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ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В. Сокольский атындағы «Жанармай,  
катализ және электрохимия институты» АҚ

# Х А Б А Р Л А Р Ы

## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН  
АО «Институт топлива, катализа и  
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## NEWS

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*NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.*

*Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.*

*НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.*

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**DETERMINATION OF REM IN URANIUM ORES  
OF THE SHU-SARYSU URANIUM-ORE PROVINCE BY INDUCTIVELY  
COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY**

**Abstract.** This article discusses the features of the determination of elements in uranium ores of the Shusarysu uranium-ore province by inductively coupled plasma atomic emission spectrometry in comparison with the ore standard of Ore Research & Exploration (Australia). A multi-stage method of sample preparation for measurement is considered. The existing and developed methods for determining the elemental composition of uranium ores are focused, as a rule, on the determination of specific elements using regulated analysis procedures. However, during routine analysis, we need to quickly and efficiently determine a larger list of elements available with one instrumental method. Various methods are used to obtain multielement analysis, among which mass spectrometry and inductively coupled plasma atomic emission analysis are most popular due to their high performance and low detection limits. The listed methods work with solutions, therefore, it is necessary to pay great attention to sample preparation and instrument calibration.

The invention relates to a method for preparing samples for the study of materials by determining their chemical properties in a liquid state, for example, using inductively coupled plasma spectrometry. The method for preparing silicate and carbonate rock samples for spectrometric analysis includes autoclave decomposition of samples in a mixture of concentrated acids in a microwave oven at temperature and pressure and cooling the samples. Then carry out the introduction of the internal standard and bringing to the standard volume.

**Key words:** uranium ore, internal standard, elemental analysis, ICP-NPP.

**Introduction.** The existing and developed methods for determining the elemental composition of uranium ores are focused, as a rule, on the determination of specific elements using regulated analytical procedures. However, during routine analysis, there is the need to quickly and qualitatively determine a large list of elements available with only one instrumental method. Various methods are used to obtain multielement analysis, among which mass spectrometry and inductively coupled plasma atomic emission analysis are most popular due to their high performance and low detection limits [1-3]. The listed methods work with solutions, therefore, it is necessary to pay great attention to sample preparation and to instrument calibration.

**Object of study.** The ore used in the experiment is mined at an operating uranium mining enterprise. It belongs to the exogenous bed-infiltration type of deposits of the Shu-Sarysu uranium-ore province, suitable for mining by in-situ leaching [2]. The composition of the ore is on average represented by 70-90% of the mineral of quartz, feldspar and siliceous rocks, the content of accessory minerals not exceeding 3%. The content of carbonates is higher than 2%. Uranium in the ore is represented by two minerals - pitchblende and coffinite. The uranium content in the reservoir-infiltration deposits is tenths and hundredths of a percent, rarely reaching 1% [4-6].

To compare the characteristics, the OREAS 120 uranium ore standard produced by Ore Research & Exploration (Australia) was used. The source of the uranium ore is the Mantra Resources Nyota Prospect uranium deposit (Tanzania), which is an exogenous deposit, with a mineralization ranging from medium

to very coarse-grained sandstones penetrated by graywackes and siltstones. The composition of macrocomponents (matrix) is similar to the ore of the Kazakh deposit. The OREAS 120 uranium ore standard is certified in the Register of GSI RK [7].

**Research methods.** The invention describes a method for preparing samples for the study of materials by determining their chemical properties in the liquid state, for example, using inductively coupled plasma spectrometry. The method for preparing silicate and carbonate rock samples for spectrometric analysis includes autoclave decomposition of the samples in a mixture of concentrated acids in a microwave oven at temperature and pressure and further cooling of the samples. Then the internal standard is introduced and brought to the standard volume. The complete decomposition of the samples is carried out in 2 stages [7-11].

