

ЖУРНАЛУ **85** ЛЕТ

ЗАВОДСКАЯ
ЛАБОРАТОРИЯ
ДИАГНОСТИКА МАТЕРИАЛОВ

1

часть II

2017

Я Н В А Р Ь

Основан в январе 1932 г.

СОДЕРЖАНИЕ

Адрес редакции:

119334 Москва, Ленинский пр-т, 49,
ИМЕТ им. А. А. Байкова,
редакция журнала
"Заводская лаборатория.
Диагностика материалов".
Тел./факс: (499) 135-62-75,
тел.: (499) 135-96-56
e-mail: zavlabor@imet.ac.ru
<http://www.zldm.ru>

Журнал включен в список изданий,
рекомендованных ВАК при защите
докторских диссертаций.

© ООО Издательство «ТЕСТ-ЗЛ», «Заводская
лаборатория. Диагностика материалов», 2017

Перепечатка материалов журнала
«Заводская лаборатория. Диагностика
материалов» допускается только
с письменного разрешения редакции.
При цитировании ссылка обязательна.

**ЗАВОДСКАЯ®
ЛАБОРАТОРИЯ**
ДИАГНОСТИКА МАТЕРИАЛОВ

Барановская В. Б., Болдырев И. В., Карпов Ю. А., Федотов П. С. Международные организации в области аналитической химии и участие России в их деятельности	7
Лабусов В. А., Гаранин В. Г., Зарубин И. А. Новые спектральные комплексы на основе анализаторов МАЭС	15
Пунышев А. А. О возможности снижения систематических и случайных погрешностей атомно-эмиссионного спектрального анализа с использованием многолинейчатой градуировки	20
Баладина Н. П., Захарова М. Л. Новые возможности применения трехфазной дуги и анализатора МАЭС для спектрального анализа горных пород	31
Пелевина Н. Г., Жарликова Т. Н., Герашенко Е. А. Опыт применения анализатора МАЭС для определения мышьяка, олова, сурьмы, таллия, галлия, германия и индия в рудах и продуктах их переработки	35
Зак А. А., Шабанова Е. В., Васильева И. Е. Новые возможности многоканального спектрометра «Колибри-2» при анализе геологических образцов	38
Лисиенко Д. Г., Домбровская М. А., Кубрина Е. Д. Синтез и испытание стандартного образца состава графитового коллектора микропримесей	45
Домбровская М. А., Лисиенко Д. Г., Гильмуллина Ч. Г., Кубрина Е. Д. Совершенствование атомно-эмиссионной методики анализа графитового коллектора микропримесей	51
Кошель Е. С., Барановская В. Б., Карпов Ю. А. Новые возможности дугового атомно-эмиссионного анализа оксида европия	54
Сафронова Н. С., Гришанцева Е. С., Гаранин В. Г., Федорова Л. П. Атомно-эмиссионное определение редкоземельных и редких элементов в эколого-геологических объектах с использованием анализатора МАЭС	57
Дробышев А. И., Рядчикова Н. А., Савинов С. С. Атомно-эмиссионное спектральное определение микроэлементов в волосах человека с использованием МАЭС	63
Буренко Н. Д. Опыт применения спектрометрических комплексов «Экспресс-Ойл» в лабораториях ОАО «РЖД»	68
Степановских В. В. Стандартные образцы чугуна и стали для спектрального анализа, разработанные ЗАО «Институт стандартных образцов».	70
Троицкий Д. Ю., Медведев Н. С., Сапрыкин А. И. Возможности установки искрового пробоотбора для анализа металлических образцов методом атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой	77
Куропятник И. Н. Метрологические характеристики вакуумного атомно-эмиссионного спектрометра «Гранд-Эксперт»	81
Заксас Н. П., Веряскин А. Ф., Лабусов В. А. Возможности двухструйной дуговой плазмы для прямого анализа проб разной природы	86
Черевко А. С., Морозова А. А. Исследование матричных помех в атомно-эмиссионной спектрометрии с дуговым аргоновым двухструйным плазматроном	90
Шаевкин А. С., Купцов А. В., Заякина С. Б., Аношин Г. Н. Сравнение результатов сцинтилляционного атомно-эмиссионного анализа, полученных с использованием установки «Поток» и дугового двухструйного плазматрона «Факел».	97
Дзюба А. А., Лабусов В. А., Васильева И. Е., Шабанова Е. В. Светосила и спектральное разрешение спектрометров «Гранд», «Гранд-1500» и СТЭ-1	101
Бабин С. А., Лабусов В. А., Селюнин Д. О., Дзюба А. А. Анализаторы МАЭС для спектрометра «Гранд» с улучшенными характеристиками в области 258–269 нм.	105
Пелипасов О. В., Пустьмаков А. Н., Чернов К. Н., Бурумов И. Д., Селюнин Д. О., Боровиков В. М. Спектрометр с источником микроволновой плазмы для одновременного многоэлементного атомно-эмиссионного анализа растворов	108
Зарубин И. А. Возможности малогабаритного спектрометра «Колибри-2» в атомно-эмиссионном спектральном анализе	114
Семенов З. В., Лабусов В. А., Зарубин И. А., Эрг Г. В. Применение многослойных диэлектрических покрытий для подавления излучения нерабочих порядков спектра в спектрометрах с дифракционной решеткой	117
Бокк Д. Н., Лабусов В. А., Зарубин И. А., Гаранин В. Г. Однолинзовая система освещения входной щели вакуумного спектрометра «Гранд-Эксперт»	122
Ращенко В. В. Спектроаналитические генераторы «Шаровая молния». Модернизация и расширение ассортимента приборов	127
Семенов З. В., Вашенко П. В., Лабусов В. А., Неклюдов О. А., Болдова С. С. Алгоритм расчета формы фона в последовательности атомно-абсорбционных спектров с непрерывным источником излучения	129
Панкратов С. В., Лабусов В. А. Оценка содержания элементов при автоматическом качественном анализе вещества с использованием функции кросс-корреляции	132
Семенов З. В., Неклюдов О. А., Гаранин В. Г. Новые модули «Экспорт спектров» и «Информация из спектра» программы «Атом»	138

