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ИЗВЕСТИЯ

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АО «ИНСТИТУТ ТОПЛИВА, КАТАЛИЗА И
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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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Адрес редакции: 050100, г. Алматы, ул. Кунаева, 142,
Институт органического катализа и электрохимии им. Д. В. Сокольского,
каб. 310, тел. 291-62-80, факс 291-57-22, e-mail: orgcat@nursat.kz

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142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22,
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I. Yu. Silachyov

Institute of Nuclear Physics, Almaty, Kazakhstan

E-mail: silachyov@inp.kz

**DETERMINATION OF RARE EARTHS IN URANIUM RAW MATERIAL
BY NEUTRON ACTIVATION ANALYSIS AND X-RAY FLUORESCENCE**

Abstract. Development of the rare-earth branch of the non-ferrous metallurgy is declared as a foreground direction of the Kazakhstan's national economy in the XXI century. High content of rare earth elements (REE) is a characteristic feature of various Kazakhstan's uranium deposits. To determine REE content of mineral raw materials the next non-destructive methods are the most suitable – instrumental neutron activation analysis (INAA) and X-ray fluorescence (XRF). A range of REE determination in a uranium ore by neutron activation technique is substantially impeded by the uranium fission products generating direct (La, Ce and Nd) and spectral interferences. Geological sample analysis for REE content by X-ray fluorescence suffers from insufficient sensitivity, as usual, and is restricted by the light lanthanides mainly.

In the present work, by the example of the ore samples from the Shu-Sarysu uranium provinces, the possibility of INAA method to analyze REEs under the conditions of severe interferences caused by high uranium content is considered. Light lanthanide determination by XRF at the level of their average crustal abundance is carried out with the help of a modernized energy dispersive spectrometer RLP-21T using K-series of their characteristic X-ray radiation. Comparison of La, Ce and Nd content of the uranium ore samples by INAA and XRF using the non-parametric Mann-Whitney statistical test showed that distinction of the results is statistically insignificant. Based on the uncertainty budget evaluation, maximum corrections of the analytical line count rates are assessed corresponding to the limits of quantitative and semi-quantitative determination of La, Ce and Nd by INAA. XRF was concluded as a more preferable method to analyze rocks for light lanthanide content.

Keywords: neutron activation analysis, X-ray fluorescence, rare earth elements, uranium ore.

Introduction. Due to the increased demand for the rare earth elements (REE) in the world's high-technological industries, additional sources for their extraction differing from the REE ores are searched now. REEs are often found associated with other valuable element mineralization including uranium ores which can contain appreciable REE contents [1,2]. E.g. the unique complex uranium deposit Melovoe in Kazakhstan belongs to this type [3].

Among the most common methods being used at present for REE determination in geological samples, the non-destructive techniques such as instrumental neutron activation analysis (INAA) and X-ray fluorescence (XRF) analysis can be noticed [4,5], since one of the main drawbacks of the other methods is sample decomposition.

Spectral interferences from the uranium fission products in INAA including light lanthanides were studied in details by many researchers [6-8]. Corrections are made by calculation apparent concentration of an element per 1 ppm of U or uranium k_0 -fission factors both theoretically and experimentally [9-11]. At present fission product correction algorithms embedded in the analytical software such as Kayzero for Windows work very well in routine practice and only the samples with very high uranium contents can give rise to trouble [12].

Unlike INAA, XRF analysis of geological samples for REEs is not impeded by high uranium contents, and this is its greatest advantage [13]. One of the main restrictions is caused by the lack of appropriate compact and safe sources of primary gamma-rays due to the relatively high energies of REE

K-series excitation – more than 39 keV. (100 kV X-ray tube, the out-of-production expensive XRF spectrometer Epsilon 5 is equipped with, is the single exception [14].) Therefore direct (without any preconcentration) energy dispersive XRF with isotope excitation by ^{241}Am suffers from insufficient sensitivity and can be used, as a rule, to analyse rocks only for La to Nd above their average crustal contents [15,16]. Some heavier REEs can be determined by their L-series, but the sensitivity is even worse and sophisticated algorithms must be used to overcome severe spectral interferences [17]. Wave dispersive XRF spectrometers gain the lower limits of detection due to better resolution in the low-energy region [18]; however, a preconcentration is necessary for reliable analysis for heavy lanthanide contents [19].

