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ДИАГНОСТИКА МАТЕРИАЛОВ

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ЗАВОДСКАЯ ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ

ЕЖЕМЕСЯЧНЫЙ НАУЧНО-ТЕХНИЧЕСКИЙ ЖУРНАЛ ПО АНАЛИТИЧЕСКОЙ ХИМИИ, ФИЗИЧЕСКИМ, МАТЕМАТИЧЕСКИМ И МЕХАНИЧЕСКИМ МЕТОДАМ ИССЛЕДОВАНИЯ, А ТАКЖЕ СЕРТИФИКАЦИИ МАТЕРИАЛОВ

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CONTENTS

ABSTRACTS

UDC 54.061; 54.064; 543.31

Rapid HPLC-MS/MS Determination of Methylphosphonic Acid in Environmental Water

Baygildiev T. M., Rodin I. A., Stavrianidi A. N., Braun A. V., Akhmerova D. I., Shpigun O. A., Rybalchenko I. V.

New rapid LC-MS/MS approach to determination of methylphosphonic acid (MPA) in environmental waters is developed. MPA is a final product of nerve agent hydrolysis. Optimal conditions of LC separation and MS/MS detection are specified. The developed method is characterized by good sensitivity, precision, selectivity. Matrix component do not interfere with the determination in analysis of natural water samples. The developed approach was tested in analysis of spiked samples of environmental water. The detection limit of MPA direct LC-MS/MS determination in environmental waters is 10 ng/ml.

Keywords: products of nerve agent hydrolysis; methylphosphonic acid; HPLC-MS/MS; water analysis.

UDC 543.42

Study of the Efficiency of Carbon Nanotubes as a Spectroscopic Additive

Zolotareva N. I., Burmii Zh. P., Khvostikov V. A.

Using of carbon nanotubes as a spectroscopic additive and their effect on the character of evaporation of the elements from dc arc upon their determination in graphite powder is studied. A method for recording evaporation of the elements from electrode crater of dc arc using photo-electronic is developed. Photoelectric registration system is based on charged coupled devices (CCD) and suggests periodical registration of emission spectra during evaporation followed by proper mathematical treatment of the results.

Keywords: emission spectra; arc discharge; photoelectric registration; evaporation curves; carbon nanotubes.

UDC 543.062

Analytical Application of $Cr_2O_7^{2-}/2Cr^{3+}$ Redox-System for Search of the Unusual Valence States of Oxygen in $BaBiO_3$ Oxide

Barkovskii N. V.

Potentiometry, potentiometric titration and spectrophotometry are used to prove that $BaBiO_3$ oxide is not oxidised with $K_2Cr_2O_7$ in 1 M HCl and thus does not exhibit the reducing properties inherent in oxygen in unusual valence states other than O^{2-}. Constant concentration of $K_2Cr_2O_7$ $BaBiO_3$ during dissolution in $K_2Cr_2O_7$ – HCl mixture also indicates to the absence of reducing properties of oxide. Cr (III) is not detected in the dissolution products. Behaviour of $BaBiO_3$ in chemical reactions in regard to reagents-reductants is attributed to a strong oxidant Bi (V) present in the oxide structure.

Keywords: oxide $BaBiO_3$; valence state; unusual valence states of oxygen; redox-systeme $Cr_2O_7^{2-}/2Cr^{3+}$.

UDC 543.424:615.074

Determination of Codeine in Drugs by Diffuse Reflection Spectroscopy

Nemikhin V. V., Kachin S. V., Metelitsa S. I., Losev V. N., Sagalakov S. A., Shakhvorostova T. S.

A procedure of diffuse reflection spectroscopy is developed for codeine (25 – 1000 μg/ml) determination in codeine-containing drugs with a preliminary TLC — separation of the contaminants. The detection calculated limit is 9 μg/ml and the relative standard deviation does not exceed 0.05. The procedure of codeine determination is successfully tested using samples of Sedalgin NEO, Pentalgin-N, Pentalgin Plus. The results of codeine determination in the certified samples give grounds to use the developed procedure in practice of expert institutions.

Keywords: codeine; determination; drugs; diffuse reflection spectroscopy.

UDC 543.544:546.289

High Sensitive Gas Chromatographic/Mass Spectrometric Determination of the Impurities in High Purity Monogermane Using Adsorption Capillary Column with a Carbon Sorbent

Krylov V. A., Chernova O. Yu., Syuzin A. Yu.

The use of quartz capillary column with a carbon adsorbent is shown to provide efficient separation and detection of the impurities in monogermane, i.e., permanent gases, carbon dioxide, nitrous oxide, hydrocarbons $C_1 - C_2$ and silicon tetrahydride. The effect of the amount of injected monogermane on the determination of ethane and carbon dioxide is studied. Limits of chromatographic/mass spectrometric detection of the impurities ranged within $(10 - 1) \times 10^{-6}$ % mol. We managed to attain sevenfold reduction of the detection limit of the limiting impurity, ethane, compared to the literature data.