CONTENTS

Baranovskaya V. B., Boldyrev I. V., Karpov Yu. A., and Fedotov P. S. International Institutions in the Field of Analytical Chemistry and Russian Participation in Their Activities	7
Labusov V. A., Garanin V. G., and Zarubin I. A. New Spectral Systems Based on MAES Analyzers	15
Pupyshev A. A. On the Possibility of Reducing the Systematic and Random Errors of Atomic Emission Spectral Analysis Using Multiline Calibration	20
Balandina N. P. and Zakharova M. L. New Applications of a Three-Phase Arc and a MAES Analyzer for Spectral Analysis of Rocks	31
Pelevina N. G., Zharlikova T. N., and Gerashchenko E. A. Experience of Using MAES Analyzers in Determination of Arsenic, Tin, Antimony, Thallium, Gallium, Germanium, and Indium in Ores and Products of Their Processing	35
Zak A. A., Shabanova E. V., and Vasil'eva I. E. New Capabilities of Multichannel Spectrometer "Kolibri-2" for Analysis of Geological Samples	38
Lisienko D. G., Dombrovskaya M. A., and Kubrina E. D. Synthesis and Testing of the Reference Material of Graphite Collector of Microimpurities	45
Dombrovskaya M. A., Lisienko D. G., Gil'mullina Ch. G., and Kubrina E. D. Improvement of the Atomic-Emission Method for Analyzing Graphite Collector of Trace Impurities	51
Koshel' E. S., Baranovskaya V. B., and Karpov Yu. A. New Opportunities for Arc Atomic-Emission Analysis of Europium Oxide	54
Safronova N. S., Grishantseva E. S., Garanin V. G., and Fedorova L. P. Atomic Emission Determination of Rare Earth and Trace Elements in Ecological and Geological Samples Using MAES Analyzer	57
Drobyshev A. I., Ryadchikova N. A., and Savinov S. S. Atomic Emission Spectral Determination of Microelements in Human Hair Using MAES	63
Burenko N. D. Experience of Using Universal Spectrometer "Express-Oil" in Chemical and Technical Laboratories of the JSC "Russian Railways"	68
Stepanovskikh V. V. Standard Cast-Iron and Steel Samples for Spectral Analysis Developed at JSC "Institute for Certified Reference Materials"	70
Troitskiy D. Yu., Medvedev N. S., and Saprykin A. I. Analytical Capabilities of Spark Sampling Device for ICP-AES Analysis of Metal Samples	77
Kuropyatnik I. N. Metrological Characteristics of a "Grand-Expert" Vacuum Atomic Emission Spectrometer	81
Zaksas N. P., Verjaskin A. F., and Labusov V. A. Assessment of the Capabilities of Using a Two-Jet Arc Plasma for Direct Analysis of the Samples of Different Nature	86
Cherevko A. S. and Morozova A. A. Study of Matrix Interference in Atomic Emission Spectrometry with a Two-Jet Arc Argon Plasmatron	90
Shavekin A. S., Kuptsov A. V., Zayakina S. B., and Anoshin G. N. Comparison of the Results of Scintillation Atomic Emission Analysis Obtained on Spectral System "Potok" and a Two-Jet Arc Plasmatron "Fakel"	97
Dzyuba A. A., Labusov V. A., Vasil'eva I. E., and Shabanova E. V. Luminosity and Spectral Resolution of "Grand," "Grand-1500," and STE-1 Spectrometers	101
Babin S. A., Labusov V. A., Selyunin D. O., and Dzyuba A. A. MAES Analyzers for a Grand Spectrometer with Improved Performance in the Range of 258–269 nm	105
Pelipasov O. V., Put'makov A. N., Chernov K. N., Burumov I. D., Selyunin D. O., and Borovikov V. M. Microwave Plasma Spectrometer for Simultaneous Multielement Atomic-Emission Analysis of Solutions	108
Zarubin I. A. Capabilities of a Compact Kolibri-2 Spectrometer in Atomic Emission Spectral Analysis	114
Semenov Z. V., Labusov V. A., Zarubin I. A., and Erg G. V. Application of Multilayer Dielectric Coatings for Suppression of Non-Working Spectrum Orders in Diffraction Grating Spectrometers	117
Bock D. N., Labusov V. A., Zarubin I. A., and Garanin V. G. Single-Lens Entrance Slit Illumination System for a "Grand-Expert" Vacuum Spectrometer	122
Rashchenko V. V. Spectroanalytical Generators "Sharovaya Molniya": Upgrading and Diversification	127
Semenov Z. V., Vaschenko P. V., Labusov V. A., Neklyudov O. A., and Boldova S. S. Algorithm of Background Shape Calculation in a Sequence of Atomic-Absorption Spectra with a Continuous Radiation Source	129
Pankratov S. V. and Labusov V. A. Evaluation of the Element Content in an Automatic Qualitative Analysis of Substances Using a Cross-Correlation Function	132
Semenov Z. V., Neklyudov O. A., and Garanin V. G. New Modules "Spectrum Export" and "Information from Spectrum" Developed for "Atom" Software	138

