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ЗАВОДСКАЯ ЛАБОРАТОРИЯ ДИАГНОСТИКА МАТЕРИАЛОВ

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ДИАГНОСТИКА МАТЕРИАЛОВ

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

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СОДЕРЖАНИЕ

Карпов Ю. А., Болдырев И. В., Барановская В. Б. Аккредитация аналитических лабораторий (прошлое, настоящее, будущее) — ожидания и проблемы	7
Лабусов В. А. Приборы и комплексы компании «ВМК-Оптоэлектроника» для атомно-эмиссионного спектрального анализа. Современное состояние	12
Васильева И. Е., Шабанова Е. В., Забанов Ю. А., Бусько А. Е. Применение МАЭС для исследования вещества стандартных образцов состава природных и техногенных сред	22
Баладина Н. П., Захарова М. Л. Методика современного приближенно-количественного эмиссионного спектрального анализа геологических объектов	29
Малюгин М. С., Гребининовская Л. В. Применение установки «Поток» и анализатора МАЭС при поисковых работах на золото	35
Заякина С. Б., Леснов Ф. П., Аношин Г. Н., Балухтин А. В. Атомно-эмиссионное определение благородных металлов в пробах из гидротерм вблизи вулканов Курильских островов с использованием комплекса «Гранд-Поток»	38
Черевко А. С. Исследование степени нарушения локального термодинамического равновесия в аналитической зоне дугового двухструйного плазмотрона	42
Заксас Н. П., Веряскин А. Ф., Лабусов В. А. Аналитические зоны двухструйной дуговой плазмы: достоинства и ограничения	48
Купцов А. В., Заякина С. Б., Сапрыкин А. И. Изучение распределения температуры и интенсивностей спектральных линий аналитов по высоте плазменного факела дугового двухструйного плазмотрона	52
Гаранин В. Г., Неклюдов О. А. Новые возможности определения примесного состава металлов и порошковых проб с учетом фракционного поступления элементов в дуговой разряд	55
Домбровская М. А., Лисиенко Д. Г., Кубрина Е. Д., Казаков А. С., Александров Е. П. Валидация методики спектрального анализа иридия для нанопорошкового металла	62
Хамдеев М. И., Васильева О. Н., Чистяков В. М., Ерин Е. А. Атомно-эмиссионный спектральный анализ фосфатных концентратов продуктов деления и примесей, получаемых в процессе регенерации облученного ядерного топлива	65
Леухин С. Г., Кочергина Г. Р. Метрологическое обеспечение аттестованных смесей для определения микропримесей	69
Пивоварова О. А., Алексеева Е. А., Васильева Ю. Г. Практика применения спектроаналитического штатива «Кристалл»	72
Сарычева Н. А. Метрологическая оценка результатов спектрального анализа углеродистой стали, выполненного на вакуумных атомно-эмиссионных спектрометрах Polivac E980 и E983 с анализатором МАЭС и генератором «Шаровая молния 250»	75
Гаранин В. Г. Применение спектрометра «Гранд-Эксперт» для определения состава металлов и сплавов на основе магния, титана и алюминия	79
Куропятник И. Н. Использование вакуумного атомно-эмиссионного спектрометра «Гранд-Эксперт» для определения состава сталей	88
Бокс Д. Н., Лабусов В. А., Зарубин И. А. Определение неметаллических включений в металлических сплавах методом атомно-эмиссионной спектрометрии с искровым возбуждением	92
Шабанова Е. В., Зак А. А., Погудина Г. А., Хмелевская И. М., Меньшиков В. И. Использование спектрометра Колибри-2 для определения Na, K, Li и Rb в геохимических объектах	98
Путьмаков А. Н., Зарубин И. А., Бурумов И. Д., Селюнин Д. О. Спектрометр «Павлин» для атомно-эмиссионного спектрального анализа с атомизацией в пламени	105
Бабин С. А., Лабусов В. А., Селюнин Д. О., Дзюба А. А. Быстродействующие анализаторы МАЭС на основе линеек БЛПП-2000	108
Зарубин И. А., Лабусов В. А., Бокс Д. Н. Оптимальная система освещения входной щели многоканальных спектрометров «Гранд» и «Экспресс»	114
Путьмаков А. Н., Пелипасов О. В., Максимов А. Ю., Боровиков В. М., Чернов К. Н. Разработка источника СВЧ плазмы для атомно-эмиссионного спектрального анализа растворов	117
Гаранин В. Г., Неклюдов О. А., Петрович Д. В., Семёнов З. В., Панкратов С. В., Ващенко П. В. Программное обеспечение атомно-эмиссионного спектрального анализа. Программа «Атом»	121
Панкратов С. В., Лабусов В. А., Неклюдов О. А., Ващенко П. В. Автоматическая градуировка спектрометров с анализаторами МАЭС по длинам волн (профилирование)	128
Семёнов З. В., Лабусов В. А., Неклюдов О. А., Ващенко П. В. Алгоритм обработки последовательностей спектров для сканирующего атомно-эмиссионного спектрального анализа	135
Дробышев А. И., Савинов С. С. Дуговой атомно-эмиссионный цифровой спектрографический анализ жидких биопроб с использованием МАЭС	142
Отмахов В. И., Петрова Е. В., Шилова И. В., Батанина А. А., Кускова И. С., Рабцевич Е. С. Дуговой атомно-эмиссионный спектральный анализ лекарственных растений	145
Болдова С. С., Путьмаков А. Н., Лабусов В. А., Боровиков В. М., Селюнин Д. О., Бейзель Н. Ф., Гуськова Е. А. О создании прибора для одновременного многоэлементного атомно-абсорбционного спектрального анализа на основе спектрометра с высокой дисперсией и источника непрерывного спектра	148
Ващенко П. В., Болдова С. С., Лабусов В. А. Алгоритм обработки последовательностей атомно-абсорбционных спектров с непрерывным источником излучения	153
Медведев Н. С., Путьмаков А. Н., Шаверина А. В., Цыганкова А. Р., Сапрыкин А. И. Снижение пределов обнаружения примесей при анализе высокоочищенных веществ методом атомно-эмиссионной спектрометрии с индуктивно-связанной плазмой	157

