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ЛАБОРАТОРИЯ  
ДИАГНОСТИКА МАТЕРИАЛОВ

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# ЗАВОДСКАЯ ЛАБОРАТОРИЯ

## ДИАГНОСТИКА МАТЕРИАЛОВ

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UDC 543+544.173

**Suppression of Ionization as a Particular Case of Matrix Effects in Liquid Chromatography-Mass Spectrometry (generalizing article)**

Chernova E. N., Russkikh Ya. V., Zhakovskaya Z. A., Lopushanskaya E. M., and Gall' N. R.

High performance liquid chromatography-mass spectrometry with ionization at atmospheric pressure is one of the most sensitive and selective methods, though, being an analytical technique, it is also greatly subjected to matrix-related effects. Suppression of analyte ionization by matrix components (IS) is one of the least understood and most unpredictable manifestation of the matrix effects in HPLC-MS analysis. As IS can greatly affect the main characteristics of the method — the detection and quantification limits, precision, and reproducibility — evaluation of the contribution and allowance for IS appeared rather important upon development and validation of the methods of analysis. Considerations on the origin and mechanisms of ionization suppression and brief review regarding the major approaches to identification, minimization, and accounting of IS effect in measurements are presented.

**Keywords:** high performance liquid chromatography-mass spectrometry (HPLC-MS); matrix effects; suppression of ionization; ionization enhancement.

UDC 543.632.495

**Determination of the Elemental Composition of Oil Using a Combined Method of Sample Preparation**

Savonina E. Yu., Maryutina T. A., and Katasonova O. N.

The trace element concentrations in four oils from different fields of the Russian Federation (Arlanskoe, Labaganskoe, Priobskoe, Romashkinskoe) are determined using ICP-MS. Sample preparation is carried out using two procedures: microwave decomposition of oils (in determination of Be, Na, Mg, Al, S, Ca, Ti, V, Cr, Co, Ni, Cu, Zn, Ga, Se, Rb, Sr, Zr, Mo, Cs, Ba, W, Re, U) and extraction concentration with rotating coiled columns (RCC) (in determination of Nb, Ru, Rh, Ag, Cd, Sb, Te, Hf, Ir, Pt, Tl and rare earth elements in oils. The use of RCC provides concentration of trace elements in oil and thus significantly improve the detection limits of ICP-MS.

**Keywords:** extraction; microwave decomposition; sample preparation; rare earth elements; oil; mass-spectrometry with inductively coupled plasma.

UDC 543.422.8, 681.3.06

**Data-Mining Algorithms in Analysis of the Samples with Unknown Matrix**

Molchanova E. I., Korzhova E. N., Stepanova T. V., and Kuzmin V. V.

Combination of Data Mining algorithms (clustering and regression tasks) proposed for determination of a limited number of analytes in the samples of complex chemical composition with an unknown matrix provides elimination of the impact of host environment components on the intensity of analytical lines of the element to be determined. The developed procedure is tested in XRFA determination of S, Fe, Cu, Zn, and As in the samples of flotation concentrate obtained upon processing of polymetallic ores and V and Fe in synthetic film samples close in their physicochemical properties to welding aerosols accumulated on a filter. The error of analysis decreased by a factor of 1.5 – 5 compared to classical Lucas – Tooth regression equation. The developed procedure significantly shortens time of analysis on an x-ray sequential spectrometer.

**Keywords:** Data Mining algorithms; cluster, heterogeneous materials; x-ray fluorescence analysis; model of calibration functions; regression equation; least squares method; calibration samples; error of the adequacy.

UDC 546.77.543.42

**Spectrophotometric Determination of Molybdenum in Ferrous and Non-Ferrous Alloys**

Mirzaeva Kh. A. and Gamzaeva U. G.

The technique of spectrophotometric determination of molybdenum in ferrous and non-ferrous alloys based on the reaction of Mo interaction with bromopyrogallol red with cationic SAS (cetylpyridinium chloride) present is developed. Molybdenum determination in a highly acidic solution does not require strict adherence of pH balance and excludes errors attributed to small changes in the acidity of the environment. The technique is highly selective and does not require additional operations to eliminate the interfering effect of associated components, including the presence of tungsten element-analogue. The technique is rapid and the available as for reagents and equipment thus used. The convergence and accuracy of the results are rather high.

**Keywords:** spectrophotometry; determination of molybdenum with tungsten present; selectivity; ferrous and non-ferrous alloys.

UDC 620.192.63

**Measurements of the X-Ray Tube Focal Spot Using a Digital Flat Detector**

Kosarina E. I., Stepanov A. V., Demidov A. A., and Krupnina O. A.