ABSTRACTS

International Institutions in the Field of Analytical Chemistry and Russian Participation in Their Activities

Baranovskaya V. B., Boldyrev I. V., Karpov Yu. A., and Fedotov P. S.

Basic information about the structure, functions and interaction of international institutions in the field of analytical chemistry is presented including the role of Russian representatives in the activity of Eurachem, CITAC, ILAC-APLAC, and IUPAC. Attention is paid to the regulatory and methodological international documents of Eurachem used by Russian analysts and metrologists. Novel publications (in Russian language) regarding inter-laboratory quality control, validation of the methods (techniques) and evaluation of the uncertainty of analytical results are presented. Information regarding the requirements of international accreditation system ILAC is summarized. A fundamental role of IUPAC in the development of chemical nomenclature, terminology and standardization in the field of chemical research is shown. An emphasis is made on the necessity and efficiency of Russian participation in the work of international institution in the field of analytical chemistry. The necessary of further expansion and promotion of scientific and technical cooperation of Russia with the world scientific community is demonstrated.

Keywords: international institutions; accreditation; metrology; analytical chemistry.

UDC 543.423

New Spectral Systems Based on MAES Analyzers

Labusov V. A., Garanin V. G., and Zarubin I. A.

We present a review of the spectral systems developed by "VMK-Opto-elektronika" company for atomic-emission spectral analysis: Grand Globula, Grand Kristall, and Express intended for direct atomic-emission spectral analysis of solid metal and powder samples; Grand-Potok desired for rapid analysis of powders by the spill-injection method; Grand-Expert and Favorit for rapid analysis of metals and alloys; spark sampling systems Aspekt for analysis of metal samples using ISP-AES and ISP-MS; Express-Oil for direct analysis of oils; and Pavlin for rapid determination of sodium, lithium, potassium, calcium, barium, cesium, and rubidium in solutions. The design of the spectrometers and performance data are presented.

Keywords: atomic emission spectrometry; spectral systems; spectrometers; spectrum analyzers; multi-element solid-state detectors; detector arrays; spectrum excitation sources.

UDC 543.423.1

On the Possibility of Reducing the Systematic and Random Errors of Atomic Emission Spectral Analysis Using Multiline Calibration

Pupyshev A. A.

