0.76 Pg of biogenic volatile organic compounds (BVOCs) per year, most of which are highly reactive and, when entering to the lower atmosphere, they react with various radicals. These reactions lead to the formation of gaseous pollutants, secondary photooxidants and atmospheric aerosols¹. They have a negative effect on the troposphere. The composition of plant emission remains incompletely studied. Since the values of BVOC concentrations calculated in various emission models of compounds are lower than the observed values under the forest canopy. When calculating VOC concentrations in the world, emissions from soil cover plants, which include herbaceous plants, shrubs, etc., are not taken into account².

More than twenty plant species of the herbaceous-shrub layer of forests in the Perm region have been studied. Method of solid-phase microextraction (SPME) was used to extract and concentrate the compounds, and their further identification was carried out using gas chromatography-mass spectrometry.

About 250 compounds belonging to ten classes were found in the gas phase of the studied plant species. The main class of compounds is terpenes, represented by 152 compounds, which include mono- and sesquiterpenes, and their oxygen-containing derivatives. They have high reactivity and can be involved in gas-phase radical oxidation processes^{1,2}.

Thus, it has been established that plants of the herb-shrub layer emit significant amounts of VOCs, therefore, they can influence the total content of VOCs affecting the atmosphere. In this regard, it is of interest to evaluate the content of VOCs emitted by soil plants and their role in photochemical processes in the atmosphere.

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ENZYMATIC INHIBITOR ANALYSIS OF THE DEGREE OF SOIL CONTAMINATION WITH TOXICANTS

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There is a need to develop fast, simple and informative methods of environmental assessment.

This study is aimed at investigating the possibility of using the conjugated enzyme system of luminescent bacteria NAD(P)H:FMN-oxidoreductase + luciferase (Red + Lutz) to predict the potential toxicity of industrial urban soils.

In the course of chemical analysis, it was found that the sampled soils have exceeded MAC levels for fluorine (44-folds), arsenic (7.7-folds) and lead (10.5-folds). Soils contaminated with fluorine have a significant inhibitory effect on the Red + Lutz system. However, no correlation between the content of water-soluble fluorine and the activity of the enzyme system was found. Considering the fact that arsenic accumulation in soils and its transfer into solution strongly depend on soil characteristics, all samples were divided into four groups based on humus content and granulometric composition. It was shown that in group 1 (low and medium humus content, medium loam) there was a significant strong inverse correlation between the gross arsenic content and the activity of the Red + Lutz system ($r = -0.72$; $p < 0.01$). This suggests that the inhibition of the system was caused not so much by soil exposure as by the sensitivity of the enzymes to arsenic. Despite the high total lead content in the soil samples (from 47 to 297 mg/kg), there was no change in the activity of the Red + Lutz system due to the low solubility of lead in water. Concentrations of lead in water extracts from soil had a low correlation coefficient with the values of residual luminescence of the enzyme system ($r = 0.2$; $p < 0.05$), which makes it incorrect to state that the inhibition of the enzyme system is caused by lead, although there is a potential sensitivity to it.

These results emphasize the importance of taking into account the properties of the studied soil samples and the solubility of toxic compounds, since enzymatic biotesting is based on measuring the toxicity of aqueous extracts from soil.

This research was funded by Russian Science Foundation № 24-14-20030, <https://rscf.ru/en/project/24-14-20030/>.

AZANITROGEN HETEROCYCLES AS STRUCTURAL ELEMENTS OF THE RECEPTOR LAYER OF ELECTROCHEMICAL (BIO) SENSORS FOR ECO-, BIO- AND PHARMACEUTICAL MONITORING

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Electrochemical (bio)sensors currently are one of the most promising devices that successfully complement laboratory methods for solving many pressing problems of rapid analysis for medical diagnostics, environmental monitoring and food safety. The nature and structure of the sensor's receptor layer play a key role in ensuring the required analytical characteristics. The use of small organic molecules as functional elements and the development of original approaches to structuring the receptor layer of electrochemical sensors make it possible to expand their analytical capabilities and areas of practical application.

We have proposed original approaches for structuring the receptor layer of electrochemical biosensors using nitrogen heterocycles such as azolo-azine, diazine, and carbazole derivatives as functional elements for the rapid determination of bacteria *E. Coli*, *St. Aureus*, antigen/antibodies to the measles virus, hemagglutinin, nitroaromatic compounds in samples with different matrices. Based on the results of a comprehensive study of the mechanism of the interaction of original molecules with analytes, electrochemical behavior, and the nature of its changes as a result of a supramolecular recognition and/or immobilization on the surface of the electrode, small organic molecules were found to act as cross-linkers, labels and elements of independent biomolecular recognition. Electrochemical structuring methods (electrodeposition, electropolymerization) allowed one to "fine-tune" the analytical characteristics of the developed sensor devices by controlling the thickness and electrical conductivity of the (bio)receptor layer. The proposed approaches and sensors could be integrated into commercially available devices and implemented in the designs of developed laboratory prototypes of original electrochemical test platforms.

The research was financially supported by the Russian Science Fund (project No. 20-13-00142-P, project No. 20-73-10077-P).

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH SIMULTANEOUS COMBINED MASS SPECTROMETRIC DETECTION BY ICP-MS AND APCI-MS METHODS

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The combination of inductively coupled plasma mass spectrometric detection (ICP-MS) and high-resolution mass spectrometry (HRMS) methods demonstrates great potential in the search, identification and comparison of new bromine-containing compounds¹⁻². This work demonstrates the possibility of simultaneous determination of bromine-containing compounds using the combination methods of HPLC-ICP-MS and HPLC-APCI-MS.

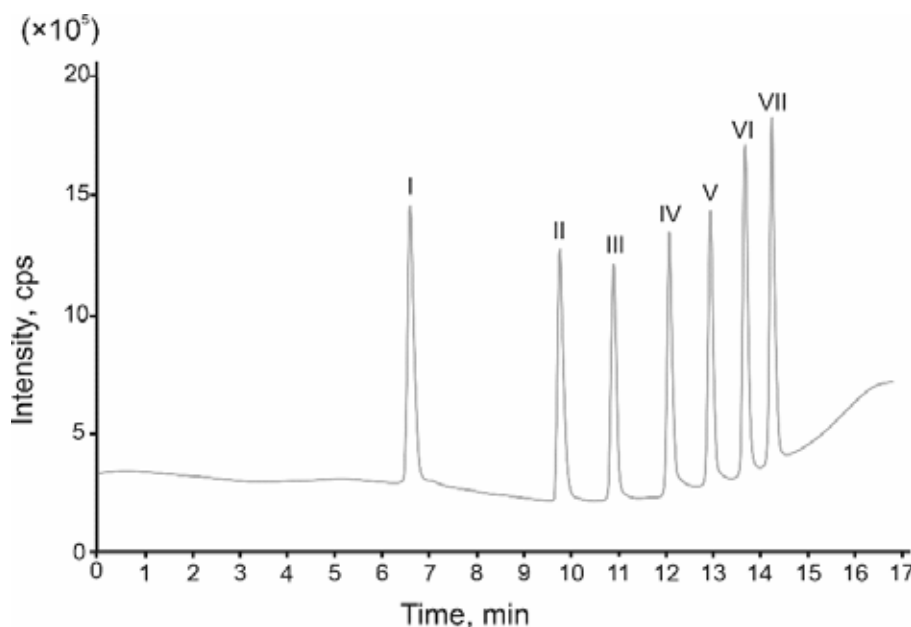


Figure 1. Chromatogram of a mixture of organobromine compounds obtained by HPLC-ICP-MS (I – ambroxol; II – bromhexine; III – 3-bromophenol; IV – 2,6-dibromophenol; V – 2,4-dibromophenol; VI – 3,5-dibromophenol; VII – 2,4,6-tribromophenol)

