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ABSTRACTS

UDC 543.423

Determination of Silver and Palladium using Atomic Spectroscopy Following Their Sorption Preconcentration on Carbon Nanotubes

Grazhulene S. S., Telegin G. F., Zolotareva N. I., Redkin A. N.

Sorption and desorption of silver and palladium ions from aqueous solutions are studied using multi-wall carbon nanotubes (CNT) produced in different conditions by chemical vapour deposition (CVD) and functionalized by subsequent treatment as sorbents. It is shown that sensible difference in the sorption capacity of CNT oxidized under the same conditions (HNO₃ conc., T = 120 °C), depends on the synthesis parameters (on the temperature and type of catalyst). Dependences of Ag⁺ and Pd²⁺ sorption isotherms on different parameters are studied to specify optimal conditions of the element preconcentration in a dynamic mode. Subsequent analysis of the concentrate in eluted solution and in solid phase reformed from the column is carried out by flame atomic-absorption method (FAAS) and arc atomic-emission spectrometry (AES), respectively, demonstrated high preconcentration factor (200 – 600) and recovery (more than 95 %), the determination limits of Ag⁺ and Pd²⁺ at K_c = 300 being 5 × 10⁻⁸ and 1 × 10⁻⁷ % wt. (AES), and 0.3 and 0.8 ng/ml (FAAS), i.e., by two orders of magnitude better compared to direct methods of analysis. Correctness and accuracy of the determinations are confirmed in analysis of spiked samples, and also by comparison of the results of both methods. Sorption capacity of CNT is shown to exceed that of activated carbon and other sorbents.

Keywords: sorption; carbon nanotubes; silver and palladium ions; flame atomic absorption analysis; arc atomic emission analysis.

UDC 54.084:543.381

A Device for Determination of Organic Carbon in Water with Photochemical Persulfate Oxidation in the System of Continuous Gas Flow and FTIR Spectrometric Detection

Zobkov M. B., Zobkova M. V.

An automated and easy to use analytic device is developed for determination of organic carbon concentration in natural, drinking and sewage waters. The operation principle is based on photochemical oxidation of the organic matter (to CO₂) contained in water sample with ammonium persulfate in a continuous gas flow and subsequent determination of CO₂ amount thus formed on a FTIR-spectrometer. Significant advantage of the device consists in a simple design that can be used as an add-on module for FTIR-spectrometer. The device provides determination of total, dissolved and particulate organic carbon up to 470 mg/liter. Standard deviation is 0.1 – 0.5 mg C/liter in the range of determination from 5.5 to 21 mg/liter, the detection limit being 0.1 mg/liter.

Keywords: device; organic carbon; TOC; DOC; POC; photochemical oxidation; ammonium persulfate oxidation; chemical analysis; FTIR spectrometer.

UDC 543.062:546.59

Refining of the Lead Buttons from Nickel and Antimony Impurities in Fire Assay of Gold and Silver

Mandrugin A. V., Serebryanyi B. L., Simakova L. G.

A new technique of refining lead buttons from nickel and antimony impurities in fire assay of gold and silver is developed. The technique is based on low-temperature (700°C) alkaline melting of lead buttons with a saltpeter oxidant present. The proposed method of lead buttons purification is more time-efficient and rather easy to use compared to traditional melting in a scorifying dish: refining at a lower temperature does not require an air blow through the furnace and permanent change of scorifying dishes. Stainless crucibles appeared resistant to alkali-induced damage upon low temperature melting and can be used multiple times. The developed technique is recommended for purification of lead buttons from nickel and antimony impurities as an alternative to traditional melting in scorifying dish.

Keywords: fire assay; low temperature alkaline melting; refining; fire assay lead buttons; nickel; antimony; gold; silver.

UDC 543.49.062:546.56

Extraction-Photometric Determination of Copper (II) with 2-Hydroxy-5-iodthiophenol (H₂R) and the Diphenylguanidine

Zalov A. Z.

Interaction of copper (II) with 2-hydroxy-5-iodthiophenol (H₂R) and the diphenylguanidine (DPHG) is studied. Copper (II) forms a complex at pH 2.5 – 4.8. The molar absorptivity of the complex is 3.30 × 10⁴ (460 nm). The single-batch extraction is 98.0%. A method of copper determination thus developed is tested in analysis of RS of alloys and a number of natural items.

Keywords: complex formation; extraction; copper; 2-hydroxy-5-iodthiophenol; Beer's law.

UDC 539.26:669.245

"PRDU KROS" X-Ray Unit for Rapid Determination of the crystallographic orientation of Cubic Single Crystals Using Back-Reflection Laue Patterns

Potrakhov N. N., Khayutin S. G., Lifshits V. A., Hoses R.