Keywords: monogermane of high purity; adsorption capillary column, impurities; detection limit; correctness; gas chromatography/mass spectrometry.

UDC 678.06–419:677.521

The Role of Heat and Humidity Tests in the Development of New Polymeric Composite Materials

Valevin E. O., Shvedkova A. K., Bukharov S. V.

A role of climatic heat and humidity tests of polymeric composite materials (PCM) in the development and comparative analysis of novel composite materials is considered. The effect of high humidity of the environment on the glass transition tempera-

ture, relaxation transition zone and strength properties of PCM at different operation temperatures. Complex study of the resistance of polymer matrices and PCM to climatic factors in the framework of the developed approach suggests treating of the lower temperature limit of the glass transition region of the polymer matrix in the state of maximum water absorption as the upper temperature limit of operation for the materials and products on their base.

Keywords: polymer composite material; binder; heat and humidity test; moisture sorption; structural transformations; region and temperature of glass transition; flexural strength.

UDC 539.24, 539.27

Method of Metallographic Sample Preparation for Electron Backscatter Diffraction(EBSD)

Zavdoveev A. V., Pashinskaya E. G., Varyukin V. N., Burhovetcky V. V., Vertsanova E. V., Maksakova A. A.

A new method of manufacturing metallographic samples for electron backscatter diffraction (EBSD) in a chamber of scanning electron microscope is developed. An important feature of the method consists in the possibility of cold pouring of metal samples into conductive filler for subsequent preparation of the samples and electropolishing. The metal sample is poured with a composite consisting of a self-hardening plastic "Protokril M," carbonyl iron powder and iron turnings about 100 μm which provide sufficient conductivity not only for shooting of the sample in a scanning microscope, but also for electropolishing. The developed composite unlike self-hardening conductive analogues that require heating to 150°C polymerizes at room temperature which is an important advantage.

Keywords: metallographic samples; electron backscattered diffraction; steel; copper; titanium; aluminum.

UDC 621.74.08:045

Comparative Evaluation of Gas Defects Formation in Investment Casting

Leushina L. I., Koshelev O. S., Leushin I. O., Ulyanov V. A.

A problem of upon investment casting is considered. A method of forecasting and assessing the probability of gas-related defect formation based on the results of traditional experimental control of gas permeability and gas-generation value of the shell material at the stage of shell mold manufacture is developed. The method is successfully tested in a comparative assessment of the risk of gas-related defect formation both in the technology of investment casting including resource-saving scheme of low-temperature calcination of investment molds and in the framework of traditional technologies accepted at the enterprise. The developed procedure appeared especially relevant in conditions of multiproduct production.

Keywords: investment casting; gas generation value; gas permeability; gas-related defect; comparative evaluation of investment casting technologies; gas generation potential of the shell mold material.

UDC 543.422.8

X-Ray Fluorescent Control of the Inhomogeneity of Used Oil Sludge of Aviation Engine

Pavlinsky G. V., Vladimirova L. I., Drokov V. G., Stepuk T. D.

A novel approach to the topical problem of rapid diagnostics of technical condition of in-service aircraft engines is developed. Diagnostics is based on assessing the inhomogeneity of the used oil sludge which is attributed to the existence of large metal particles in the sludge, the size and number of which increases with tearing of rubbing surfaces of aircraft engines. The degree of the inhomogeneity of the sludge is determined by X-Ray fluorescence method. The intensity of x-ray fluorescence of the sludge provided determination (using single-factor analysis of variance) of the substantial effect of the inhomogeneity factor of the analyzed sludge. The limit value of the inhomogeneity factor is determined on the array of measured intensities of sludges of used oils of the engines in good condition, thus sorting out the engines with high inhomogeneity factor, i.e., a high content of large particles in used oil sludges. The elemental composition of large particles enables us to specify the degree of tearing of individual parts of the aviation engine under study.

Keywords: diagnosis; used aviation oil; size of the particles; factor of inhomogeneity; x-ray fluorescence signal; dispersion analysis; elemental composition.

UDC 531.75

Flotation Method in Determination of the Density of Low Weight Solid Samples

Guliutin A. V.

A review of the known methods of density determination of low weight samples of solid substances is presented. A modification of the flotation method is developed for the samples which weigh at least 1 mg and have a density of 1 g/cm^3 and more. The sample under study is coupled with a sample of a plastic material, namely crude bee wax, and placed into a working liquid consisting of the density regulator (potassium bromide), solvent (water) and wetting agent (n-propyl alcohol). After 30-min dwell a composite sample is tranformed into a suspension by changing concentration of potassium bromide at a temperature close to 20°C to measure the liquid density using the pycnometric method. The density of the studied material is calculated using an empirical expression. The results of the density determination for the variety of solid samples of different mass and density match satisfactory the reference date.