Split the eluent stream after chromatographic separation allows a portion of it to be sent to APCI-MS, where the release time and structure are confirmed. The other part of the eluent enters the ICP-MS (Fig. 1). Due to the presence of bromine atoms in the structure of the compounds under study, the ICP-MS method showed high selectivity and sensitivity. A single analytical response enables quantitative analysis without the need for separate reference standards.

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This research was performed using instrumentation at the Core Facility Center “Arktika” of the Northern (Arctic) Federal University and was supported by the Russian Science Foundation, grant 24-43-00153.

APPLICATION OF NMR/COS-IR COMBINATION SPECTROSCOPY FOR THE COMPLEX CHARACTERIZATION OF LIGNIN STRUCTURAL FEATURES

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The application of 2D-COS in IR spectroscopy allows to obtain extensive information about the structural and group composition of complex substances or their mixtures, including biopolymers. In this study, we have successfully combined ^{31}P NMR with 2D COS IR spectroscopy to comprehensively analyze the structural features of lignin samples of various origins. NMR spectroscopy data on the content of OH-groups in lignins were used to obtain 2D-COS IR spectra (Fig. 1).

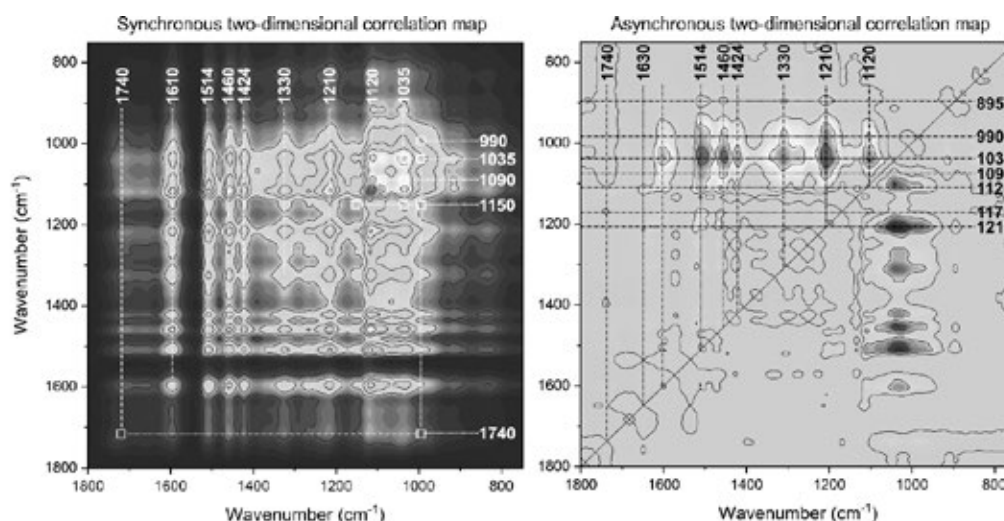


Figure 1. 2D-COS synchronous and asynchronous IR spectra

The corresponding autopeaks, cross-peaks and correlation squares indicated a significant influence of the delignification conditions on the S-units of lignin, and the presence of a polysaccharide component in their structure. Changing the delignification conditions primarily affected the processes of demethoxylation, dehydration, polymerization/condensation of lignin, and then the breaking of lignin-carbohydrates linkages. The development of the proposed approach will provide additional insights into the structural features of lignin preparations and the effects of different delignification conditions on them.

This research was supported by the Russian Science Foundation, project No. 22-13-20015. The equipment of the Core Facility Center "Arktika" of the Northern (Arctic) Federal University named after M.V. Lomonosov was used.

SEMI-QUANTITATIVE DETERMINATION OF KEROGEN IN ROCKS BY IR SPECTROSCOPY

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The generation potential of sedimentary rocks of unconventional reservoirs, such as the Bazhenov and Tyumen formations, depends on the origin of kerogen and the maturity of organic matter (OM). Ordinarily, Fourier-transform infrared spectroscopy (FTIR) is used to determine the structure-group composition, but there are practically no methods for quantitative determination of kerogen in rock without the preliminary stage of kerogen isolation.

Shale samples of the Bazhenov Formation of the West Siberian petroleum Basin were studied by transmission IR spectroscopy to develop a method for semi-quantitative determination of kerogen in rocks.

In the IR spectra of shales, most of the absorption bands characterize vibrations of the functional groups of clay, siliceous and carbonate minerals. OM can be identified in the regions 3000–2800 and 1700–1600 cm^{-1} as stretching vibrations of C–H bonds of aliphatic groups and C=C bonds of aromatic rings, respectively. Kerogen content was calculated as the sum of aliphatic and aromatic OM fragments using optical density values and normalizing IR spectra by absorption bands of quartz (797 cm^{-1}), clay (1100–1000 cm^{-1}) and carbonate (1450–1400 cm^{-1}) minerals, as well as aromatic (1630 cm^{-1}) and aliphatic (2925 cm^{-1}) OM structures. The values of kerogen content found by FTIR in terms of total organic carbon (TOC_{IR} , wt. %) correlate well with the results of programmed Rock-Eval pyrolysis (TOC, wt. %) with a relative error of up to 50% for clayey-carbonate-siliceous rocks.

Correction indices should be used in the normalization formula to improve the accuracy of kerogen determination. The absorption coefficients of C–H and C=C bonds were determined by the method of calibration graph in monocomponent mixtures (with KBr) of model compounds: D-glucose (2944 cm^{-1} , $0.67 \pm 0.04 \text{ mm}^{-1} \cdot \text{mg}^{-1}$), naphthalene (1593 cm^{-1} , $0.43 \pm 0.04 \text{ mm}^{-1} \cdot \text{mg}^{-1}$), and lauric acid (2920 cm^{-1} , $0.22 \pm 0.06 \text{ mm}^{-1} \cdot \text{mg}^{-1}$).