In the first stage, a mixture of hydrofluoric, hydrochloric and nitric acids is used in a ratio of 1:3:1 - 1:4:1 for 45-50 minutes, and the cooling of the samples is carried out in a closed autoclave to a temperature of 80-40 °C. In the second stage of decomposition, a mixture of concentrated hydrochloric, nitric and dilute boric acids is used in a ratio of 1:1:10 - 1:1:12 for 10-15 minutes. The technical result achieved in this case is the increase of the efficiency of preparation of rock samples while reducing the labor intensity of work and increasing the purity of the final sample. As referred, the invention describes methods for studying materials by determining their chemical properties in the liquid state, for example, using inductively coupled plasma spectrometry (ICP-AES, ICP-MS). The aim of the invention is to improve the efficiency of preparation of rock samples while reducing the complexity of work and increasing the purity of the final sample [12]. This goal is achieved by the fact that the method of preparing silicate and carbonate rock samples for spectrometric analysis, including autoclave decomposition of the samples in a mixture of concentrated acids in a microwave oven at a temperature, cooling the samples, introducing an internal standard, bringing it to a standard volume and the complete decomposition of samples is carried out in 2 stages. In the first stage, a mixture of hydrofluoric, hydrochloric and nitric acids is used in a ratio of 1:3:1 - 1:4:1 for 45-50 minutes and the cooling of the samples is carried out in a closed autoclave to a temperature of 80-40 °C. In the second stage of decomposition, a mixture of hydrochloric, nitric and boric acids is used in a ratio of 1:1:10 - 1:1:12 for 10-15 minutes.

Moreover, the decomposition of the samples is carried out in the microwave system MARS-5. The pressure in the first stage of decomposition is maintained at 12-14 atm and in the second stage of decomposition is equal to 5-7 atm. For the second stage of decomposition, boric acid with a concentration of 4-4.5% is used. The temperature of the decomposition process is maintained in the range of 180-210°C [13-16].

The use in the first stage of decomposition of a mixture of hydrofluoric, hydrochloric and nitric concentrated acids in a ratio of 1:3:1 - 1:4:1 at a pressure of 12-14 atm for 45-50 minutes makes it possible to almost completely transfer the sample into solution, transferring fluoride metals to chlorides. The acid ratios were determined experimentally. When the ratio in the mixture of hydrofluoric, hydrochloric and nitric acids is less than 1:3:1 and the time is less than 45 minutes, the sample does not completely decompose. The minimum time is needed to achieve the specified parameters. In addition, such an amount of hydrochloric acid is not quite enough to convert fluorides into chlorides, which is evident from the presence of gray precipitates in some autoclaves [17].

The ratio of acids in the mixture of more than 1:4:1 is impractical, since at such ratio of acids the fluorides of the sample completely pass into chlorides, which can be seen from the transparent solution for carbonates and white crystalline precipitate for silicates.

Cooling autoclaves to a temperature higher than or equal to 85 °C leads to a loss of silicon. Cooling to a temperature below or equal to 35 °C leads to the precipitation of insoluble fluorides of some metals. Therefore, the samples are cooled with a closed autoclave to a temperature of 80-40 °C.

The use in the second stage of decomposition of a mixture of concentrated hydrochloric, nitric and diluted boric acids in a ratio of 1:1:10 - 1:1:12 for 10-15 minutes and with exposure to microwave radiation allows completely transferring the samples into a solution, further decomposing silicon-rich samples and binding unreacted HF into a complex. The ratios of the components of acids in the mixture and the time required for the complete decomposition of the samples were selected experimentally, the data being shown in table 2.

The presence of boric acid in the acid mixture makes it possible to create conditions under which subsequent addition of water does not form precipitated hydroxides of the elements. Thus, boric acid acts

as a kind of buffer, i.e. inhibits the hydrolysis of some salts. The boric acid concentration was selected experimentally.

When a saturated solution (6.5%) of boric acid is added, the results are good, but the solution has a high density and subsequently clogs the capillary of the spectrometer. Therefore, the boric acid concentration was selected in order to obtain the lowest density of the final sample without compromising the achievement of the final goal - obtaining a transparent solution that is stable over time.

The decomposition of samples in the MARS-5 microwave system allows to carry out the entire process from start to finish in one autoclave, reducing the laboriousness of sample preparation, eliminating process losses and losses of volatile components, thus increasing the reliability of the results.

The temperature regime of the decomposition process is determined by the equipment used and by the holding time. For 40-45 minutes of decomposition, the process temperature reaches 180-210 °C.

An example of a specific implementation of the method is described in the following.

Before analysis, the samples were dried at a temperature of 105 °C for at least 1 hour. Then a sample of 0.1 g was weighed on an analytical balance from Mettler Toledo AX 204, with a graduation of 0.1 mg. The weighed portion was quantitatively transferred into an autoclave and moistened with 0.25 mL of deionized water obtained on an ultrapure water unit from Millipore, USA.