Адрес редакции:

119991, Москва, ГСП-1,
Ленинский пр-т, 49,
ИМЕТ им. А. А. Байкова, Редакция
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Диагностика материалов».

Тел./факс: (499) 135-62-75,
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CONTENTS

ABSTRACTS

Karpov Yu. A., Boldyrev I. V., Baranovskaya V. B. Accreditation of Analytical Laboratories (Past, Present and Future) : Expectations and Concerns. 7

Labusov V. A. Devices and Systems for Atomic Emission Spectroscopy Produced by "VMK-Optoelektronika": State-of-the-Art 12

Vasil'eva I. E., Shabanova E. V., Zabanov Yu. A., Bus'ko A. E. The Use of MAES in the Study of Reference Standard Materials of Natural and Man-Made Composition 22

Balandina N. P., Zakharova M. L. Method of Approximate-Quantitative Emission Spectral Analysis of Geological Specimens 29

Malugin M. S., Grebinovskaya L. V. Application of the "Potok" Device and MAES Analyzer to Gold reconnaissance at the Baikal Branch of "Sosnovgeologiya" 35

Zayakina S. B., Lesnov F. P., Anoshin G. N., Balukhtin A. V. Capabilities of a "Grand-Potok" System for Atomic-Emission Determination of Noble Metals in the Samples from Hydrotherms near the Volcanoes of the Kuril Islands 38

Cherevko A. S. Study of the Degree of Violation of Local Thermodynamic Equilibrium in Analytical Zone of Two-Jet Plasmatron 42

Zaksas N. P., Veryaskin A. F., Labusov V. A. Analytical Zones of High-Power Two-Jet Plasma: Advantages and Limitations. 48

Kuptsov A. V., Zayakina S. B., Saprykin A. I. Study of the Temperature Distribution and Spectral Line Intensity of Noble Metals in the Height of the Plasma Plume of Two-Jets Plasma Torch 52

Garanin V. G., Nekludov O. A. New Program Possibilities of Time-Resolved Spectrometry with DC-Arc Source for Determination of Impurities in Metal and Powder Samples. 55

Dombrovskaya M. A., Lisienko D. G., Kubrina E. D., Kazakov A. S., Aleksandrov E. P. Validation of Iridium Spectral Analysis for Nanopowder Materials 62