The size of the radiation source focal spot affects the image quality upon x-ray control of the objects. Prolonged use of x-ray tubes changes the size of the focal spot. Thus, both domestic and foreign regulatory documents contain requirements regarding a recurrent control of the focal spot of the radiation source which is rather time-consuming operation. Usually, the spot image obtained on a high contrast radiographic film is measured with a measuring magnifier or comparator. The method is inaccurate, since it suggests manual scanning of the image with a given step. A possi-



bility of measuring the spot size using analysis of a digital radiographic image of the spot on a monitor of the digital radiographic unit and method of calculating adjustment factors are demonstrated.

**Keywords:** x-ray testing; radioscopy testing; radiation source; radiological apparatus; focal spot; chamber with a diaphragm; geometric unsharpness of image; flat detector; software.

UDC 541.6:677.494

#### Study of Polymer Crazing Using Thermoactivation Current Spectroscopy

*Goldade V. A., Zotov S. V., Ovchinnikov K. V., Kravtsov A. G., Kurbanov M. A., Bajramov A. A., and Nuralyev A. F.*

Thermoactivation current spectroscopy is used to study crazing of polyethylene terephthalate fibers upon their orientation stretching. It is found that each stage of fiber crazing is characterized by specific temperature ranges of extrema location in the spectra of thermally stimulated currents, and crazing is accompanied by the competing processes of redistribution and relaxation of the electret charge. Thus, thermoactivation current spectroscopy can be used to assess microdeformation changes in the structure of polymer dielectrics.

**Keywords:** thermoactivation current spectroscopy; polyethylene terephthalate fibers; crazing; thermally stimulated currents.

UDC 532.137

#### Viscosity Measurements of Liquids by Capillary Viscometer with a Measuring Needle

*Merzlyakov K. S., Shtin S. I., and Uglev N. P.*

The use of a capillary viscometer with a measuring needle as working medium providing accurate viscosity measurements of small volume of low transparent liquids is discussed. Operating principle of the viscometer is described. A working formula of the viscometer is derived proceeding from Stokes equation. Presented results of water viscosity measurements in a range of 25 – 70°C match the referenced data. Pressure distribution over the perimeter of the perpendicular section of working medium (cylindrical needle) is analyzed as a function of the needle shift from central position on a capillary axis. Self-focusing mechanism of the measurement system is demonstrated.

**Keywords:** capillary viscometer; viscosity.

UDC 537.226.621.317

#### Measurements of Dielectric Permeability of Silicon Dioxide at a Superhigh Frequency upon Heating in a High Temperature Waveguide Resonator

*Krylov V. P., Grachev V. A., and Rogov D. A.*

Thermal simulation of temperature fields is used to increase the accuracy of determining dielectric permeability of materials in high temperature waveguide resonator with allowance for the impact of inhomogeneous temperature distribution. Experimental results of measuring the temperature dependence of the dielectric constant  $\varepsilon(T)$  of solid samples of silicon dioxide (up to 1668 °C) at microwave frequencies are presented. The value of  $\varepsilon(T)$  is shown to range within  $\pm 2\%$  of the initial value throughout the whole temperature interval.

**Keywords:** superhigh frequency; dielectric properties; measurement of the dielectric permeability; waveguide resonator; high-temperature heating; silicon dioxide.

UDC 620.179.16:678.5

#### Ultrasonic Control of the Strength Properties of Polymer Composite Materials

*Murashov V. V.*

We present an ultrasonic technique for determination of the physical and mechanical properties of polymer composite materials (PCM) using a complex parameter including the value of the ultrasonic pulse velocity in the plane of the article and frequency value of the main spectral component of the ultrasonic pulse got through the article back and forth. We also propose to assess the degree of accumulation of the microdamages to PCM by their mechanical characteristics (e.g. tensile interlaminar shear and compression strength), determined by nondestructive method. A degree of microdamage accumulation in polymer composite materials can be assessed using their mechanical characteristics (e.g., strength upon interlaminar shear and strength upon compression) determined by a non-destructive method. We also consider a fundamentally new informative diagnostic parameter characterizing the decay of ultrasonic waves in materials: frequency of the main component of the spectrum of ultrasonic pulse passed through the article in forward and backward directions. It is shown that for destruction diagnostics of the material it is advisable to excite a longitudinal wave and a wave similar to a zero-order symmetrical wave.

**Keywords:** polymeric composite materials (PCM); physical and mechanical properties; ultrasonic method; complex parameter of diagnostics; pulse velocity; zero order symmetric wave; frequency of the main spectrum component; degree of microdamage accumulation.