The main systematic and random errors of atomic emission spectral analysis and various procedures used for their reduction are considered: classical internal standard approach, non-spectral matrix effects, the use of several spectral lines of the analyte and internal standard with and without weight coefficients, signal drift, instability of sample injection and conditions of spectra excitation. The necessity of incorporating those methods into the software of the devices intended for atomic emission spectral analysis with various sources of spectrum excitation, including inductively coupled plasma is demonstrated. An algorithm and stand-alone program for optimization of the calibration curve based on multiline recording of the spectral lines of matrix elements and analyte, internal standards, solvent, and discharge atmosphere, capable of implementing the above methods of error reduction both in calibration and analysis are developed.

Keywords: atomic emission spectroscopy; systematic and random errors of analysis; internal standard; non-spectral interference; multi-signal calibration.

UDC 550.4.08

New Applications of a Three-Phase Arc and a MAES Analyzer for Spectral Analysis of Rocks

Balandina N. P. and Zakharova M. L.

The results of using a multi-channel analyzer of emission spectra (MAES) and "Atom" software for multielement atomic-emission spectral analysis of rocks using spill-injection method are presented. Replacing of the visual interpretation of spectra by high-quality digital processing significantly in-

increases the performance of the analysis, extends the range of detectable concentrations, and improves the metrological characteristics of determination of micro and macro components of rocks.

Keywords: atomic emission spectroscopy; three-phase arc; spill-injection method; multichannel emission spectrum analyzer.

UDC 543.423

Experience of Using MAES Analyzers in Determination of Arsenic, Tin, Antimony, Thallium, Gallium, Germanium, and Indium in Ores and Products of Their Processing

Pelevina N. G., Zharlikova T. N., and Gerashchenko E. A.

The advantages of atomic-emission determination (validated for photoelectric spectra recording) of arsenic, tin, antimony, thallium, gallium, germanium, and indium in rare metal ores and products of their processing are demonstrated. Replacement of the spectrum recording system of a DFS-8-3 diffraction spectrograph by MAES analyzer transforms it into a modern spectrometer capable of rising labor efficiency of the analytical laboratory.

Keywords: atomic emission spectroscopy; MAES; rare and trace elements; ores and products of their processing.

UDC 543:(225+423.1)

New Capabilities of Multichannel Spectrometer "Kolibri-2" for Analysis of Geological Samples

Zak A. A., Shabanova E. V., and Vasil'eva I. E.

A method of sample preparation is developed for geological samples of various compositions to provide simultaneous determination of cesium and other alkali metals (Na, K, Li and Rb) on a multichannel spectrometer "Kolibri-2" using flame photometry. The lower limit of cesium determination is close to its average abundance. The procedures of sample preparation and calibrations using reference material standards (RMS) of rocks, soils, sediments and coal ash provided the following ranges of the determinable contents (% wt.): Na 0.025 – 7.4; K 0.085 – 15.0; Li 0.00025 – 0.33; Rb 0.0005 – 0.15; Cs 0.0002 – 0.0070. The accuracy of the results for cesium is confirmed in analysis of RMS and spiking tests. The reduction of time spent for each stage of analysis compared to existing techniques increases the efficiency of the proposed flame photometric technique of determination of alkali metals on a spectrometer "Kolibri-2" and diminishes the time of the classical silicate analysis.

Keywords: flame photometry; spectrometer "Kolibri-2"; cesium; alkali metals; geological samples.

UDC 543.089.68 : 661.666

Synthesis and Testing of the Reference Material of Graphite Collector of Microimpurities

Lisienko D. G., Dombrovskaya M. A., and Kubrina E. D.

Synthesis features of a set of six samples of the reference material of the new type of graphite collector of microimpurities SOG-30 are discussed. The results of analysis of the matrix composition (high purity graphite HP-8-4), degree of unhomogeneity, stability, and mutual consistency of the samples are presented. The results of testing SOG-30 carried out in Federal State Unitary Enterprise "UNIIM" using state secondary standard GUAT 196-1-2012 by the method of mass spectrometry with inductively coupled plasma (mass-spectrometer NexION-300D) are presented.

Keywords: reference material standard; graphite collector; material synthesis; testing.

UDC 543.42:661.666

Improvement of the Atomic-Emission Method for Analyzing Graphite Collector of Trace Impurities

Dombrovskaya M. A., Lisienko D. G., Gil'mullina Ch. G., and Kubrina E. D.

Conditions for atomic emission spectroscopic determination of trace impurities in graphite powder using fractional distillation in arc AC discharge are optimized. A PGS-2 spectrometer with MAES analyzer are used for spectra recording. The effect of the discharge current, composition and concentration of buffering agents, as well as sample dispensing process on the results of analysis is studied.

Keywords: graphite collector of trace impurities; atomic emission spectral analysis; fractional distillation.