Khamdeev M. I., Vasilieva O. N., Chistyakov V. M., Erin E. A. Atomic Emission Spectral Analysis of Phosphate Concentrates of Fission Products and Impurities Obtained upon Pyrochemical Reprocessing of the Irradiated Nuclear Fuel 65

Leukhin S. G., Kochergina G. R. Metrological Support of Certified Mixtures for the Analysis of Microimpurities. 69

Pivovarova O. A., Alekseeva E. A., Vasil'eva Yu. G. Practice of Using a Spectroanalytical Stand "Kristall". 72

Sarycheva N. A. Metrological Evaluation of the Results of Spectral Analysis of Carbon Steel Performed on Polivac E980 and E983 Atomic Emission Vacuum Spectrometers with a MAES Analyzer and a Sharovaya Molniya 250 Generators 75

Garanin V. G. Application of a «Grand-Ékspert» Spectrometer to Analysis of Metals and Alloys of Magnesium, Titanium and Aluminum Used in Aerospace Production 79

Kuropyatnik I. N. The Use of Vacuum Atomic Emission Spectrometer "Grand-Ékspert" for Determination of Steel Composition 88

Bock D. N., Labusov V. A., Zarubin I. A. Determination of Non-Metallic Inclusions in Metal Alloys by Spark Optical Emission Spectrometry. Shabanova E. V., Zak A. A., Pogudina G. A., Khmelevskaya I. M., Men'shikov V. I. The Use of a Kolibri-2 Spectrometer in Determination of Na, K, Li and Rb in Geochemical Objects 92

Put'makov A. N., Zarubin I. A., Burumov I. D., Selyunin D. O. "Pavlin" Spectrometer for Flame Atomic Emission Spectrometry 98

Babin S. A., Labusov V. A., Selyunin D. O., Dzyuba A. A. BLPP-2000 Array Based High-Speed Multichannel Analyzers of Atomic Emission Spectra. 105

Zarubin I. A., Labusov V. A., Bock D. N. Optimum System for Illuminating the Entrance Slit of Grand and Ékspress Multichannel Spectrometers. 108

Put'makov A. N., Pelipasov O. V., Maksimov A. Yu., Borovikov V. M., Chernov K. N. Development of a Microwave Plasma Source for Atomic Emission Spectral Analysis of Solutions 114

Garanin V. G., Nekludov O. A., Petrochenko D. V., Semenov Z. V., Pankratov S. V., Vashchenko P. V. Software for Atomic Emission Spectral Analysis. "Atom" Software 117

Pankratov S. V., Labusov V. A., Nekludov O. A., Vashchenko P. V. Automatic Wavelength Calibration of the Spectrometers with MAES Analyzers (Profiling) 121

Semenov Z. V., Labusov V. A., Nekludov O. A., Vashchenko P. V. Algorithm for Processing Sequences of Spectra for Scintillation Atomic Emission Spectral Analysis 128

Drobyshev A. I., Savinov S. S. Arc Atomic-Emission Digital Spectrography of Liquid Biosamples Using MAES 135

Otmakhov V. I., Petrova E. V., Shilova I. V., Batanina A. A., Kusko-va I. S., Rabtsevich E. S. Arc Atomic Emission Analysis of Medicinal Herbs 142

Boldova S. S., Put'makov A. N., Labusov V. A., Borovikov V. M., Selyunin D. O., Gus'kova E. A. On the Development of a Device for Simultaneous Multi-Element Atomic Absorption Spectral Analysis Based on a High-Dispersion Spectrometer and a continuous spectrum source 145

Vashchenko P. V., Boldova S. S., Labusov V. A. Algorithm for Processing Sequences of Atomic Absorption Spectra with a Continuous Radiation Source 148

Medvedev N. S., Put'makov A. N., Shaverina A. V., Tsygankova A. R., Saprykin A. I. Reduction of the Detection Limits in Trace Analysis of High Purity Substances by ICP-AES. 157

Accreditation of Analytical Laboratories (Past, Present and Future) : Expectations and Concerns
Karpov Yu. A., Boldyrev I. V., Baranovskaya V. B.
Stages of developing the national system of accreditation of analytical laboratories, advantages and shortcomings of the system, as well as topical issues regarding accreditation in the context of the Federal Law No. 412-FZ (2013.12.28) are considered.
Keywords: analytical laboratories; accreditation; national system of accreditation.