The study was financially supported by Government of Khanty-Mansiysk Autonomous Okrug - Yugra (order from 20.06.2023 № 10-P-1534).

METHOD FOR QUANTITATIVE DETERMINATION OF VEGETABLE OILS IN DAIRY PRODUCTS USING DIGITAL COLOROMETRY

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One of the most popular types of vegetable fat is palm oil. Due to its low cost and long shelf life, it is the most cost-effective substitute for dairy fat¹.

Existing methods for determining the content of palm oil in food products are characterized by lengthy analysis, the use of expensive equipment, and toxic chemical reagents. Therefore, currently, digital colorimetry in combination with fluorescence is a promising and accessible direction in analytical chemistry with good metrological characteristics, and the development of an express method for determining the content of palm oil in food products is a relevant analytical task^{2,3}.

The study proposes a methodology for fluorescent analysis of palm oil content in dairy products using digital colorimetry based on the blue component of color. The influence of factors on the nature of fluorescence was thoroughly studied, optimal analysis conditions were selected, and calibration dependencies for the content of palm oil in butter and curd products were obtained with equations $y = 1,1676 \cdot x + 108,73$ ($r^2=0,99$) and $y = 1,1025 \cdot x + 100,6$ ($r^2=0,99$) respectively.

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STEREOSELECTIVE QUANTITATIVE ANALYSIS OF DIHYDROQUERCETIN DIASTEREOMERS

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One of dihydroquercetin (DHQ) *trans*-isomers – (2*R*,3*R*)-2-(3,4-dihydroxyphenyl)-3,5,7-trihydroxy-2,3-dihydro-4*H*-1-benzopyran-4-one – is registered as an active pharmaceutical ingredients (API). Depending on the production approach, the impurity of *cis*-DHQ may occur. The quantity of this isomer not regulated by specification documents.

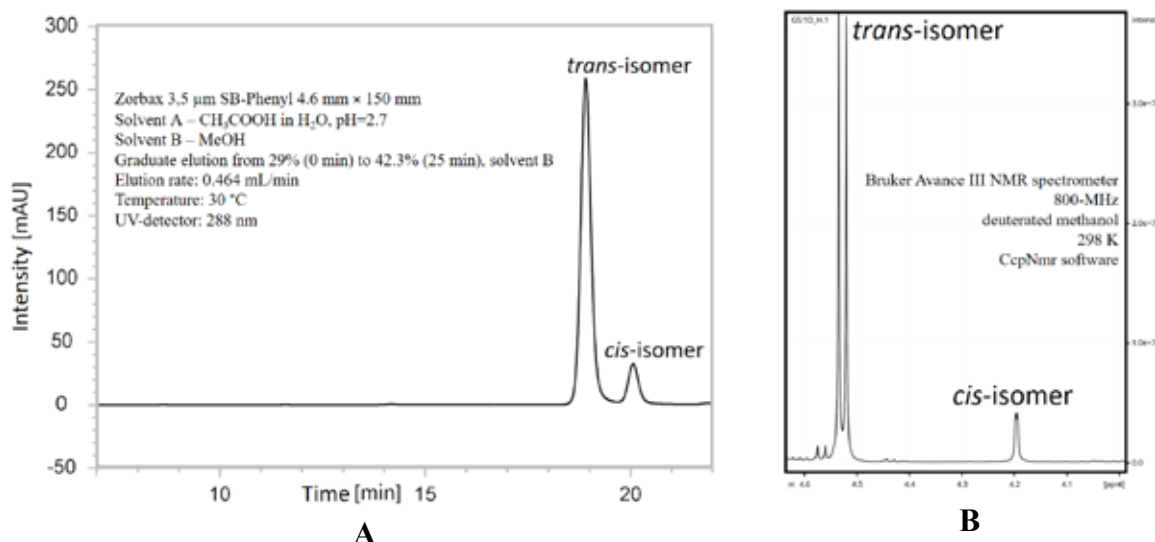


Figure 1. Results of analysis of *cis*-isomer-enriched DHQ: A – HPLC, B – NMR ¹H.

The relative abundance of DHQ diastereomers was determined by the method of internal normalization. According to HPLC under achiral conditions and NMR ¹H spectroscopy, the amounts of *cis*-isomer were 9.5±1.3% and 12.5±2.0%, respectively. There was a good correlation between the results of the methods ($r^2 = 0.9958$). Both methods are appropriate for pharmaceutical analysis based on the following validation characteristics: “Specificity”, “Trueness”, and “Precision”.

This work was financed by the Russian Science Foundation (No 075-15-2022-305), <https://rscf.ru/project/23-75-01130/>.

SORPTION-COLOROMETRIC DETERMINATION OF FOOD DYES USING IMMOBILIZED MICROPROBES

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The combination of sorption preconcentration with subsequent determination of an analyte on the surface of sorbent is one of the effective approaches in chemical analysis. For the development of this area, the attention of researchers is attracted to the engineering of miniature devices for carrying out sorption preconcentration of analytes; in some cases, a single sorbent particle is used.

In this work, sorption microprobes based on silica chemically modified by QAB groups (CMS-QAB) and aluminum oxide were used to determine the anionic food dyes Fast Green (FG), Ponceau-4R (P-4R) and Sunset Yellow (SY) by the method of colorimetry using a laboratory scanner.

The device for sorption isolation of dyes using microprobes was produced by immobilizing a small dense array of sorbent particles (the area size was 1 mm) onto a polymer holder. The conditions for recording the analytical signal using a scanner were optimized: color coordinates: "R" for FG, "G" or "B" for P-4R and SY, resolution of at least 1200 dpi, registration mode – reflection.

The time to achieve sorption equilibrium was 30 minutes. On CMS-QAB, maximal extraction was achieved in the pH range of 2-6, on aluminum oxide – in 0.05-0.15 M HCl. The region of exponential dependence of the analytical signal on the concentration of dyes was observed in the range of 1 – 7 mg/l. It was found that the determination of dyes is not affected by ionic strength in the range of 0.1-0.5 M, as well as 1000-fold excess of citric acid, 500-fold excess of sucrose, and 100-fold excess of starch. The developed method using microprobes based on aluminum oxide was applied to determine P-4R in beverages and FG in tablet capsules.

A device was developed containing an array of remote microprobes, which are individual sorbent particles (aluminum oxide, particle size 100-150 μm). It was found that when recording the signal of individual microprobes, the sensitivity coefficients of the calibration dependences of the color coordinates on the analyte concentration are two times higher than those found for the dense array.

ANALYSIS OF TITANIUM ALLOYS BY MP-AES METHOD

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Widespread atomic emission spectrometers with spark or arc excitation of the spectrum impose strict requirements on the analyzed samples: the minimum sample size must cover the opening of the scouring chamber or be at least the diameter of the counter electrode. The use of special adapters in spark discharge makes it possible to analyze rods and wires with a diameter of at least 3 mm. However, it is not possible to analyze thin welding wires with a diameter of less than 3 mm, as well as thin-walled titanium pipes. In these cases, it is necessary to resort to the use of more suitable equipment, for example, a spectrometer with spectrum excitation in plasma, as well as an additional procedure – acid mineralization.