UDC 543.423

New Opportunities for Arc Atomic-Emission Analysis of Europium Oxide

Koshel' E. S., Baranovskaya V. B., and Karpov Yu. A.

A possibility of direct arc atomic emission analysis of europium oxide is analyzed using a high-resolution spectrometer "Grand Extra" ("VMK-Optoelektronika," Russia). The curves of burning-out the impurities in the presence of different carriers are studied. We have optimized conditions of the analysis and parameters of the spectrometer. The developed advanced techniques of arc atomic emission analysis of europium oxide with improved (compared to standard procedure) metrological characteristics provides determination of the rare earth impurities in the concentration range of $3 \times 10^{-6} - 1 \times 10^{-1}$ % wt.

Keywords: atomic-emission analysis; arc excitation source; rare earth metals; europium; multichannel analyzers of emission spectra (MAES).

UDC 543.423, 550.4, 502.65(282.247.411.6)

Atomic Emission Determination of Rare Earth and Trace Elements in Ecological and Geological Samples Using MAES Analyzer

Safronova N. S., Grishantseva E. S., Garanin V. G., and Fedorova L. P.

A method of direct simultaneous determination of rare earth (Y, La, Ce, Nd, Sm, Gd, Tb, Ho, Er, Yb, Sc) and trace elements (V, Cr, Ga, Rb, Sr, Zr, Nb, Ag, In, W, Pb), Fe and Mn in sediments and ash of aquatic vegetations of Ivan'kovo Reservoir using atomic emission spectrometry with ac arc discharge is developed. MAES spectrum analyzer ("VMK-Optoelektronika") and software package "Atom 3.2" are used with a high-resolution (0.003 nm) diffraction spectrograph DFS-13-2 (flat grating 1200 lines/mm), thus providing determination of 11 from 17 rareearth elements within an optical range of 402.71 – 452.64 nm. The detection limits of the method range within 1 – 3 mg/kg, depending on the element. Spectral overlapping of the molecular bands of CN and analytical lines of Pr, Eu, Dy, Tm, Lu renders determination of these elements impossible.

Keywords: atomic-emission spectral method; multichannel spectrum analyzer (MAES); rare-earth elements; rare elements; bottom sediments; aquatic vegetation.

UDC 543.423.1

Atomic Emission Spectral Determination of Microelements in Human Hair Using MAES

Drobyshev A. I., Ryadchikova N. A., and Savinov S. S.

Biomonitoring of essential and toxic elements present in human is an urgent task of modern medicine. In that context, human hair is a promising matrix for the analysis. We present the results of atomic emission spectral analysis of hair mineralizates (obtained after acid digestion) with excitation of a dried residue spectrum from the end of carbon electrode in ac arc. Spectrum recording was carried out using a MFS-8 spectral device modernized by linear photodiode array MAES. The developed technique is used for determination of Al, B, Ca, Cu, Mg, Mn, Fe, P, Pb, and Zn in 42 different hair samples. Using the obtained data we derived a law of the element distribution in the sample and compare the results with the literature data. Statistically significant influence of the donor gender on the content of several elements in human hair is revealed.

Keywords: atomic emission spectrometry; dried residue; hair; microelements; average concentrations; subpopulation factors.

Experience of Using Universal Spectrometer "Express-Oil" in Chemical and Technical Laboratories of the JSC "Russian Railways"

Burenko N. D.

Experience of using a universal spectrometer "Express-Oil" (developed by "VMK-Optoelektronika") in combination with a set of devices available in railway chemical and technical laboratories and used for spectral analysis of oils, lubricants and alloys is described. A complete specification and advantages of the spectrometer are presented are considered.

Keywords: fuel/diesel oil; abrasive wear; journal-box; railway laboratory; "Express-Oil"; spectrometer; stirrer.

UDC 006.9:53.089.68:543.42:669.14

Standard Cast-Iron and Steel Samples for Spectral Analysis Developed at Institute for Certified Reference Materials*Stepanovskikh V. V.*

Current state and prospects of developing reference standards in the present context are considered. Metrological characteristics of the reference specimens of cast iron and steel developed at Institute for Certified Reference Materials (ICRM) over the last 5 years and certified for spectrometric analysis are presented.

Keywords: standard sample of cast-iron; standard sample of steel; testing of standard samples; spectral analysis.

