

ЗАВОДСКАЯ  
ЛАБОРАТОРИЯ  
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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## ABSTRACTS

UDC 543.62

**Control of the Composition of Welding Aerosols**

*Korzhova E. N., Stepanova T. V., Lodousamba S., Smagunova A. N.*

Physicochemical properties of the solid component of welding aerosols (SCWA) are characterized. The features of the SCWA sampling and the methods most frequently used for SCWA analysis in domestic and foreign analytical practice are considered. Destructive and nondestructive methods of SCWA analysis are compared from the viewpoint of their advantages and shortcomings. It is shown that losses of the elements under control frequently observed upon decomposition of exposed filters are attributed to the complexity of the phase and chemical composition of the SCWA samples. Methods of developing synthetic calibration samples are discussed for both classes of methods considered: when using x-ray fluorescence method it is difficult to prepare the calibration samples adequate to the real SCWA samples collected on the filter, whereas in comparator variant of the neutron activation analysis synthetic mixtures containing one or two components are used to calculate the content of analytes. Metrological characteristics of the methods of SCWA analysis are presented.

**Keywords:** solid component of welding aerosols; physicochemical properties of the particles; inorganic components; destructive and nondestructive methods of analysis; sample preparation; calibration samples; validation.

UDC 543.848:543.257.2.

**Simultaneous Determination of Halogens in Organic Compounds Using Ion-Selective Electrodes**

*Fadeeva V. P., Deryabina Yu. M., Nikulicheva O. N., Tikhova V. D.*

A technique of simultaneous determination of halogens (Cl, Br, I) in halogen-containing organic substances is developed. Sample preparation consists in combustion of the substances in a flask filled with oxygen and the absorption of combustion products with 0.1 M NaOH solution added with  $\text{H}_2\text{O}_2$ . Halide ions are determined by potentiometric titration with 0.02 N solution of  $\text{AgNO}_3$  with ion-selective electrodes in aqueous-alcoholic medium added with an acetic acid which prevents adsorption of free halide ions on a precipitate of silver halide thus formed. The developed method is used in analysis of a variety of halogen-contained organic substances that differ both in the elemental composition and structure. N, S, P, F, Na, K, Ca, Ba, and Ti present do not interfere with halogen determination or affect the results of their determination.

**Keywords:** halogen-contained organic compounds; determination of halogens; potentiometric titration; ion-selective electrodes.

UDC 544.653.22

**Voltammetric Determination of Glutathione Using Graphite Electrodes Modified with Gold Nanoparticles**

*Perevezentseva D. O., Gorchakov É. V.*

The electrochemical behavior of glutathione is studied by the method of cyclic voltammetry using graphite electrodes modified with gold nanoparticles and gold microdeposit. When using gold nanoparticles as a modifier at  $E = 0.05$  V in strong alkaline medium the height of the "inverse" maximum on the cathodic branch of cyclic voltamperogram increases with increasing concentration of glutathione. Linear dependence of the height of the "inverse" maximum on glutathione concentration in the solution in the range of 1 – 14 pmole/liter allowed us to develop a simple, sensitive and rapid technique of glutathione determination with a detection limit 0.7 pmole/liter.

**Keywords:** glutathione; graphite electrode; gold nanoparticles; voltammetry.

UDC 539.26:519.21

**Recovery of the Orientation Distribution Function of MA2-1PCh Magnesium Alloy Subjected to Equal Channel Angular Pressing**

*Ivanova T. M., Serebryany V. N.*

Recovery of the orientation distribution function (ODF) using approximation by circular normal distributions is considered for a magnesium sample. The incomplete unnormalized experimentally measured X-ray pole figures (PF) are used as initial data. The choice of method is attributed to the possibility of using the procedure for the materials with low

symmetry of the sample, uniqueness of the solution in the selected class of functions and tolerance to the errors of measuring pole figures.

**Keywords:** recovery of orientation distribution function using pole figures; equal channel angular pressing; existence and uniqueness of the solution.

UDC 54.03:548.4

### The Effect of High-Temperature Annealing on the Microhardness Anisotropy in Crystals of the Langanite ( $\text{La}_3\text{Ga}_5\text{SiO}_{14}$ ) Family

*Kugaenko O. M., Torshina E. S., Petrakov V. S., Buzanov O. A., Sakharov S. A.*

Microhardness of the single crystals from the family of lanthanum gallium silicate (trigonal symmetry class 32, space group P321) is studied by Knoop method. The microhardness anisotropy of the II kind (the microhardness of different crystallographic planes of the crystal) is observed on the crystallographic planes  $(11\bar{2}0)$ ,  $(01\bar{1}0)$ ,  $(0001)$ . The effect of high temperature annealing on the imprint recovery after indentation is studied by Knoop method. The influence of annealing atmosphere on the microhardness anisotropy of the II kind is observed for the main crystallographic planes  $(11\bar{2}0)$ ,  $(01\bar{1}0)$ ,  $(0001)$  of the single crystals of the langasite family.

**Keywords:** microhardness; microhardness anisotropy; Knoop method; mass transfer; single crystals of the langasite family; annealing in vacuum and in air; atomic-force microscopy (AFM).

UDC 53.082.6

### Measurement of the Thermal Diffusivity of Metals

*Shevchenko A. I.*

A method of measuring thermal diffusivity of the samples (round long rod) of metals and alloys upon pulsed heating is presented. The main purpose of the method is to increase the accuracy of measuring thermal diffusivity of metals and reduce time of measurements assessing the sample heating thermogram. We consider the parabolic heat conduction equation for a semi-infinite thin rod with insulated lateral surface. Two variants of the method are analyzed. The first one is based on the determination of the second space derivative of the temperature, and the second method is based on the use of the first time derivative of the temperature. The developed method can be used in metallurgy and foundry to measure the thermal diffusivity of metals.