Atomic emission spectrometers with microwave plasma do not consume flammable and expensive gases (compared to flame in atomic absorption spectrometry or inductively coupled plasma), work using air and allow achieving detection limits better than for flame atomic emission and atomic absorption spectrometers.

The authors propose a method for determining the main alloying and impurity elements (Al, V, Mo, Zr, Cr, Si, Fe) in titanium alloys using an atomic emission spectrometer with microwave plasma Agilent 4120 MP-AES: analytical lines of elements free from significant spectral overlaps were selected; the absence of a matrix effect using aqueous standard solutions was found; the limits of the definition of elements are estimated; an assessment of the metrological characteristics of the technique using model solutions was carried out. The correctness of the developed methodology was verified using standard samples of titanium alloys and the “introduced-found” method.

The developed technique is successfully used in the practice of the Department of Analysis of the composition of substances and materials of the CRISM «Prometey» to determine the elemental composition of the titanium alloys being developed.

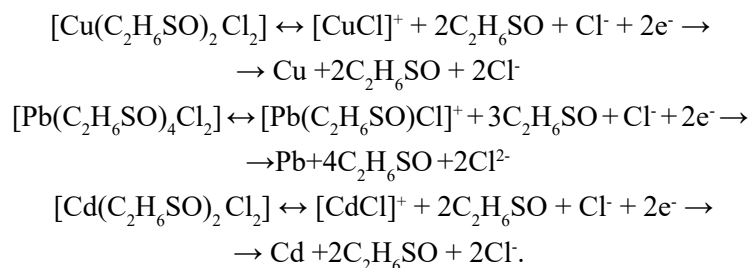
SIMULTANEOUS VOLTAMMETRIC DETERMINATION OF Cu(II), Pb(II) AND Cd(II) USING DMSO-CONTAINING BACKGROUND ELECTROLYTES

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The use of mineral-organic background electrolytes in voltammetry containing organic solvents with high solvating ability makes it possible to increase the selectivity and accuracy of the determination of metallocations due to the formation of metal complexes in solution, which include in the coordination sphere molecules of organic solvents and electrochemically active particles of various compositions. Among organic solvents with high donor numbers, the greatest effect is achieved when using dimethylformamide (DMFA, DN=26.6) and dimethylsulfoxide (DMSO, DN=29.8).

It was found that in acidic chloride media (pH=2; 0.1 M KCl + 0.1 M LiCl) in the presence of 4-6 M DMSO, Cu(II), Pb(II) and Cd(II) are reduced at the electrode with the formation of clear cathode current peaks, while their potentials are shifted to the region of more negative values with an increase in the difference of $\Delta E_{Cu/Pb}$ to 350 mV and $\Delta E_{Pb/Cd}$ to 250 mV (in the absence of DMSO, their values are 200 and 150 mV, respectively). It was found that the following electrochemical reactions take place under these conditions:



Electroreduction is irreversible in the case of copper(II) and cadmium(II), in the case of lead(II) the process is reversible, while the limiting current in all cases is controlled by diffusion. The linear dependence of the limiting current on the content of depolarizers in solution is observed within the following concentration limits: for copper(II) - $4 \cdot 10^{-5} \div 2 \cdot 10^{-3}$ M, for lead(II) - $1 \cdot 10^{-5} \div 6 \cdot 10^{-4}$ M, for cadmium(II) - $5 \cdot 10^{-5} \div 1 \cdot 10^{-3}$ M. A method for the voltammetric determination of copper, lead and cadmium with their simultaneous presence in complex objects against the background of DMSO-containing chloride electrolytes is proposed.

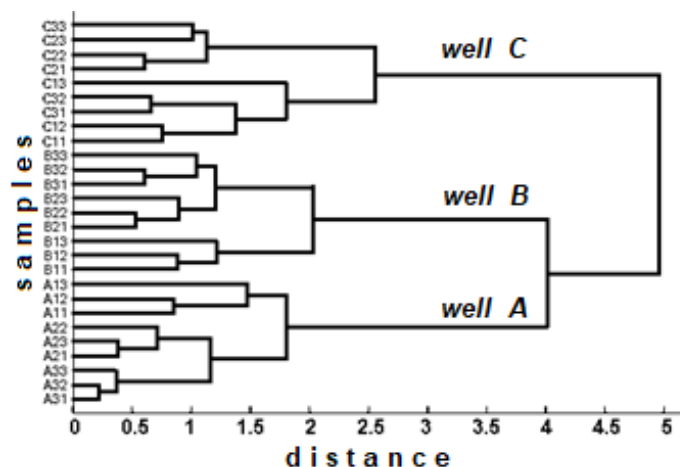
«DATA MINING» ALGORITHMS IN INTERPRETATION RESULTS OF OIL ANALYSIS UNDER UNCONTROLLED UNCERTAINTY IN DATA

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To characterize the facial-genetic conditions of the formation and origin of oil, classify and identify samples, the isomeric composition of various classes of compounds is considered the most effective and informative. However, there are no standard samples of the isomer composition of multicomponent petroleum mixtures for metrological assessment of analysis results. Therefore, the question arises: what and to what extent are the observed differences in the results of the analysis of samples from the same source at different times due to uncontrolled errors in the analysis or real differences in their composition?

In this work the isomeric composition of paraffins and aromatic compounds in oil samples from three wells (*A*, *B* and *C*) was studied using chromatography-mass spectrometry methods in three series of samples with a sampling interval of ~ 2 months. The data array was processed by one of the «DATA MINING» algorithms – the Ward cluster analysis method using the Euclidean metric between points in the space of normalized relative concentrations of isomers in the samples. The result in the form of a dendrogram is shown in the Figure.



Thus, despite on the unavoidable errors in the data, it was possible to differentiate the samples by specific sources (wells) and determine the belonging of wells *A* and *B* to the same productive horizon.

The study was financially supported by Government of Khanty-Mansiysk Autonomous Okrug - Yugra (order from 20.06.2023 № 10-P-1534).

CADMIUM SULFIDE QUANTUM DOTS WITH VARIABLE COMPOSITION SHELL – NEW EFFICIENT PIEZO SENSOR MODIFIERS

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New nanoscale multifunctional materials are being actively introduced into all areas of human life, including providing warning of potential danger, increasing the level of protection of health and life when working in adverse conditions.

The main sensing layer of the sensor is a quantum dot microphase (QDs) based on cadmium sulfide with a different protective shell of variable composition. Sensitivity and sorption affinity are regulated by various macromolecular compounds and dopants to detect, accumulate, and identify hazardous substances.