UDC 543.423.1

Analytical Capabilities of Spark Sampling Device for ICP-AES Analysis of Metal Samples*Troitskiy D. Yu., Medvedev N. S., and Saprykin A. I.*

The analytical performance of a spark sampling device "Aspect" ("VMK-Optoelektronika") is evaluated for steel analysis by atomic emission spectrometry with inductively coupled plasma (ICP-AES). The results of studying the effect of pulse duration, frequency and current of the spark discharge on the character of sample erosion, size and quantity of aerosol particles are presented. The dependences of the analytical signal on the spark duration are obtained and the optimal duration of preliminary sparking is specified. The limit of Cr, Cu, Mn, Mo, Ni, P, Si, and V detection ranges from $n \times 10^{-5}$ to $n \times 10^{-2}$ % wt. The accuracy of the ICP-AES analysis with a spark sampling using standard reference sample showed the good agreement between certified and determined values of analyte concentration.

Keywords: atomic emission spectrometry with inductively coupled plasma (ICP-AES); spark sampling; analysis of steels and alloys.

UDC 543.423

Metrological Characteristics of a "Grand-Expert" Vacuum Atomic Emission Spectrometer*Kuroyatnik I. N.*

The results of studying the repeatability and stability of quantitative determination of different chemical elements in steels on a "Grand-Expert" vacuum atomic emission spectrometer (time scale from 10 min up to 3 months) are presented. The validity of the method is estimated from analysis of the calibration characteristics. It is shown that a vacuum atomic emission spectrometer ("Grand-Expert") provides a reliable determination of alloying elements in steels with a relative error of 1–2 %.

Keywords: atomic emission spectral analysis; steel; multichannel analyzer of emission spectra; MAES.

UDC 543.423

Assessment of the Capabilities of Using a Two-Jet Arc Plasma for Direct Analysis of the Samples of Different Nature*Zaksas N. P., Verjaskin A. F., and Labusov V. A.*

An expediency of application of two-jet arc plasma to analytical problems, which are not easily solved by other spectral, is shown. Atomic emission techniques for analysis of brown coal and protein complex compounds of small weight, both direct and after thermal treatment of the sample, are suggested using reference samples based on graphite powder and simple sample preparation procedure of dilution of the sample by a spectroscopic buffer. Owing to "ATOM" software which provides spectrum registration of each basic exposure, a procedure of qualitative determination of the element inclusions in diamond particles (weight less than 1 mg) is developed. Advantages of this method compared to other spectral methods used for solving specific analytical tasks are discussed. All the techniques described here are performed using the new plasmatron developed at the "VMK-Optoelektronika" Company.

Keywords: two-jet plasma; brown coal; protein; diamond; inclusions.

UDC 533.9

Study of Matrix Interference in Atomic Emission Spectrometry with a Two-Jet Arc Argon Plasmatron*Cherevko A. S. and Morozova A. A.*

The effects of Rb, K, Na, Ba, Sr, Ca, Ce, La, and Y matrix elements on the spectra excitation conditions in plasma flow of argon two-jet plasmatron (DDP) are studied. The dominant excitation mechanisms in atoms and ions of analyte with different energy structure in the presence of a particular

matrix are revealed. A new kind of matrix interference attributed to the low second ionization potential of matrix elements is found. Those elements, especially having low-lying thermal levels of double-charged ions, are shown to quench the population of argon excited state in plasma, thus decreasing the efficiency of ion emission excitation of the analyte through Penning ionization.

Keywords: two-jet arc argon plasmatron; matrix interference; electronic configuration; Penning ionization; charge transfer; ion-electron recombination.

UDC 543.423.1;543.07

Comparison of the Results of Scintillation Atomic Emission Analysis Obtained on Spectral System "Potok" and a Two-Jet Arc Plasmatron "Fakel"*Shavekin A. S., Kuptsov A. V., Zayakina S. B., and Anoshin G. N.*

Gold, palladium, and platinum contents are determined by scintillation atomic-emission spectral analysis of comparison standard specimens made of standard GSO KP-1 (Platinum Concentrate sample) using its serial dilution with a pure graphite powder. Two spectral systems ("Grand-Potok" and "Grand Fakel") which differ only in the source of spectrum excitation were used: "Potok" (an electric arc device for analysis of powder samples by the spill-injection method) and "Fakel" (a two-jet arc plasmatron). It is shown that the detection limits of noble metals determined on a "Grand Fakel" spectral system are lower than those obtained on the "Grand Potok" spectral system: 56 times lower for gold, 11 times for palladium, and 77 times for platinum. The measured plasma temperature in the sources was 5346 K ("Potok") and 7478 K ("Fakel"). It is shown that increase in the plasma temperature entails significant 10-fold increase in the spectral line intensity of the analytes which determines the advantage of a two-jet arc plasmatron in the detection of noble metals.

Keywords: scintillation atomic emission spectral analysis; two-jet arc plasmatron; multichannel spectrometers; MAES analyzer.