To measure disorientation of the fragments in monocrystalline turbine blades the devices based on Laue method are developed for several decades. A mobile x-ray unit PRDU «KROS» for rapid control of orientation of cubic crystals with a symmetrical

geometry of back-reflection Laue patterns provides a complete determination of the orientation parameters. The computerized system identifies the reflexes on the back Laue pattern, measures their coordinates, and transforms the back reflection Laue pattern into the stereogram. The initial combination of experimental stereogram with the fragment of theoretical one is usually performed in an interactive mode. Precise alignment of stereo-patterns is automated, which improves the reproducibility of the results. To calculate the orientation matrix the computer system used all the reflexes found in the diffraction pattern. Disorientation is resented in the form of a single rotation angle of the diffraction lattice and *HKL* indexes of the rotation axis.

PRDU «KROS» mobile units are successfully used in the aviation industry of the Russian Federation for more than four years.

Keywords: aircraft engine; turbine blades; single crystal; orientation; subgrain disorientation; control; Laue pattern; X-ray unit.

UDC 536.2.08

Measuring System Based on an IT-3 Device Used to Study the Thermal Conductivity of Materials

Rogov I. V., Polunina N. Yu., Rozhkov A. V., Zhukov N. P.

A system for measuring thermal conductivity of a wide class of substances and materials by stationary method of a flat layer including automatic monitoring of the onset of the stationary phase is described.

Keywords: measuring system; mathematical simulation; subcooling parameter; automation of the measurement process.

UDC 534–16

To the Theory of SAW Gas Detectors

Kazakov L. I., Nikolaev Yu. N.

An exact dispersion equation is derived for Surface Acoustic Waves (SAW) propagating along the boundary of an isotropic elastic half-space, coupled with the isotropic elastic layer, free on the other side. Then, under an assumption that the transverse (shear, viscous) wave is completely damped through the layer, the approximate expressions are derived from the exact equation for small amendments to the SAW wave number caused by the reaction of the load, such as: (a) a thin layer of viscoelastic «water-like» substance; (b) thin layer of a viscous liquid; (c) infinitely viscous gas. In the first two cases, the mass of the layer is the load that affects the change in the phase velocity of SAW. Gas loading (variant (c)) in the first approximation does not affect the phase velocity and only causes attenuation of SAW attributed to emission of acoustic wave of compression in infinite gas. For losses in gas the expression refining the basic formula of the known approximate Arzi's et al. theory is specified. The final approximate expression is derived for the wave number of SAW propagating along an isotropic substrate covered with a layer of isotropic material with infinite gas behind. A small increase in the mass of the layer due to absorption of the molecules of the studied gas impurity leads to a decrease (proportional to the impurity concentration) in the phase velocity of SAW and the frequency of the self-oscillator of the SAW detector, thus playing a role of the «microbalance».

Keywords: surface acoustic waves; isotropic substrate; Rayleigh's equation; SAW phase velocity; SAW damping; Arzi's formula; microbalance SAW gas detector.

UDC 53.082.5:681.723

Multispectral Microscopy: Current State and Trends of Development

Kaplunov I. A., Molchanov V. Ya., Yushkov K. B., Kolesnikov A. I., Ilyashenko S. E., Grechishkin R. M.

Main configurations of multispectral microscopes making use of either changeable or continuously tunable optical filters for spectral or spatial scanning of the objects under study including Fourier spectroscopy of their interferograms are considered. Integration of the methodologies of visualization and optical spectroscopy results in a concept of spectral image in the form of 3D data array («cube of information»). Surge of interest in this method of analysis is attributed to growing application potential of the multispectral microscopy in medicine, biotechnologies, materials science, chemistry, mineralogy, forensic science and many other scientific and technological lines. Electronically controlled variable-wavelength acoustooptical filters provide an efficient instrumentation for both spectral and polarization analysis of optical images in real-time operation mode.

Keywords: multispectral microscopy; optical spectroscopy; videospectrometry; acoustooptical filters; electronically tunable filters; paratellurite crystals.

UDC 620.172.2

Adjusting of the Loading Rate in Cyclic Strength Tests

Sierlin A. Ya.

A method of automated self-tuning of the loading rate of test specimens in conditions of multicycle strength tests is developed. The method provides optimal use of the energy potential of the equipment loading the specimen and shortens the analysis procedure. The essence of the method consists in determination of the maximum current that controls electrohydraulic amplifier, comparison of the current value with the maximum control current capacity and (in the case it becomes below the admissible control current value) in leveling both values by increasing the rate of loading.

Keywords: operational life testing; control current; monitoring system; loading program; loading rate.

UDC 620.194.22

Study of the Fracture Characteristics of Aluminum Alloys at Small Deformation Rates in Normal and Aggressive Conditions

Semenychev V. V., Salakhova R. K.