The aim of the study was to assess the effect of the nature of stabilizing chitosan shells of different composition on the selectivity of sensor arrays with cadmium sulfide quantum dot phases to volatile organic compound (VOC) vapors.

Based on the results of detection of VOC vapors of different classes by an array of sensors based on CT CdS/chitosan with additives, the resolving properties of the “electronic nose” in relation to vapors of water, alcohols, morpholine, chloroform, methyl ethyl ketone, methoxyethanamine, and cyclic amines are highly estimated.

Summarizing all the results allows us to draw a number of important practical conclusions: quantum dots based on cadmium sulfide increase the reliability of detection of marker gases of pathological processes; the use of solid-state and nanostructured sensors makes it possible to highly sensitively and accurately assess the direction of VOC processes in biosamples without sample preparation in 60 seconds; express analysis using gas piezosensors makes it possible to determine the onset of inflammation at earlier stages; The proposed new way of ranking the dataset has shown good classification for samples of complex chemical composition and can be characterized by signs of universality in addition to known chemometric methods.

The work was carried out with the financial support of the RSF grant 23-23-00609.

FROM THE “ELECTRONIC NOSE” TO THE ARTIFICIAL SENSE OF SMELL

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For 25 years, the Department of Analytical and Physical Chemistry of VSUIT has been developing a direction for the development of analytical systems of the “electronic nose” type based on piezoquartz resonators of bulk acoustic waves. Until recently, the main focus of research was on the establishment and description of fundamental properties in sorption systems of volatile organic compounds and micro- and nanophases of piezoresonator modifiers of various natures – from chromatographic phases to nanomaterials, the development of new methods for analyzing various objects using a set of multi-selective sensors. The development of new devices based on 1-12 sensors and software for them did not lag behind. To date, 3 unique devices have a declaration of serial production in Russia. These are the first Russian “noses” based on piezosensors.

However, in order to widely implement odor measurement devices and algorithms in practice, developers, on the one hand, are improving the characteristics of sensors (sensitivity, selectivity/versatility, linearity of responses, reliable operation time and as many measurements as possible without changing properties), and on the other hand, they are looking for new parameters and approaches to obtain more information about complex mixtures of volatile compounds. To create a complete odor recognition system for grading samples in a sample based on a sensor response matrix, chemometric methods are widely used without identifying components.

We have proposed and substantiated new design parameters that allow us to identify individual components or groups of sorption similar in nature and characteristics in complex mixtures. We consider the identification of individual volatile compounds based on the signal of just one sensor with a nanostructured coating sensitive to living molecules common in biosamples of humans, animals, food, and microbiota to be a particular success. Unique approaches to authentication of complex mixtures of molecules with the assignment of certain odor labels are proposed. All this together allows us to talk not only about the success in the development of “electronic noses”, but also about the electronic sense of smell as an integral system that can be used to analyze volatile compounds of a very different nature.

The work was carried out with the financial support of the RSF grant 23-23-00609.

NONIONIC OXYETHYLATED SURFACTANT NEONOL AF 9-10 IN EXTRACTION OF METAL IONS

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Nonionic surfactants are actively used in extraction methods due to their availability and wide extraction capabilities. Neonol AF 9-10 ($C_9H_{19}C_6H_4O(C_2H_4O)_{10}H$) is one of the promising surfactants for concentrating metal ions due to the availability, high degree of biodegradability and low cloud point of its aqueous solutions.

It has been established that extraction with neonol AF 9-10 is possible at temperatures above 80°C, as well as at 25°C using salting out agents - sodium or ammonium sulfate. Delamination in systems persists with the introduction of sulfuric or hydrochloric acids up to 0.6 mol/l, ammonia and sodium hydroxide solution - at a concentration of more than 3 mol/l.

A study of the extraction ability of the proposed systems showed that neonol AF 9-10 in acidic environments concentrates metal ions by a hydrate-solvation mechanism, while the extractability can be increased by introducing complexing agents - halide and thiocyanate ions or neutral oxygen-containing extractants. Conditions have been found for the quantitative extraction of thallium (III) and iron (III) in the form of thiocyanate complexes, as well as conditions for the separation of copper (II) and iron (III) from zinc and cobalt (II), as well as copper (II) from zinc and cobalt (II) during extraction with acetylacetone.

A study of the distribution of organic complexing reagents showed that in a system with ammonium sulfate as a salting out agent, the following are extracted with coefficients of more than 40: arsenazo I, arsenazo III, beryllone II, xylenol orange, and chromasurol S. In the absence of a salting out agent, the distribution coefficients of the studied reagents do not exceed 40. The data obtained made it possible to develop a method for the extraction-spectrophotometric determination of cobalt with 4-(2-pyridalazo) resorcinol in the system neonol AF 9-10 - water (range of determined contents 1.2–2.4 µg/ml, $\varepsilon = 19500$).

USING THE METHOD OF SYNCHRONOUS FLUORESCENCE SCANNING IN ANALYSIS OF PREMIUM VEGETABLE OILS

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Fluorescence spectroscopy is one of the most dynamically developing areas in the analysis of food products and raw materials for their production. The demand for the method is due to the non-destructive type of control, minimal requirements for sample preparation, low cost, high sensitivity and others.

However, the main problem of fluorescence spectroscopy in the study of virgin vegetable oils is the presence in them of a large number of natural fluorophores with similar spectral characteristics: tocopherols, phenolic acids, chlorophylls, vitamins, etc. The method of synchronous scanning of emission and excitation spectra (SF- synchronous fluorescence) consists of carrying out simultaneous scanning excitation and emission while maintaining a fixed wavelength interval ($\Delta\lambda$) between them. The SF method not only simplifies the emission spectrum, but is also less affected by Rayleigh scattering and Raman scattering compared to conventional excitation emission fluorescence (EEF).

A distinctive feature of the method is that, unlike the classical method of studying the emission spectrum λ_{em} or excitation spectrum λ_{ex} , monochromators simultaneously scan both types of spectrum in the studied range with a certain amount of shift. Thus, fixing the difference in wavelengths over the entire range using the formula

$$\lambda_{em} = \lambda_{ex} + \Delta\lambda,$$

the energy degradation parameter is set - the Stokes shift. Similar fluorophores will produce groups of peaks in the spectrum.

SF scanning produces a narrower and simpler spectrum, allowing it to be used in multifluorophore analysis. The SF analysis of virgin grape seed oil made it possible to separate the characteristic fluorescence peaks of tocotrienols and tocopherols with intensity maxima at 302 and 282 nm, respectively, as well as chlorophyll α ($\lambda_{max} = 620$ nm) and chlorophyll β ($\lambda_{max} = 663$ nm).