UDC 543.423

Luminosity and Spectral Resolution of "Grand," "Grand-1500," and STE-1 Spectrometers*Dzyuba A. A., Labusov V. A.,**Vasil'eva I. E., and Shabanova E. V.*

We present the results of measuring the luminosity, inverse linear dispersion, and spectral resolution in the range of 235–344 nm for Grand, Grand-1500, and STE-1 spectrometers operating in combination with an electric-arc unit ("Potok") intended for analysis of powder samples by spill-injection method and being a part of a scintillation atomic emission facility. The resolution of the Grand-1500 spectrometer is 1.5–2 times higher than that of other spectrometers thus considered. In the fifth diffraction order the resolutions of Grand and STE-1 spectrometers are almost the same while in the fourth diffraction order the resolution of Grand spectrometer is significantly better than that of STE-1. The luminosity of Grand spectrometer in the range of 240–270 nm is by an order of magnitude higher than that of STE-1, however, in the vicinity of 342 nm the advantage decreases up to 4-fold difference. Compared to the luminosity of STE-1, the luminosity of the Grand-1500 spectrometer being slightly higher near 240 nm, becomes 2 and 5 times lower at 267 nm and 342 nm, respectively.

Keywords: MAES; spectrometer; arc atomic emission spectrometry; spectral resolution; luminosity.

UDC 543.423

MAES Analyzers for a Grand Spectrometer with Improved Performance in the Range of 258–269 nm*Babin S. A., Labusov V. A.,**Selyunin D. O., and Dzyuba A. A.*

A multichannel atomic emission spectrum analyzer MAES is developed for a spectrometer "Grand" with a hybrid assembly of 12 BLPP-369M1 photodetector arrays (recorded range 190–350 nm) and one BLPP-2000 array (recorded range 258–269 nm). The minimum exposure time is 3 msec. It is shown that at a basic exposure time of 3 msec the signal-to-noise ratio of Au 267.595 nm analytical spectral line recorded by a BLPP-2000 array is 10-fold higher on average than that recorded by a BLPP 369M1 array.

Keywords: time-resolved spectroscopy; atomic emission spectrometry; powdered geological samples; scintillation; multi-element solid-state radiation detectors; detection limit reduction.

UDC 543.07:543.423

Microwave Plasma Spectrometer for Simultaneous Multielement Atomic-Emission Analysis of Solutions*Pelipasov O. V., Put'makov A. N., Chernov K. N., Burumov I. D., Selyunin D. O., and Borovikov V. M.*

A design and principle of operation of a microwave plasma spectrometer for elemental analysis of inorganic and organic solutions are presented. The spectrometer operates at a frequency of 2.45 GHz in the atmosphere of both atomic and molecular gases. The plasma is sustained by TEM₁₀ mode field. The detection limits of the spectrometer (with argon as a plasma forming agent) attain 10 µg/liter for most of the elements.

Keywords: atomic emission spectrometry; microwave plasma; spectrum excitation source; magnetron; resonator; spectrometer; analytical characteristics.

UDC 543.423:543.08

Capabilities of a Compact Kolibri-2 Spectrometer in Atomic Emission Spectral Analysis*Zarubin I. A.*

The main characteristics of a compact Kolibri-2 spectrometer are presented. The expediency of using the spectrometer in atomic-emission analysis for expanding the range of detectable concentrations and extending a line of the elements to be determined due to simultaneous determination of the alkali and alkaline earth elements using flame photometry and spark sampling devices is demonstrated.

Keywords: atomic emission spectroscopy; multichannel spectrometer; flame photometry; linear photodiode array.

UDC 543.4:539.23;681.785.557

Application of Multilayer Dielectric Coatings for Suppression of Non-Working Spectrum Orders in Diffraction Grating Spectrometers*Semenov Z. V., Labusov V. A., Zarubin I. A., and Erg G. V.*

The calculation of the combined optical filter configuration for suppression (reflection) of the non-working spectrum orders in diffraction grating spectrometers is presented. Combined filter consists of a substrate with deposited dichroic filters based on multilayer optical coatings. The calculation takes into account the acceptable width of spectral transitions in the filters transmission spectra and dichroic filters positioning accuracy on the substrate. A filter was designed and manufactured for suppression of the non-working diffraction spectrum orders of small-sized spectrometer "Kolibri-2" with operating spectral range of 190 – 1100 nm. The filter's degree of intensity suppression of non-working orders is at least 2×10^4 times. Artifacts observed in the spectrum are attributed to the light refraction at the edges of the multilayer coatings. A method providing reduction of the aforementioned negative effect is developed.