UDC 543.423
Devices and Systems for Atomic Emission Spectroscopy Produced by "VMK-Optoelektronika": State-of-the-Art
Labusov V. A.
A review of the devices and systems on their base developed and produced by "VMK-Optoelektronika": multichannel analyzers of emission spectra (MAES) based on multichip assemblies of linear solid-state detectors; Grand, Grand-Ékspert, Favorit, Ékspress, and Kolibri multichannel spectrometers; Vezuvii and Sharovaya Molniya electric-arc and spark-discharge generators; Globula and Kristall spectroanalytical instruments; Potok electric-arc facility for powder analysis using tspl-injection method, etc. are presented. Technical characteristics of the reviewed equipment are listed and discussed.
Keywords: atomic emission spectroscopy (AES); spectrometers; spectrum analyzers; multi-element solid-state detectors; detector arrays; spectrum excitation sources.

UDC 543.432+53.089.68
The Use of MAES in the Study of Reference Standard Materials of Natural and Man-Made Composition
Vasil'eva I. E., Shabanova E. V., Zabanov Yu. A., Bus'ko A. E.
Formation of the collection of multielement reference standard materials (RSM) of natural and man-made composition in the Vinogradov Institute of SB RAS is focussed on ensuring the uniformity of measurements in geoanalysis. The use of MAES in arc atomic-emission analysis substantially simplified the analysis of reference material thus providing data for certification and assessing the heterogeneity and representative mass of the substance upon determination of the certain elements, stability of the substance during prolonged storage; checking of various standard samples against each other. Examples of the effective use of arc atomic-emission analysis in studying RSMs of different composition and genesis are given.
Keywords: multielement certified reference materials natural and man-made specimens; MAES; arc atomic-emission analysis.

UDC 550.4.08
Method of Approximate-Quantitative Emission Spectral Analysis of Geological Specimens
Balandina N. P., Zakharova M. L.
The results of using a multichannel analyzer of the emission spectra (MAES) with «Atom 3» program intended for multielement atomic-emission analysis of rocks are presented. The curves of evaporation in the arc discharge are studied for some chemical elements obtained from the rocks samples of different composition. The replacement of the visual decryption by the high-quality digital spectra processing enabled us to increase the analysis performance, extend the range of the designated contents and significantly improve the metrological characteristics of the determination of micro- and macro-components in rocks.
Keywords: multichannel analyzer of the emission spectra; multielement atomic-emission analysis; arc discharge; analytical line.

UDC 543.423
Application of the "Potok" Device and MAES Analyzer to Gold reconnaissance at the Baikal Branch of "Sosnovgeologiya"
Malugin M. S., Grebinovskaya L. V.
Application of the equipment produced at the "VMK-Optoelektronika" Ltd. for analysis of rocks, gold ores and non-ferrous metal ores at the Baikal branch of "Sosnovgeologiya" of the Federal State Unitary Geological Enterprise "Urangologorazvedka" is considered. The advantages of using the aforementioned equipment in gold reconnaissance against classi-

cal approximate quantitative atomic emission analysis (gold atomic spectroscopy) with a visual interpretation of the spectrum are shown. The sets of analytical lines selected for gold determination provide a satisfactory quality of the results that match the requirements of industry guidelines. The metrological characteristics of the analysis procedure are evaluated in accordance with the current industry requirements for the development of methods of quantitative chemical analysis.

Keywords: multichannel analyzer of emission spectra; atomic emission spectroscopy; AC arc; analysis procedure.

UDC 543.423.1:54.08

Capabilities of a "Grand-Potok" System for Atomic-Emission Determination of Noble Metals in the Samples from Hydrotherms near the Volcanoes of the Kuril Islands

Zayakina S. B., Lesnov F. P., Anoshin G. N., Balukhtin A. V.

Study of natural nanoparticles of noble metals (NMs) is a rapidly developing area of modern geology and thus requires developing new precise instrumental methods of analysis. Further development of scintillation emission spectral analysis (SES) based on the use of a new spectrum recording system provides unprecedented opportunities for material research. We present the results of studying the samples obtained from hydrotherms near the volcanoes of the Kuril Islands using a modified SES method. It is shown that the proposed method of recording is not only a powerful tool for geochemists in determination of the quantitative content of NMs in a sample, but also provides information about the forms of the element present in the sample.