DEVELOPMENT OF APPROACHES TO DETERMINE THE TOTAL AMOUNT OF PROTEIN IN PLANT MATERIALS

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The reference method for protein determination in the food industry is the Kjeldahl method. It indirectly determines the total protein content by measuring the amount of nitrogen in the sample and then multiplying it by an appropriate conversion factor. The disadvantage of this method is that it determines total nitrogen, regardless of whether it is organic and a part of protein molecules or has been introduced into the product with an extraneous nitrogen-containing substance.

Alternatives to the determination of total nitrogen are direct spectrophotometric analysis and biochemical methods (Bradford, Lowry, micro-biuret). These methods are broadly applicable for determining the individual protein form, but difficulties arise when moving on to plant objects, which contain different proteins, both in terms of their natural composition and molecular weight. These objects are characterized by a complex matrix composition.

The aim of this work is to develop approaches to determine the total protein content in plant raw materials, using the example of sunflower meal (cake) and protein isolates.

The fractional composition of three commercial sunflower protein samples was determined by gel electrophoresis. It includes proteins of two types: water-soluble albumin (SFA) and salt-soluble globulin (helianthinin) with different molecular weights.

Total nitrogen was determined using the Dumas automated method, which unlike the Kjeldahl method is easy to use and does not require the use of toxic reagents.

It was found that the Dumas method combined with the Lowry spectrophotometric method, which is based on a peptide bond reaction, is the most suitable for determining total amount of protein in plant samples. The sufficiently high sensitivity of the Lowry method (the range of detectable concentrations is 10 to 100 µg/mL), allows to exclude the interference of matrix components, such as polysaccharides and phenolic compounds.

The work was supported by the LLC "Biryuch-NT" project number 16112020-1 and by the Russian Science Foundation, project number 23-13-00276.

ELEMENTAL ANALYSIS OF PALLADIUM ALLOYS - MATERIALS FOR HYDROGEN ENERGY

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When conducting research on the creation of new materials for hydrogen energy - palladium alloys of a given chemical composition of systems: Pd - Cu, Pd - Ag, it is necessary to determine the content of both impurity elements and macrocomponents (Cu, Ag).

The use of atomic emission spectrometry with inductively coupled plasma (AES - ICP) and atomic absorption spectroscopy (AAS) - methods of analysis with complementary analytical capabilities, provided high-precision determination of the contents of macrocomponents (Cu, Ag) and functional elements (Ru, In, Y, Sc, Pb, Co) in a wide range of concentrations ($n \cdot 10^{-3}$ - $n \cdot 10\%$) from a limited amount of material (from 5 mg) without the use of certified solid composition standard samples.

Depending on the genesis of the studied compounds, the optimal schemes of sample preparation were found and the composition of acids was selected to obtain stable solutions of easily hydrolyzable matrix and impurity elements. Dissolution of samples was carried out in open or closed systems (Mars 5 microwave oven - CEM Corporation, USA). In the case of difficult-to-open samples (with a ruthenium content of more than 2%), the method of fusion with sodium peroxide was used.

The optimal analytical parameters for AES - ICP and AAS determination of elements were established.

Analytical control techniques provided research on the development of new alloys with enhanced physical and mechanical properties, as well as high specific hydrogen permeability, for hydrogen power generation.

The work was carried out according to the state assignment No. 075-00320-24-00

DEVELOPMENT OF A TECHNIQUE FOR THE RAPID DETERMINATION OF SULFIDES IN CONSTRUCTIONAL STEEL BY THE METHOD OF SPARK ATOMIC EMISSION SPECTROMETRY

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Non-metallic sulfide inclusions (NMI) negatively affect the mechanical properties and corrosion resistance of steel, that's why monitoring the MnS and CaS content in Mn-alloyed structural steel is especially important. Quantitative assessment of the level of NMI is traditionally carried out by optical (GOST 1778, ASTM E1245) or scanning electron microscopy (ASTM E2142) with energy-dispersive X-ray spectroscopy (SEM-EDS), which is characterized by high duration (up to 18 hours) and labor intensity.

To increase the rapidity of determining the content of NMI, a method of spark atomic emission spectrometry (SAES) was proposed using an ARL iSpark 8860 spectrometer (Thermo Fisher Scientific, Switzerland), equipped with OXSAS and Spark-DAT software, which allows processing the spectra of single sparks.

The conditions for determining sulfides using the SAES method were studied according to the scheme described earlier for the determination of oxysulfides, based on the Spark-DAT software option. The determination of sulfides was carried out in two stages. First, the software option algorithms were experimentally selected by comparing high- and low-intensity sparks. As a result of processing the spark diagrams according to a given algorithm, the ratio of the content of the element in the composition of the NMI to its total content was established. Then, at the second stage, pseudo-formulas were proposed to calculate the mass fractions of inclusions in the sample (taking into account the results obtained at the first stage; conversion factors of elements per inclusion component; concentration values of elements included in the inclusion). Since all sulfur is present in steel mainly in the form of sulfides, the possibility of excluding manganese from the algorithm and pseudo-formula, as well as excluding sulfur included in oxysulfides, was investigated. The correctness and high precision of MnS determination using the proposed S_AlCa iRatio algorithm (1) and pseudo-formula (2) has been confirmed:

$$\text{SdatQuIC3}(500; 1900; \text{S01_0}; \text{Al07_0}; \text{Ca03_0}; \text{a}; \text{b}; \text{c}).\text{d}, \quad (1);$$

$$\text{S_AlCa iRatio} \cdot \text{S01_8} \cdot 2.77, \quad (2),$$

where 2.77 is the conversion factor of sulfur to MnS.

The absence of systematic error in the results of sulfide determination was confirmed by Student's test statistics when analyzing a standard sample of ISO UG124 steel (ISO, Yekaterinburg), previously analyzed by SEM-EDS. The consistency of the results of determining sulfides in production steel samples using the SAES method and SEM-EDS is shown.

SERS-SENSORY SYSTEMS FOR THE DETERMINATION OF LOW- AND HIGH-MOLECULAR MARKERS OF NEUROTRANSMITTER METABOLISM

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The total number of patients suffering from Alzheimer's disease (AD) in Russia is about 1.8 million people. Most commonly, more than 90% of cases of the disease are not diagnosed, since existing psychological tests are not applicable for making a reliable diagnosis in the early stages, when treatment is most effective.

AD is a complex of pathogenic processes, the most characteristic feature of which is the formation of oligomeric forms of a peptide, β -amyloid, in brain tissues. In addition, compounds of the catecholamine group (CA) are the key low-molecular markers of neurotransmitter metabolism. Neurodegenerative diseases are accompanied by progressive death of nerve cells and a decrease in the content of CA in the body, respectively.

Currently, a sufficiently large number of sensor systems have been proposed to detect CA in various biological objects. Their main limitations are relatively low sensitivity, reproducibility, and selectivity to related compounds and/or the matrix of the studied samples.

Thus, the aim of the work is to develop highly sensitive sensor systems based on the surface-enhanced Raman spectroscopy (SERS) method for the determination of β -amyloid, as well as catecholamines.