Fracture characteristics obtained upon destruction of the samples cut from the slabs and extruded profile of D16chT aluminum alloy are studied. Tests are carried out on

a tensile testing machine VME-10TM in air and in 3% NaCl solution at low strain rates. Fracture characteristics – time to fracture, fracture energy, elongation, specific reduction, maximum stress and maximum load – are obtained at a strain rate of 4.2×10^{-5} to 4.2×10^{-6} sec⁻¹. Comparison of the fracture characteristics obtained in air and in 3% NaCl solution revealed that time to failure and fracture energy appeared to be the most sensitive parameters in assessing the susceptibility of semiproducts made of D16nhT alloy to corrosion cracking alloy. For the samples cut from the slabs, the most sensitive characteristic is elongation in contrast to the samples obtained from the extruded panels is which elongation remained practically the same.

Keywords: aluminum alloys; corrosion cracking; small deformation rates; fracture characteristics.

UDC 620.172.22:676.017

Determination of the Elastic Constants of Pulp and Paper Materials under In-Plane Tension

Kazakov Ya. V., Kazakova O. Ya., Manakhova T. N., Malkov A. V.

The results of determination of the elastic moduli E_{11} and E_{22} , and Poisson's ratios ν_{12} ν_{21} , shear modulus G_{12} of most common pulp and paper materials under uniaxial in-plane tension at a constant speed mode are presented. A vertical testing machine with microprocessor control is used in the experiments to obtain the stress-strain dependence. Digital photography with high resolution provided registration of the longitudinal and transverse strains. The anisotropy of the pulp and paper materials regarding the elastic moduli and Poisson's ratio is confirmed and the value and range of variation of the elastic constants are determined for the main types of paper and cardboard.

Keywords: paper; cardboard; elastic modulus; Poisson's ratio; shear modulus; anisotropy.

UDC 620.1.05

Testing of the System Polymer Adhesive Composition — Metal Alloy for Adhesive Strength

Mikhailchenkov A. M., Komogortsev V. F., Dyachenko A. V.

The use of polymer adhesive abrasion-resistant dispersion-strengthened materials as a coating requires testing of the system «polymer adhesive composition — surface of the detail» for adhesion strength. Current methods may not always ensure the desired accuracy and comparability of test results for such materials that differ in the composition and structure. A test method which consists in bursting of the studied material formed on the inner cylindrical surface of the die punch using any loading device is developed. The technique and device allow testing of the materials of different composition, structure and mechanical properties thus providing reliable data on an easy to use device, ready for the repeated use.

Keywords: testing; adhesion strength; dispersion-hardened composite; matrix; punch; the loading device; composition; structure; properties; coatings; materials; polymers.

UDC 543.4:543.062:550.4

Developing and Use of the Different Types of Standard Samples of Rocks, Ores, and Soils for Analytical Support for Geological Exploration

Vaganov I. N., Funtikov B. V.

Identification and interpretation of weak geochemical signals entails the necessary of determination of chemical elements at a level of the percent abundance and below, which is impossible without using standard samples (SS) of chemical composition of different objects: multicomponent SS — in analyses of rocks, soils, sediments, and SS certified to 2–5 components — in analysis of various types of ores. We present 16 active SSS of rocks, soils, and ores recognized as international standard samples in CIS countries. SSS are developed in the laboratories of the Analytical center of Bronnitsy Helio-geochemical expedition (BGGE) FSUE «IMGRE». Certified artificial mixture («spectral standards») close in composition to a variety of natural rocks and certified for a wide range (up to 40–45) elements are also used in the laboratories of the geological sector as standard samples. Analytical center of BGGE developed several sets of «special standards» such as «granite», «ultrabasic rock» (alumosilicate-based), «limestone», «dolomite» (carbonate-based), «pyrite» (sulfide-silicate based). Two of them, namely «granite» and «ultrabasic rock» are certified as sectorial standards.

Keywords: standard samples of chemical composition of natural objects; analytical support of exploration activities; certified artificial mixture; sets of «spectral standards»; multielement spectral analysis

UDC 535.434:519.237.7:54–31

Measurements of the Size of Solid Oxide Particles by Laser Diffraction: Case Study of Aluminum Oxide

Petrova E. V., Dresvyannikov A. F., Galiullina N. T.,

Akhmadi Daryakenari M.

The impact of variable conditions (ultrasound and blackout) on the results of laser diffraction measurements of the particle size of electrochemically obtained aluminium oxide and hydroxide is analysed. The algorithm of quantitative metrological evaluation of the measurement results including identification of the types of errors is proposed.

Keywords: measurement of the particle size; laser diffraction method; metrological evaluation; methods of measurement; error of measurements.