Keywords: density; solid substance; bee wax; build-up specimen; working liquid; wetting agent; suspension; pycnometry; flotation.

UDC 620.163.3:531.781

Evaluation of the Range of Reliable Residual Stress Measurements Using Hole Drilling Technique

Apalkov A. A., Odintsev I. N., Plotnikov A. S.

Finite-element method is used to obtain numerical evaluation of the range of reliable measuring of residual stresses using hole drilling technique. It is shown that approach based on basis functions provides rather accurate (for practical applications) recovery of the ratio between stress components, but more accurate determination of the stress components is possible only when the intensity of the residual stresses ranges within 0 – 0.7 of the yield strength of tested material.

Keywords: hole-drilling technique; finite element method; residual stresses; range of the reliable measurements; plastic strain.

UDC 620.162.2:534.1:534.121.2

Study of Axisymmetric Oscillations of a Circular Composite Membrane

Kravchuk A. S., Kravchuk A. I., Tarasyuk I. A.

We have found a mistake in the classical oscillation equation for a round membrane [1] attributed to the kind of interactions. Correction of an erroneous idea regarding the nature of the impact and substitution of tensile forces by tensile stresses provides us to derive the equations of small transverse oscillations of circular membrane with allowance for mechanical and rheological characteristics of the membrane. We derived the oscillation equation for a homogeneous circular membrane and determined its Eigen frequency taking into account the mechanical and rheological properties of the membrane material. Solution of a differential equation by the Fourier method requires an additional change of variables to avoid incorrect results when calculating Eigen frequencies. The oscillation equation for a circular membrane and Eigen frequencies are obtained as function of the component concentration in the case of linear elastic composite material. Recommendations regarding taking into account the creep value of the composite material of the membrane are specified proceeding from the technical theory of aging. It was found that the calculation of the effective characteristics of the membrane material in accordance with the Voigt hypothesis corresponds to the solution of the problem of averaging set for horizontally layered membrane. Application of Reiss hypothesis corresponds to oscillations of vertical coaxial layered or vertically fibrous membrane, whereas application of Kravchuk-Tarasyuk method to narrowing of the Reuss-Voigt "range" corresponds to the best approximation of the effective properties of structurally inhomogeneous composite membrane.

Keywords: composite material; Kravchuk – Tarasyuk approximation of the effective properties of materials; small transverse oscillations; component concentrations; linearly elastic material; uniformly aging quasi-elastic material.

UDC 620.191.33:621.532.3

Study of the State of Pipe Metal and Welded Joints upon Expert Evaluation of the Objects of PJSC "Gazprom"

Konnov V. V., Konnov Vl. Vl., Vazhenin D. V., Pronin N. S., Savin V. G., Solov'ev D. S.

We present the experience of the JSC SPC "Molniya" in expert evaluation of industrial safety of gas pipelines manufactured according to State Standard GOST 10705–80 using butt welding by high frequency currents. The use of steel sheets with a high content of non-metallic inclusions and non-uniformity of their distribution results in reduced ductility of welded joints and cracking of the pipes in service which entails the necessity of studying the state of the pipe metal using a complex of non-destructive and destructive methods of testing for proved forecasting of safe operation of the pipeline.

Keywords: pipeline; crack; base metal; welded joint; non-metallic inclusion; chemical composition; metallographic control; fractographic analysis; static tensile; yield strength; impact toughness.

UDC 543.08+511.33

Application of Table of Congruences and Correspondence Analysis to Comparative Assessment of Wine Quality in Rating Scale

Khalafyan A. A., Yakuba Yu. F., Temerdashev Z. A.

An actual problem of wine quality assessment by the methods of mathematical statistics is discussed. Different approaches to assessment of the wine quality in rating scale (high, average, low, falsification) using the content of volatile substances and wine tasting results are considered. Using two-dimensional frequency analysis — tables of congruences (crosstabulation) a strong correlation is shown between two ways of wine's division into groups — using a tasting assessment and concentration of volatile substances. Structure of the correlation is also studied. Method of correspondence analysis revealed a significant resemblance between classifications of the wine samples of high quality and falsification; much weaker — between wines of low quality and small — between wines of average quality.

Keywords: assessment of wine quality; rating scale; two-dimensional frequency analysis; tables of congruences; crosstabulation.

UDC 006.015.5

Analysis of the Regulatory and Procedural Support of Voluntary Certification of Products

Ivanova V. A., Yablonskii O. P.

Methodology of functional modeling IDEF0 is in analysis of the regulatory and procedural support of voluntary certification of products. The requirements to the regulatory and procedural support of voluntary certification of products to be considered when drafting documentation are specified.

Keywords: voluntary product certification; regulatory and procedural documentation; process; modeling.