Keywords: noble metals; nanoparticles; scintillation emission spectroscopy (SES).

UDC 533.9

Study of the Degree of Violation of Local Thermodynamic Equilibrium in Analytical Zone of Two-Jet Plasmatron

Cherevko A. S.

Population of the energy levels is calculated for 25 Fe I (atom) and 26 Fe II (ion) spectral lines in analytical zone of a two-jet arc argon plasmatron. It has been shown that Saha – Boltzmann equilibrium exists between the ion ground state and highly excited levels of the atom. The neutral ground and low excited energy levels (<4.0 eV) of the atom, however, are not in Saha – Boltzmann equilibrium with these levels. The results of the study are treated in terms of partial local thermodynamic equilibrium (pLTE) regarding ionizing mechanism of analyte spectrum excitation.

Keywords: two-jet arc argon plasmatron; partial local thermodynamic equilibrium (pLTE); ionizing and recombining plasma state; radiative decay.

UDC 543.423

Analytical Zones of High-Power Two-Jet Plasma: Advantages and Limitations

Zaksas N. P., Veryashin A. F., Labusov V. A.

Capabilities of the analytical zones of high-power two-jet plasma are compared. A zone ahead of plasma jet confluence is shown to provide lower detection limits of the elements, however, strong dependence of the evaporation efficiency on the particle size is observed in analysis of heat-resistant materials. More effective evaporation of the samples occurs in a post-confluence, but the element detection limits are worse than in the region ahead of the jet confluence due to high background emission. The choice of the observation zone depends on a specific analytical goal.

Keywords: two-jet plasma; analytical zones; detection limits, evaporation efficiency; matrix effects.

UDC 543.423

Study of the Temperature Distribution and Spectral Line Intensity of Noble Metals in the Height of the Plasma Plume of Two-Jets Plasma Torch

Kuptsov A. V., Zayakina S. B., Saprykin A. I.

Distributions of the temperature, intensity of the spectral lines of noble metals and the background signal in the height of the plasma plume are studied on a multi-channel emission spectrometer «Grand» with a two-jets plasma torch as the excitation source. It is shown that the angle between the electrodes of the plasma torch has a significant impact on the temperature distribution and intensity of the analytical lines of noble metals

Keywords: two-jets plasma torch; atomic emission spectroscopy; noble metals.

UDC 543.423

New Program Possibilities of Time-Resolved Spectrometry with DC-Arc Source for Determination of Impurities in Metal and Powder Samples

Garanin V. G., Nekudov O. A.

An experience of using atomic emission optical time-resolved spectrometry in analysis of copper and ruthenium metal samples and geological powder samples is presented. Some analytical parameters of time-resolved spectrometry and characteristics are given.

Keywords: atomic emission spectroscopy; time-resolved spectrometry; powder sample; copper; ruthenium; spectrum analyzers; multi-element solid-state detectors; globular direct current arc spectrum source.

UDC 543.42:546.93

Validation of Iridium Spectral Analysis for Nanopowder Materials

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

Kazakov A. S., Aleksandrov E. P.

We present the unique properties of nanoscale powder of metal iridium produced at the JSC «SPA «Ural Metals». The features of atomic emission spectral determination of the impurities present in the iridium nanopowder using evaporation from the graphite electrode channel in ac arc discharge are considered.

Keywords: iridium; nanoscale powder; atomic emission spectral analysis; MAES.

UDC 621.039.7:543.423

Atomic Emission Spectral Analysis of Phosphate Concentrates of Fission Products and Impurities Obtained upon Pyrochemical Reprocessing of the Irradiated Nuclear Fuel

Khamdeev M. I., Vasilieva O. N., Chistyakov V. M., Erin E. A.