In the course of the work, an indicator system was proposed for the determination of CA based on a complex with copper(II) ions and 4-aminoantipyrine. This approach made it possible to reach limits of detection of the order of units of nM. The SERS sensor system was tested in the analysis of human blood plasma, in which it was possible to detect CA at the level of nanomolar contents.

An indicator system was also developed for the selective determination of the aggregate forms of β -amyloid based on two types of sensor surfaces, which were obtained by introducing silver nanoparticles into a polymer chitosan film and by laser-induced deposition from silver benzoate. These surfaces made it possible to reach limits of detection of order 1.5 nM and 1.5 pM, respectively. When analyzing the aggregate forms of the peptide in human blood plasma, the accuracy of the determination was 98%.

This work was carried out with the financial support of the Russian Science Foundation, project number 20-13-00330.

SEQUENTIAL ELUTION TECHNIQUE ADAPTATION FOR PROBLEMS OF GEOCHEMISTRY BACKGROUND MONITORING

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One of the objectives of analytical chemistry is to support various environmental and geochemical studies, including biogeochemical monitoring. In such studies, speed is extremely important.

We applied SET¹ (sequential elution technique) to mosses and lichens as bioindicators of geochemical background. The results indicate several drawbacks: 1) large standard deviation for parallel initial samples; 2) time-consuming solutions filtration for the solid components separation; 3) control experiment with state standard reference sample for standard elution techniques is unsatisfactory.

For eliminating these shortcomings, the following adaptations were made in the scheme of biomass fraction sample preparation: 1) use larger-volume sample; 2) eliminate the filtration stage; 3) use new decomposition method for solid fraction samples; and 4) use the state standard reference sample for control.

After completing the adapted experiment, the result show that 1) the relative standard deviation does not exceed 20% for all items; 2) good agreement for 14 elements out of 20 for TR-1 standard and BIL-2 standard; 3) the experiment time was halved.

Thus, the following conclusions can be presented: 1) the use the larger-volume sample improves the results; 2) the stage of liquid fraction filtration can be excluded; 3) the scheme of sample preparation of liquid fractions is simplified, the number of stages is reduced.

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DETERMINATION OF THE CEFUROXIME RESIDUES IN LIVESTOCK PRODUCTS

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A method for determining the residues of cefuroxime in food products has been developed. The work belongs to the field of quantitative chemical analysis in the application of the determination of drug residues by HPLC MS/MS.

Cefuroxime belongs to the group of cephalosporin antibiotics of the 2nd generation, relating to the family of beta-lactam drugs. Several drugs with this active substance have been registered in the territory of the Russian Federation: cefuroxime, zinnate, and the combined drug primalact. To date, the actual method¹ for the quantitative determination of cephalosporins is being implemented in the Russian Federation, which does not include cefuroxime in the spectrum of detectable compounds. Technical regulation² establishes control over the residual of antibiotics of the cephalosporin group, and in some countries of the Pacific region, the MRL of cefuroxime is established.

The liquid chromatograph 1290 (Agilent, USA) and the mass spectrometric detector 6500 QTRAP (Sciex, Canada) were used in the work. Stock solutions of cefuroxime and cefapirin-d4 were prepared by dissolution in methanol. The values of the adjustment of the quadrupole analyzer were obtained by direct injection of solutions with a concentration of 200 ng/ml into the mass spectrometer: cefuroxime in the negative mode MRM (ESI) m/z of the precursor ion 423.2, product ions 206.9/318.2, DP -15, CE -18 CXP -12. Sample preparation, chromatography conditions and the parameters of cefapirin-d4 were used from the actual method¹. Quantitative processing of chromatograms was performed by the internal standard method. Metrological characteristics of the method were evaluated. The range of detectable cefuroxime is 5-1000 mkg/kg, the accuracy (limits of relative error with probability $p = 0.95$), $\pm \delta l$, % does not exceed 30.

References:

1 National Standart 34137-2017 Method for determining the residual content of cephalosporins using high-performance liquid chromatography with mass spectrometric detection

2 Technical Regulation of the Customs Union 034/2013 «On the safety of meat and meat products.»

DETERMINATION OF ELEMENTAL COMPOSITION OF FELDSPAR BRECCIA (NWA 11828) BY ICP-MS/AES METHODS

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The complexity of studying meteorites, including lunar ones, whose representatives are the feldspar breccias, lies in their uniqueness and, often, in the limited amount of the material. To create and refine different types of the sample preparation and analysis using various instrumental methods, the terrestrial analogues are required, such as, for instance, Certified Reference Material (CRM) similar to the analyzed objects in petrogenetic characteristics.

For feldspar breccia (lunar matter) NWA-11828 such materials are the certified reference samples of anorthosite MO-6 (IGEM; CRM: GSO 2120-81, Russia) and granite GS-N (ANRT, France).

The quality of the bulk ICP-MS/AES analysis is strongly depends on the completeness of decomposition of the sample mineral components, which may include refractory minerals. In other case it can significantly distort the results of the analysis and affect the geochemical data interpretation.

For sample preparation, the mineral phases of feldspar breccia NWA-11828 were studied and a technique of acid decomposition at atmospheric pressure after solid-phase fluorination (by means of ammonium bifluoride) was developed. The technique consisting of two main operations: reaction with ammonium bifluoride (230° C, 5 hours)

The testing of the developed technique was carried out by analysis of the CRM anorthosite (MO-6) and granite (GS-N) samples. The results obtained were compared with other methods of sample preparation: sample mineralization with acids at atmospheric pressure and in autoclaves. The correctness of methods of sample preparation and analysis was evaluated by comparing the results with certified values of CRM.

It was shown that the use of the developed technique –provides metrologically reliable results for a large number of elements.

MULTILAYER COATINGS OF FUSED-SILICA CAPILLARY WALLS FOR ELECTROPHORETIC SEPARATION OF DRUG ENANTIOMERS

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Determination of enantiomers of drugs is an urgent task of analytical chemistry, since one of the isomers may have a negative effect on the human body. The method of capillary electrophoresis (CE) has fundamental reserves in solving such problems due to the possibility of implementing various modes of analytes separation with usage of electrophoretic systems modifiers.

This study combines the unique properties of nanoparticles and positively charged polyelectrolytes in creation of physically adsorbed multilayer coatings by alternating oppositely charged layers of modifiers. This method of formation allows to enhance the analytes separation selectivity by increasing the concentration of active sites of chiral selector on the capillary walls. The CE separation of enantiomers requires the use of chiral selectors as modifiers of the inner surface of the capillary and/or background electrolyte.

Thus, the goal is to create chiral multilayer coatings using chitosan and albumin as chiral selectors in combination with citrate-stabilized gold nanoparticles (cGNPs) for the separation of drug enantiomers.