The first stage of decomposition was then carried out. For this purpose, concentrated, highly pure or chemically pure acids were added to each autoclave, the purity of which was checked on the device before operation [1]. All acid addition operations were carried out in a fume hood with automatic pipette dispensers.

Hydrofluoric acid HF ( $d=1,15\text{g/cm}^3$ ) GOST 10484-78

Nitric acid HNO<sub>3</sub> ( $d=1,42\text{g/cm}^3$ ) GOST 4461

Hydrochloric acid HCl ( $d=1,19\text{g/cm}^3$ ) GOST 3118-77

1 mlHF + 1 mlHNO<sub>3</sub> + 4 mlHCl

The autoclaves were assembled, placed in a turret (a rotating table with slots for assembled autoclaves) and in an oven (a MARS-5 microwave sample decomposition system (CEM Corporation, USA). 14 autoclaves are placed in the turret simultaneously, one of which is a control autoclave with control sensors temperature and pressure. The conditions for the first stage of decomposition were set as in the following:

- power - 1200W with a full load of the furnace (14 autoclaves);
- pressure - 12-14 atm (190-200 psi, where 14 psi = 1 atm);
- temperature - 200-210 °C;
- decomposition time - 45-50 minutes (time to reach these parameters - 25 minutes; holding time with these parameters - 20-25 minutes);
- cooling time to a temperature of 80-40 °C - 30 minutes.

Data on the results of the first stage of decomposition are summarized in table 1.

The turret with the autoclaves was removed from the oven, the autoclaves were disassembled and acids were added for the second stage of decomposition: 1 mL HCl + 1 mL HNO<sub>3</sub> + 10 ml 4% H<sub>3</sub>BO<sub>3</sub>

Boric acid was prepared from chemically pure boric acid powder (40 g per 1 L of deionized water, heated if necessary for better dissolution. The purity of the reagent was checked on the instrument). This concentration is optimal for boric acid.

The autoclaves were reassembled, placed in the turret and in the oven. Conditions for the second stage of decomposition were set as in the following:

- power - 1200W with a full furnace load (14 autoclaves);
- pressure - 5-7 atm (70-100 psi, where 14 psi = 1 atm);
- temperature - 180-210 °C;
- decomposition time - 15-20 minutes (time to reach these parameters - 10 minutes, holding time with these parameters - 5-10 minutes);
- cooling time to room temperature - 50 minutes.

The data with the results of decomposition in the second stage are summarized in table 2.

After cooling, the turret was removed from the oven, the autoclaves were disassembled, the resulting solutions were poured into 25 mL volumetric flasks, the autoclaves and caps were rinsed with 5 mL of



deionized water, and 2 mL of cadmium solution (250 ppm) was added to each flask, which was used as an internal standard.

Table 1 - Data on stage I of decomposition

| №  | Concentrated acid ratios |     |                  | Time, min | Sample decomposition result                           |
|----|--------------------------|-----|------------------|-----------|---|
|    | HF                       | HCl | HNO <sub>3</sub> |           |   |
| 1  | 4                        | 1   | 1                | 35        | Not completely decomposed, dark precipitate lacks HCl |
| 2  | 4                        | -   | 4                | 25        | Not completely decomposed, dark precipitate lacks HCl |
| 3  | 4                        | -   | 3                | 35        | Not completely decomposed, dark precipitate lacks HCl |
| 4  | 3,5                      | 0,5 | 1,5              | 25        | Not completely decomposed, dark precipitate lacks HCl |
| 5  | 4                        | 3   | 1                | 35        | Not completely decomposed, not enough time            |
| 6  | 4                        | 1   | 2                | 50        | Bad, not enough HCl                                   |
| 7  | 2                        | 2   | 1                | 35        | Bad, dark precipitate lacks HCl                       |
| 8  | 1                        | 3   | 2                | 50        | Almost complete decomposition, after one              |
| 9  | 1                        | 3   | 1                | 35        | Almost complete decomposition, not enough time        |
| 10 | 1                        | 3   | 1                | 50        | Complete decomposition                                |
| 11 | 1                        | 4   | 1                | 40        | Almost complete decomposition, not enough time        |
| 12 | 1                        | 4   | 1                | 50        | Complete decomposition                                |
| 13 | 0,5                      | 4   | 1                | 35        | Bad, dark sediment, not enough HF                     |
| 14 | 0,5                      | 4   | 1                | 50        | Bad, dark sediment, not enough HF                     |
| 15 | 0,5                      | 3   | 2                | 50        | Bad, dark sediment, not enough HF                     |
| 16 | 0,5                      | 3   | 1                |           | Not decomposed  |
| 17 | 0,5                      | 2,5 | 0,5              |           | Not decomposed  |