Keywords: order filters; dichroic filter; multilayer dielectric coatings; deposition control; spectrophotometry; thin films.

UDC 543.423

Single-Lens Entrance Slit Illumination System for a "Grand-Expert" Vacuum Spectrometer*Bock D. N., Labusov V. A., Zarubin I. A., and Garanin V. G.*

The advantages of a single-lens slit illumination system of a "Grand-Expert" vacuum spectrometer compared to a three-lens system are considered. It is shown that the analytical gap can be chosen using a diaphragm located near the excitation source. Operating range of the residual gas pressure in the vacuum case of the device has been determined experimentally. A possibility of expanding the spectral band of "Grand-Expert" in the vacuum ultraviolet region up to 130 nm is demonstrated.

Keywords: optical emission spectroscopy (OES); analysis of steels and alloys; spectrum analyzers; multi-element solid-state detectors; "Grand-Expert" spectrometer.

UDC 543.423

Spectroanalytical Generators "Sharovaya Molniya": Upgrading and Diversification*Rashchenko V. V.*

A brief overview of the new electric arc and spark discharge generators of ShM series ("Sharovaya Molniya") developed at the "VMK-Optoelektro-

nika company" over a period of 2011 – 2016 and designed for atomic-emission spectral analysis is presented. A number of hardware changes that improve characteristics of existing generators are considered. In addition to well-known ShM-40 and ShM-250 generators, lightweight modifications of ShM generators have been developed and manufactured (mainly for the laboratories of educational institutions). "Sharm" arc generators have the same parameters as ShM-40 with an arc current of up to 20 A. "SKAT" spark generators forms a spark discharge of a positive polarity with the parameters similar to those of ShM-250. ShM-20 arc generators form an ac arc discharge (frequency 25 kHz) and dc arc discharge with a current up to 20 A. The generator control system has been upgraded using advanced microcontrollers to extend the capabilities of the devices and improve their performance.

Keywords: atomic emission spectrometry; generator; arc discharge; spark discharge; generator "Sharovaya Molniya."

UDC 543.421

Algorithm of Background Shape Calculation in a Sequence of Atomic-Absorption Spectra with a Continuous Radiation Source*Semenov Z. V., Vaschenko P. V., Labusov V. A.,**Neklyudov O. A., and Boldova S. S.*

An algorithm of mathematical processing of the sequence of atomic-absorption spectra in time intended to be used for background shape calculation is presented. Each spectrum of the sequence considered in terms of the optical density is approximated by Savitzky – Golay algorithm (polynomial approximation with a moving window) after removal of the absorption lines. Removal of the absorption lines is done through determination of the line position by the inflection points, evaluation of the amplitudes and removal of the lines up to the local minima. The algorithm is implemented as a load library for "Atom" software.

Keywords: absorption; time-resolved spectroscopy; algorithm of spectra processing; atomic-absorption spectrometry.

UDC 543.423:543.08

Evaluation of the Element Content in an Automatic Qualitative Analysis of Substances Using a Cross-Correlation Function*Pankratov S. V. and Labusov V. A.*

A method and tools for automated evaluation of the element content in a sample using the only one sample spectrum is presented. The presence of elements in the sample is determined by qualitative cross-correlation analysis with a low probability of false alarm and high performance. The element content is calculated by modified spectral-line detection and enhancement method and adjusted to use in modern automated systems. The tool kit requires tuning to a particular spectral system using previously obtained results of quantitative spectral analysis for this system. The efficiency of the procedure is exemplified in evaluation of the content of matrix elements of geological powder samples of Si, Ti, Al, Mg, Fe, Ca, and Mn using two different analytical spectral systems.

Keywords: cross-correlation function; qualitative analysis; content evaluation; atomic emission spectra; multi-element solid-state detectors.

UDC 543.423:543.421:543.08

New Modules "Spectrum Export" and "Information from Spectrum" Developed for "Atom" Software*Semenov Z. V., Neklyudov O. A., and Garanin V. G.*

New software modules developed for "Atom" software are presented: module for spectral data export to divers file formats and module for operational visualization of the information attached to spectra of analytical table. Export module supports export of the integral spectra and time resolution spectra to external files thus allowing processing spectral data gained using a MAES analyzer in external software such as spreadsheet editors, software for numerical calculation, simulation, and mathematical processing. This is required, in particular, for developing new algorithms of spectrum signal processing. Information visualization module allows specialist to display information attached to the storage of spectra in analytical table in the form of columns, thereby increasing the efficiency and usability of the "Atom" software.

Keywords: atomic-emission spectrometry; absorption spectrometry; data export; storage of spectral information.