The formation of chitosan-based coatings is an understudied area. Its properties are highly dependent on the degree of deacetylation, which primarily affects its solubility. Chitosan can act both the function of a binding layer, forming dense coatings, and as a chiral selector. One- and three-layers coatings based on chitosan were formed which in combination with (2-hydroxypropyl)- β -cyclodextrin (double chiral system) provided separation of enantiomers of β -blockers with high value enantioselectivity.

Multilayer coatings based on albumin as a chiral selector were obtained due to ligand exchange in the coordination sphere of cGNPs. By scanning electron microscopy shown that five-layer coatings demonstrate high modifier density that allowed to achieve chiral separation under capillary electrokinetic chromatography mode.

The study was financially supported by RSF, project 24-13-00378.

Экологические проблемы планеты становятся все более значимыми и тревожащими, поэтому инновации, которые помогали бы эти проблемы решить, не просто привлекают интерес, а приобретают поистине глобальную актуальность. Сохранение окружающей среды и природных ресурсов для будущих поколений – суть новой этики прогресса.

ПРОГРЕСС, БЕЗОПАСНЫЙ ДЛЯ ПРИРОДЫ

Компания «ФосАгро» – лидер российской химической промышленности выступила с инициативой создания фонда для финансовой и научной поддержки перспективных ученых-химиков, в сферу интересов которых попадают охрана окружающей среды, здравоохранение, продовольствие, повышение энергоэффективности и рациональное использование природных ресурсов.

В 2013 году между ЮНЕСКО, Международным союзом теоретической и прикладной химии (IUPAC) и компанией «ФосАгро» было подписано соглашение о партнерстве в создании программы грантов для ученых, занимающихся так называемой «зеленой химией». Предметом этого направления в науке является создание производственных технологий и материалов, безопасных для природы и человека.

Так родился проект «Зеленая химия для жизни», помогающий талантливой молодежи не только материальными средствами для

продолжения исследовательской работы и реализации проектов, но и плодотворным общением со старшими коллегами – видными современными химиками, и организацией информационного обмена в научной среде. Это первый проект в истории ЮНЕСКО и ООН, реализуемый за счет средств российского бизнеса. ФосАгро выделила более \$2,5 млн на развитие и реализацию проекта.

ТРИНАДЦАТЬ МУДРЕЦОВ

Отбор заявок на участие в проекте – обязанность компетентного жюри, в которое входят 13 ученых из 11 стран. Проект-победитель должен соответствовать нескольким критериям. Оцениваются его научная новизна, глобальная и локальная значимость, соответствие принципам «зеленой химии», компетентность молодого ученого и его команды, оснащенность института и лабораторий, в которых предполагается вести проект. Качество работ очень высокое, и членам жюри приходится порой вступать в длительные диспуты по той или иной кандидатуре.

НАУЧНЫЙ ПОИСК БЕЗ ГРАНИЦ

В 2016 году был учрежден специальный грант за исследования в области применения фосфогипса. В 2019 году в штаб-квартире ЮНЕСКО в Париже в рамках открытия Года Периодической таблицы химических элементов было принято решение продлить грантовую программу «Зеленая химия для жизни».

Проблемы экологического характера, к сожалению, есть во всем мире, и во всем мире есть ученые, которые предлагают решения для многих из этих проблем методами и технологиями «зеленой химии». Это наглядно демонстрирует география проекта «Зеленая химия для жизни». За время реализации программы на рассмотрение жюри поступило более 1000 заявок, 55 грантов было присуждено молодым ученым из 33 стран. Самый главный результат грантовой программы – практическое применение разработок молодых исследователей.



ЖИЗНЬ ПОСЛЕ ГРАНТА

Дважды за первые 10 лет существования проекта победителями конкурса становились ученые из России. Одна из них – Галина Калашникова, заведующая лабораторией синтеза и исследования минералоподобных функциональных материалов Центра наноматериаловедения ФИЦ КНЦ РАН. В 2019 году грант «Зеленая химия для жизни» был присужден проекту «Разработка универсального и экологичного метода гранулирования синтетических титаносиликатных материалов (сорбентов, катализаторов, регенерируемых матриц), полученных на основе отходов региональных горно-обогатительных и металлургических производств».

Галина Калашникова:

– В рамках проекта удалось опробовать 5 разных методов грануляции для новых материалов с широким функционалом свойств (сорбенты, катализаторы для органического синтеза, керамические матрицы), которые мы синтезируем именно в нашей лаборатории. Мы смогли выбрать наиболее подходящий метод получения гранул для каждого из них, а также приоритетные нетоксичные связующие. Полученные гранулы были опробованы в качестве сорбентов для очистки жидких радиоактивных отходов от радиоизотопов 137-цезия и 90-стронция совместно с лабораторией хроматографии радиоактивных элементов и станцией переработки радиоактивных отходов Института физической химии и электрохимии им. А. Н. Фрумкина РАН.

Работа по проекту продолжается, несмотря на окончание действия гранта. Все полученные средства были потрачены только на выполнение основной задачи, благодаря чему мы приобрели необходимое оборудование, получили хороший опыт в работе с зарубежными коллегами и выстроили приоритетные направления дальнейшей совместной работы со многими научно-исследовательскими организациями.

Хочется пожелать организаторам конкурса еще больше новых проектов, которые были бы направлены на самые острые вопросы взаимосвязи экологии и современной промышленности.



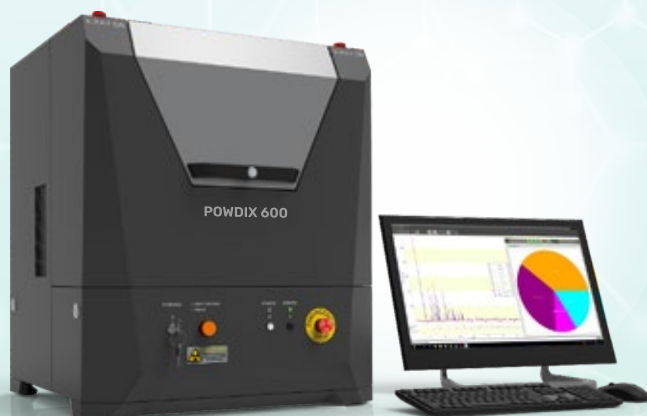
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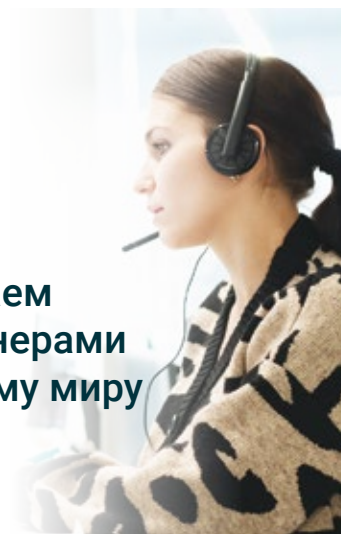
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