The results of using atomic emission spectral analysis in determination of the chemical composition of phosphate concentrates of fission products and impurities obtained upon pyrochemical reprocessing of the irradiated nuclear fuel are presented. A method of direct analysis of powders with their evaporation and electric arc starting is used to determine the chemical composition of low-solubility phosphates. To eliminate the impact of the physical and chemical properties and sample composition on the results of analysis we propose to open the sample in the electrode crater through phosphate recovery with carbon dust. To improve the reproducibility and minimize the impact of the sample chemical composition on the correctness of analysis we used a spectroscopic carrier BaCl₂. The optimal sample mass, «snifter»-type electrode parameters, current strength and exposure time are specified. The relative error of determinations ranges within 6.5 – 25.0%.

Keywords: atomic emission spectroscopy; irradiated nuclear fuel; pyrochemical reprocessing; phosphate concentrates of fission product; spectroscopic buffer and carrier.

UDC 543.423.1:543.621

Metrological Support of Certified Mixtures for the Analysis of Microimpurities

Leukhin S. G., Kochergina G. R.

An approach to calculating the metrological characteristics of certified mixtures used to determine microimpurities in powder samples of steel, nickel-based alloys, ferro-alloys, and pure metals is developed. A comparison is made of the calculation mechanisms proposed by State Standard GOST 8.531 and the recommendation M-25 developed at the Institute of Standard Samples. The proposed approach provides a more rapid and accurate determination of the certified performance. The developed technique (comprising PGS-2 spectrometer equipped with a MAES analyzer) has been successfully used by the Department of Analytical Control since 2005 to certify more than 20 sets for the content of seven elements (As, Bi, Cd, Pb, Sb, Sn, Zn).

Keywords: certified mixture; microimpurities; particulate material; error.

UDC 543.07

Practice of Using a Spectroanalytical Stand «Kristall»

Pivovarova O. A., Alekseeva E. A., Vasil'eva Yu. G.

The results of practical application of a spectroanalytical stand «Kristall» produced by the VMK-Optoelektronika Ltd. company are presented. The main parameters that affect the rapidity of analytical procedures and easy

Keywords: software for atomic emission spectroscopy; multichannel spectrometers; MAES analyzer; automation.

UDC 543.423

Automatic Wavelength Calibration of the Spectrometers with MAES Analyzers (Profiling)

Pankratov S. V., Labusov V. A., Nekludov O. A., Vashchenko P. V.

A software for automatic profiling (wavelength calibration) of the spectrometers with MAES analyzers which implements a quality filtering of experimental values is developed. This quality parameter is estimated using an analytic function describing the shape of spectral lines with allowance for the effect of merged spectral lines and other criteria. Integral evaluation of the quality of calibration points provides an automatic significant reduction of the profiling error. The method is implemented in the framework of the "Atom" program.

Keywords: cross-correlation function; wavelength calibration; multichannel spectrometers; atomic emission spectra; spectrum analyzers; multi-element solid-state detectors; photodiode arrays.

UDC 543.423

Algorithm for Processing Sequences of Spectra for Scintillation Atomic Emission Spectral Analysis

Semenov Z. V., Labusov V. A., Nekludov O. A., Vashchenko P. V.

An "Extended 2014" algorithm for mathematical processing of the sequences of time-resolved atomic emission spectra is developed to extract weak scintillations (flashes) of analytical lines from the noise and, as a consequence, to lower the element detection limits. Sequences of spectra are recorded using a multielement solid-state radiation detector with powder excitation in arc sources of spectra (for example, using the spill-injection method of powder introduction). The algorithm is compared with spectra processing algorithms previously implemented in "Atom 3.3" program. The algorithm described in this paper is advantageous for fewer adjustments, more correct determination of the background mask saving more points of the background that are used in the background shape determination. This improves the results of processing sequences of spectra, including statistic parameters such as the standard deviation of the calibration curve and the variances of convergence and adequacy. The signal-to-noise ratio of spectral line scintillations is also improved. The algorithm is implemented in the "Atom" program.

Keywords: scintillation; time-resolved spectroscopy; algorithm of spectrum processing; atomic emission spectroscopy.

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Arc Atomic-Emission Digital Spectrography of Liquid Biosamples Using MAES

Drobyshev A. I., Savinov S. S.