**Keywords:** measurement; metal; thermal diffusivity; method of pulse heating.

UDC 620.179:331.821.004.64.003

### Technical Testing and Control in Estimation of Accident Risk

*Ivanov V. I., Kornilova A. V., Musatov V. V.*

Complex estimation of the industrial safety of dangerous objects suggests using of the failure risk index. Current procedures of risk evaluation do not consider technical condition of the object and extent of the object fault. We demonstrate a possibility and necessity of increased use of nondestructive testing (NDT) and technical diagnostics (TD) for estimation of the probability of the accident, consider the requirements to the performance of the risk analysis and chart the ways of developing the considered approaches. Introduction of the new requirements to NDT and TD attributed to the necessity of using quantitative NDT indicators, i.e., the reliable data on the size of defects and errors of measurements, and expediency of using the probabilistic methodology of the fracture mechanics is demonstrated for estimation of the real probability of the accident.

**Keywords:** risk of failure; risk of accident; probability; technical diagnostics; NDT; reliability.

UDC 620.179.6:539.43

### Comparative Analysis of the Surface Layer Effect on the Stress-Strain Characteristics of D16AT Alloy

*Zakharchenko K. V., Kapustin V. I.*

A method providing study of the effect of changes in the surface layer on the stress-strain characteristics and fatigue resistance of samples is considered and exemplified in the study of D16AT alloy. The strain diagrams obtained in the step-stress test provided estimation of strain resistance of structurally inhomogeneous samples in the extreme stress area. It has been experimentally proved that a cladding layer having the mechanical properties lower than the material of the substrate reduces the strain resistance, whereas the layer produced by vibration shock treatment increases the strain resistance of samples. Methodology of the study is presented.

**Keywords:** surface layer, elastoplastic deformation, fatigue strength.

UDC 620.168.37:531.781

### Determination of the Mechanical Stresses in Steel by Magnetoelastic Demagnetization

*Kulak S. M., Novikov V. F.*

Dependence of the strength of the stray magnetic field of the local remanent magnetization of steel  $\Delta H_r$  resulted from changes (removal and recovery) of the mechanical load on the value of the mechanical stress in steel sample is analyzed. A possibility of indirect measuring (assessment) of the mechanical stress in metal constructions using magnetic method of dosed decreasing of the mechanical stress  $\sigma$  to the desired value  $\Delta\sigma$  and recovery of the initial stress value. Physical basis of the method is A nonlinear dependence of the magnetoelastic demagnetization of steel  $\Delta M$  on the mechanical stress value  $\sigma$  and stress variation value  $\Delta\sigma$  forms a basis of the method. Possible practical realization of the method is discussed.

**Keywords:** ferromagnetic material; magnetoelastic demagnetization; local magnetization; mechanical stress; dosed load removal.

UDC 519.24

### Structure of Nonparametric Statistics (generalizing paper)

*A. I. Orlov*

Nonparametric statistics is one of the five points of growth in applied mathematical statistics. Despite the large number of publications on specific issues of nonparametric statistics, the internal structure of this research direction has remained undeveloped. The purpose of this article is to consider sectioning of nonparametric statistical and to classify investigations on nonparametric statistical methods proceeding from a concept of nonparametric statistics worked out in the course of existing scientific activity. Nonparametric statistics allows us to make statistical inference, in particular, to estimate the characteristics of the distribution and testing statistical hypotheses without, as a rule, weakly proven assumptions about the distribution function of samples included in a particular parametric family. For example, the widespread belief that the statistical data are often have the normal distribution. Meanwhile, analysis of the results of observations, in particular, measurement errors, always leads to the same conclusion that in most cases the actual distribution significantly differs from normal. Uncritical use of the hypothesis of normality often leads to significant errors, in such areas as rejection of outlying observation data (overshoots), statistical quality control, and in other cases. Therefore, it is advisable to use nonparametric methods, in which only weak requirements are imposed on the distribution function of the results of observations: only their continuity is usually assumed. Generalization of numerous studies allows us to state that nonparametric methods can be successfully used to solve almost the same number of tasks that parametric methods used previously. Certain statements encountered in the literature regarding the assertion that nonparametric methods have less power, or require larger sample sizes than parametric methods are incorrect. However, a number of unresolved problems still remain in nonparametric statistics, as in mathematical statistics as well.

**Keywords:** mathematical statistics; applied statistics; statistical methods; non-parametric statistics; estimation; hypothesis testing; rank tests; the statistics of non-numeric data.

UDC 519.24:543.429.23-42.062

### Verification of Distribution Normality and Independence of Measured Integral Intensity of Broad Groups of Signals in High-Resolution $^1\text{H}$ NMR Spectra

*Smirnov M. B.*

It is shown that in high-resolution  $^1\text{H}$  NMR for broad groups of signals at a rather high noise level the measurements of the integrated intensities in sequentially recorded spectra are independent. Distribution of the measurement errors with sufficient accuracy correspond to the normal law. However, at a low noise successive measurements performed during short periods of time are not independent. To obtain data suitable for statistical processing it is necessary to record spectra with an interval of more than 1 hour. Distribution of the measurement errors is bimodal regardless of the method of the baseline correction. Normal distribution law is only a rough approximation in that case.

**Keywords:**  $^1\text{H}$  NMR; distribution of the measurement errors; independent measurements; crude oil; lignin.