Table 2 - Data on the II stage of decomposition

| №  | Acid ratios |                  | Warming up                        | MARS-5 | Time, min | Sample decomposition result      |
|----|-------------|------------------|-----------------------------------|--------|-----------|----------------------------------|
|    | HCl         | HNO <sub>3</sub> |                                   |        |           |                                  |
| 1  | -           | -                | 4% H <sub>3</sub> BO <sub>3</sub> | yes    | 15        | The presence of a crystal, draft |
| 2  | 1           | -                | 15                                | no     | 15        | The presence of a crystal, draft |
| 3  | 1           | -                | 15                                | yes    | 15        | The presence of a crystal, draft |
|    | 1           | 1                | -                                 | yes    | 15        | White flakes of sediment         |
| 9  | 1           | 1                | 5                                 | yes    | 15        | The presence of a crystal, draft |
| 11 | 1           | 1                | 9,0                               | yes    | 15        | Almost good, sediment on cooling |
| 12 | 1           | 1                | 9,5                               | yes    | 15        | Sediment after one               |
|    | 1           | 1                | 10                                | yes    | 15        | Clear solution                   |
| 13 | 1           | 1                | 11                                | yes    | 15        | Clear solution                   |
|    | 1           | 1                | 12                                | yes    | 15        | Clear solution                   |
| 3  | 1           | -                | 12                                | yes    | 15        | Presence of sediment             |
|    | 1           | 1                | 10                                | yes    | 20        | Clear solution                   |

The completely cooled samples were brought to the mark with deionized water. All solutions, regardless the type of rock, turned out to be transparent, i.e. the rock completely passed into solution. The samples were poured into containers made of chemically resistant plastic [5, 13]. It is impossible to store solutions in glassware even for a short time, since there is an acidic "leaching" of silicon and boron, which go into solution and fall out in the form of hardly soluble salts. The samples are then sent for analysis to an IRIS DUO spectrometer from Thermo Elemental (USA).

The process of preparing samples from the rock, from weighing the sample to receiving a clear solution for analysis takes 3-3.5 hours. In this case, the samples are not "burdened" with sodium, lithium and retained silicon and, therefore, these elements can be determined.

### Conclusion.

1. A method of preparing silicate and carbonate rock samples for spectrometric analysis, including autoclave decomposition of samples in a mixture of concentrated acids in a microwave oven at temperature and pressure, cooling the samples, introducing an internal standard and bringing them to a standard volume, is characterized by a complete decomposition of samples carried out in 2 stages. In the

first stage, a mixture of hydrofluoric, hydrochloric and nitric acids is used in a ratio of 1:3:1 - 1:4:1 for 45-50 minutes, the cooling of the samples being carried out in a closed autoclave to a temperature of 80-40 °C. In the second stage the decomposition use a mixture of concentrated hydrochloric, nitric and diluted boric acids in a ratio of 1:1:10 - 1:1:12 for 10-15 minutes[14].

2. The method for preparing silicate and carbonate rock samples for spectrometric analysis according to claim 1 is characterized by the fact that the autoclave decomposition of the samples is carried out in the MARS-5 microwave system.

3. The method for preparing silicate and carbonate rock samples for spectrometric analysis according to claims 1 or 2 is characterized by the fact that the pressure in the first stage of decomposition is maintained at 12-14 atm.

4. The method for preparing silicate and carbonate rock samples for spectrometric analysis according to claims 1 or 2 is characterized by the fact that the pressure in the second stage of decomposition is maintained at 5-7 atm.

5. The method for preparing silicate and carbonate rock samples for spectrometric analysis according to claim 1 is characterized by the fact that for the second stage of decomposition dilute boric acid with a concentration of 4-4.5% is used [2].

6. The method for preparing silicate and carbonate rock samples for spectrometric analysis according to claim 1 is characterized by the fact that the temperature of the decomposition process is maintained in the range of 180-210 °C.