Results of studying an analytical potential of atomic (optical) emission digital spectrography with DC arc excitation of the spectrum of dried residue of liquid samples are presented. The results have been obtained on a MFS-8 spectral device modified with photodiode linear detector MAES. A possibility of increasing signal-to-noise ratio using higher arc current strength (20 A) and large amount of spectral buffer (0.15 mg NaCl) is shown. A universal technique of liquid biological samples analysis is developed using aqueous solutions of the salts of the elements as standard samples. The absence of the matrix impact is demonstrated in analysis of human saliva with dilution-addition method. The developed technique provides determination of a number of elements starting from $n \times 10^{-7}$ g/liter in the sample $n \times 10 \mu\text{l}$ with the reproducibility less than 15–20 rel.%. Linear dynamic range of calibration curve is 3–4 orders of the concentration of the elements to be determined.

Keywords: AES (OES); digital spectrography; dry residue; saliva; biological fluids.

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Arc Atomic Emission Analysis of Medicinal Herbs

Otmakhov V. I., Petrova E. V., Shilova I. V.,

Batanina A. A., Kuskova I. S., Rabtsevich E. S.

A modified procedure of preparing samples of medicinal herbs — hubs of trace elements — for spectral analysis is presented. The procedure consists

in introduction of corrective additives into standard samples and can be taken as a basis of developing pharmacopoeial methods of arc atomic emission analysis of medicinal herbs.

Keywords: atomic emission spectroscopy; arc discharge; medicinal plants; analysis; trace elements; metrology.

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On the Development of a Device for Simultaneous Multi-Element Atomic Absorption Spectral Analysis Based on a High-Dispersion Spectrometer and a Continuous Spectrum Source

Boldova S. S., Put'makov A. N., Labusov V. A., Borovikov V. M., Selyunin D. O., Gus'kova E. A.

An experimental setup for simultaneous multi-element atomic absorption spectral analysis comprising a continuous spectrum source, electrothermal atomizer (ETA), and a spectrometer with an operating range of 190–360 nm and a resolution of 0.025 nm is developed. The width of the entrance slit and the basic exposure time are optimized to reduce the limits of detection (LOD) of the elements to be determined. Calibration curves are constructed using a standard MES-11 sample with the following elements: Zn, Cd, Co, Cu, Ni, Bi, B, V, Ba, Pb, Mg, Sn, and Sb. The LOD for each element is determined using the calibration curve from the minimum value of the absorption signal that can be reliably determined. For a number of elements, the LOD values are comparable with those obtained on a ContrAA device with ETA; about an order of magnitude inferior to those of conventional atomic absorption spectrometry; and for most elements the LOD values match the Sanitary Requirements for the maximum permissible concentrations in drinking water in containers.

Keywords: atomic absorption spectrometry; simultaneous multi-element analysis; detection limits; electrothermal atomization; multi-element solid-state detector; continuous spectrum source.

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Algorithm for Processing Sequences of Atomic Absorption Spectra with a Continuous Radiation Source

Vashchenko P. V., Boldova S. S., Labusov V. A.

An algorithm for processing time sequences of atomic-absorption spectra recorded with a spectrometer with a MAES, electrothermal atomizer, and a continuous spectrum source is developed. The efficiency of the algorithm in comparison with the existing algorithm of the Atom program is tested in copper determination in aqueous solutions. The same recorded sequence of spectra has been processed using novel and existing algorithms. A more than twofold decrease in the detection limit is obtained using the developed algorithm.

Keywords: atomic absorption spectrometry; simultaneous multi-element analysis; detection limits; electrothermal atomization; multi-element solid-state detector; continuous spectrum source.

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Reduction of the Detection Limits in Trace Analysis of High Purity Substances by ICP-AES

Medvedev N. S., Put'makov A. N., Shaverina A. V.,

Tsygankova A. R., Saprykin A. I.

Application of a device for electrothermal sample vaporization ("VMK-Optoelectronika" Ltd., Russia) used to reduce the detection limits in determination of trace elements by ICP-AES is considered. Dependences of the analytical signal vs time for trace elements are studied. Operating conditions of electrothermal vaporization (ETV) are specified. The efficiency of using ETV in combination with ICP-AES is exemplified in analysis of high purity germanium dioxide with preliminary concentration of trace elements. Advantages and limitations of using ETV-ICP-AES procedure for trace analysis of high purity substances are discussed.

Keywords: atomic emission spectrometry; inductively coupled plasma; electrothermal vaporization; trace analysis; preconcentration of trace elements; detection limits.