UDC 620.172.216

#### Determination of the Yield Stress of ÉK-181 Steel in Tensile Tests of Ring Samples

*Leont'eva-Smirnova M. V., Izmailov I. N., Valitov I. R., Loshmanov L. P., Kostyukhina A. V., Fedotov P. V., Murzakhanov G. Kh., and Baskakov A. V.*

A method for determination of the yield strength of the material (steel ÉK-181) of fast reactor cladding is developed and substantiated. Mechanical tests for stretch-

ing are carried out on ring specimens (2 mm height on 4-mm cylindrical posts). Processing of the resulted machine stress-strain diagrams in coordinates of displacement of the active capture of the testing machine — force provides determination of the value of the conditional yield strength of the material with a 1.1 % tolerance for the residual deformation.

**Keywords:** ÉK-181 steel; cladding; mechanical tensile tests; yield stress; method of finite elements; super computer; stress intensity; strain intensity; machine stress-strain diagram; true stress-strain diagram.

UDC 620.1.051:620.172

#### Universal Device and Methods of Tribotechnical Tests of Materials and Coatings

*Sutyagin O. V., Meshkov V. V., and Medintsev S. V.*

A universal device for tribotechnical tests of materials and coatings is presented. An example of its practical realization in a prototype is given. Methods of calibration of the measuring systems of the device are described. Ratios for estimation of the average contact pressure and friction distance are determined for a number of renewable joints used in the device in tests for wear and friction. Testing of the prototype showed a reasonable accuracy of the constituent measurement systems, versatility and economic efficiency of the device

**Keywords:** universal module (device); tribotechnical tests; average contact pressure; friction distance; friction and wear.

UDC 620.172.2:677.2

#### GOST 6611.2–73 and ASTM D7269/D7269M-11 for Tensile Testing of Aramid Yarns: Features of Testing

*Perechesova A. D. and Soloveva G. A.*

A problem of experimental determination of the physical and mechanical characteristics of aramid torsion bars ( $D = 0.046$  mm) is considered. An emphasis is made on the choice of test procedure for tensile testing of such materials. In Russia tensile tests of aramid yarns are performed according to standard GOST 6611.2–73 “Textile threads. Methods for determination of breaking load and elongation upon rupture.” However, this standard has been developed before the onset of aramid yarns production in Russia. Current international standards used for tensile testing of aramid yarns are analyzed. Standard ASTM D7269/D7269M-11 is selected as the most suitable. The main principles of standards ASTM and GOST are considered and analyzed.

**Keywords:** test method; breaking strength (force at rupture); breaking tenacity; elongation at break; modulus; microfilament aramid yarns; torsion; tensile test.

UDC 519.24

#### The Use of Multivariate Analysis for the Final Evaluation of the Results of Expert Assessments

*Khalafyan A. A., Temerdashev Z. A., Yakuba Yu. F., and Guguchkina T. I.*

We developed an alternative to summation score method for assessing the results of expert evaluations. The method is based on representation of the objects to be examined in the form of points of multidimensional space in a coordinate system with axes corresponding to experts estimations, the dimension of the space being equal to the number of experts. A distance to the object with the best possible estimations (according to the rule: the smaller the distance, the preferable object) is used as a criterion for final evaluation of the objects with a possibility of their further ranking according to the degree of preferences. For example, expert estimations of tasting wine demonstrated that the developed method is more representative and mathematically justified compared to traditional aggregation of points.

**Keywords:** multivariate statistical analysis; expert evaluation; integral indicator; product quality.

UDC 543.062:543.6:543.621:543.68

#### CFAL of the Analytical center LLC “R&D and Manufacturing Company “Regis”: Decennial Period of Continuous Development

*Andrushchenko V. Yu., Starkova A. A., and Tsiplukhina T. V.*

The activity and developmental landmarks of the Central Fire Assay Laboratory (CFAL) of the Analytical center of LLC “R&D and manufacturing company “Regis” (Blagoveshchensk) are presented. The laboratory was honored with the “Silver mole” award in 2012 and in December 2014 celebrated the tenth anniversary of the lab foundation. Over the past decennial period an ordinary assay lab turned into a multifunctional center focused on comprehensive analytical support to geological exploration, mining, geology and geochemistry, mineralogy, civil-engineering survey, and environmental monitoring. Despite a large scope of analytical work and remoteness from the center of the country, CFAL provides methodological assistance for industrial laboratories of the relating group of companies. Stages of the development, structure and guidelines of lab activities as well as the quality management system are considered. An emphasis is made on the development of the methodology of quantitative chemical analysis (QCA) and availability of standard samples, hiring and training of the assay lab personnel.

**Keywords:** Central fire assay laboratory of the Analytical center LLC “R&D and manufacturing company “Regis”; analytical support; quality management system; environmental monitoring; development of the QCA methodology; occupational guidance; training of the personnel; the “Silver mole” award.