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#### **ШУ-САРЫСУ УРАН-КЕН ПРОВИНЦИЯСЫНДАҒЫ УРАН КЕНДЕРІНДЕГІ РРМ ИНДУКТИВТІ БАЙЛАНЫСҚАН ПЛАЗМАЛЫҚ АТОМДЫҚ-ЭМИССИЯЛЫҚ СПЕКТРОМЕТРИЯ ӘДІСІМЕН АНЫҚТАУ**

**Аннотация.** Мақалада Шу-сарысу уран-кен провинциясындағы уран кендеріндегі элементтерді индуктивті байланысқан плазмалық атомдық-эмиссиялық спектрометрия әдісімен анықтау ерекшеліктері талқыланады, Ore Research & Exploration (Австралия) кен стандартымен салыстырғанда, өлшеуге сынаманы дайындаудың көп сатылы әдісі қарастырылған.

Уран кендерінің элементтік құрамын анықтаудың қолданыстағы және жасалынып жатқан әдістемелері, әдетте, талдаудың регламенттелген процедураларын пайдалана отырып, нақты элементтерді анықтауға бағытталған. Алайда, рутинді талдау кезінде элементтердің үлкен тізімін қол жетімді бір аспаптық әдіспен жылдам және сапалы анықтау қажет. Көп элементті талдау алу үшін арасында жоғары өнімділігінің және анықтаудың төмен шегінің арқасында кең танымалдылыққа ие плазмамен индуктивті байланысқан масс-спектрометрия және атомды-эмиссиялық талдау бар әртүрлі әдістер қолданылады. Аталған әдістер ерітінділермен жұмыс жасайды, демек, сынамаларды дайындау мен құрылғыны калибрлеуге үлкен назар аудару қажет.

Өнертабыс сұйық күйдегі химиялық қасиеттерін анықтау арқылы материалдарды зерттеуге сынамаларды дайындау әдісіне жатады, мысалы: индуктивті байланысқан плазмасы бар спектрометрияны қолдану. Силикатты және карбонатты тау жыныстарын спектрометриялық талдау үшін дайындау әдісі концентрацияланған қышқыл қоспасындағы сынамаларды температура мен қысым кезінде микротолқынды пеште автоклавты ыдыратуды және сынамаларды салқындатуды қамтиды. Содан кейін ішкі стандартты енгізу және стандартты көлемге жеткізу жүзеге асырылады.

**Түйін сөздер:** уран рудасы, ішкі стандарт, элементтік талдау, ИСП-АЭС.

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**ОПРЕДЕЛЕНИЕ РЗМ В УРАНОВЫХ РУДАХ ШУ-САРЫСУЙСКОЙ  
УРАНОВО-РУДНОЙ ПРОВИНЦИИ МЕТОДОМ АТОМНО-ЭМИССИОННОЙ СПЕКТРОМЕТРИИ  
С ИНДУКТИВНО-СВЯЗАННОЙ ПЛАЗМОЙ**

**Аннотация.** В статье обсуждены особенности определения элементов в урановых рудах Шу-сарысуйской ураново-рудной провинции методом атомно-эмиссионной спектроскопии с индуктивно-связанной плазмой в сравнении со стандартом руды компании OreResearch&Exploration (Австралия), рассмотрен многостадийный способ пробоподготовки проб к измерению.

Существующие и разрабатываемые методики определения элементного состава урановых руд ориентированы, как правило, на определение конкретных элементов с использованием регламентированных процедур анализа. Однако при рутинном анализе нам необходимо быстро и качественно определить большой перечень элементов доступный одним инструментальным методом. Для получения многоэлементного анализа используются различные методы, среди которых масс-спектрометрия и атомно-эмиссионный анализ с индуктивно-связанной плазмой имеют наибольшую популярность, благодаря высокой производительности и низким пределам обнаружения. Перечисленные методы работают с растворами, следовательно, необходимо уделить большое внимание пробоподготовке проб и калибровке прибора.

Изобретение относится к способу подготовки проб к исследованию материалов путем определения их химических свойств в жидком состоянии, например, с использованием спектроскопии с индуктивно-связанной плазмой. Способ подготовки силикатных и карбонатных проб горных пород для спектроскопического анализа включает автоклавное разложение проб в смеси концентрированных кислот в микроволновой печи при температуре и давлении и охлаждение проб. Затем осуществляют введение внутреннего стандарта и доведение до стандартного объема.

**Ключевые слова:** урановая руда, внутренний стандарт, элементный анализ, ИСП-АЭС.

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