In this work, high sensitive energy dispersive XRF with REE K-series excitation and INAA using the XRF-based internal standard method (ISM) were applied to analyze REE content of the uranium core samples collected from a range of new sites of the Shu-Sarysu uranium provinces in Southern Kazakhstan.

Methods

A great number of grinded samples were studied by the gamma-ray spectrometric method and more than thirty of them characterized by uranium content not exceeding ≈ 500 ppm were selected. Y, La, Ce, Pr, and Nd contents of the samples were measured by XRF technique using a modified energy dispersive spectrometer RLP-21T by JSC “AspapGeo”, Almaty, Kazakhstan. 12-15 g of the powdered assays to provide “the saturated layer geometry” were pressed in Al dishes with mylar film bottom and analyzed in the automatic operating mode for twenty minutes.

The modified RLP-21T represents a new generation of reasonably priced portable XRF spectrometers since it can excite K-series of the X-ray spectra by a miniature low-powered X-ray tube. Maximum power of the X-ray tube with tungsten anode and air cooling is 10 W, and it can operate at up to 70 kV. The spectra are counted by a Peltier-cooled high-purity Si detector. No intermediate target is used due to a drastic loss of primary gamma-ray intensity. Since light lanthanide K-series is registered over the low-energy slope of the X-ray tube scattered bremsstrahlung radiation, a high-efficiency spectrum treatment algorithm is applied to calculate peak areas under the unfavourable signal-to-background ratio conditions. In order to get maximum accuracy, the element content of the samples is calculated using a reference-free modified method of the fundamental parameters.

As the spectrometer was designed to analyze geological samples, first of all, the accuracy of its software was repeatedly confirmed with the help of different corresponding certified reference materials (CRMs). E.g., the ascribed uncertainty of Fe content measuring in rock samples is (2-8)%, and this made possible to use the results of Fe analysis as an internal standard in INAA to calculate REE and other element contents [20]. RLP-21T is enrolled in the State Register of Measuring Devices, and the certified analytical technique is registered by the National Body for Certification of Kazakhstan

INAA of the same samples for the long-lived REEs only (without Pr, Dy and Er) was then carried out. About 100 mg of the assays were sealed in small double polyethylene bags, divided into several parties, and packed in Al foil for independent irradiations. A zirconium monitor to calculate epithermal to thermal neutron flux ratio $1/f$ (20 mg of ZrO_2) and about 50 μg of uranyl to evaluate the contribution of the uranium fission products were added to each package. The latter samples were prepared by drying a drop of uranyl nitrate solution pipetted on a piercing of Al foil.

To verify the accuracy of REE analysis two samples of the uranium-rich CRMs were used – OREAS 100a (uranium-bearing multi-element reference material by Australian “Ore Research & Exploration”), and OSO 528 (phosphorus-REE-uranium ore by the All-Russian Scientific Research Institute of Mineral Resources). Due to the enhanced REE contents, the sample masses were reduced to 30 mg to avoid possible self-shielding of the neutron flux. However, since the U/REE ratios of these CRMs are not too high, two additional control samples were prepared by adding about 100 μg of uranyl to 100 mg of two rock CRMs with low REE contents – GBW-07110 (trachyte andesite by the Institute of Rock and Mineral Analysis, China), and GBPG-1 (garnet biotite plagiogneiss by the A.P.Vinogradov Institute of Geochemistry, Russia).

All the packages were placed in the irradiation containers and irradiated in different days for 2.5 hours in the position № 4 inside the “wet” channel № 10-6 of the new modified active zone of the research reactor WWR-K with a typical neutron flux density $n \cdot 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$.

Two high-purity Ge detectors of different types were used to collect gamma-ray spectra: an extended-range coaxial GX5019 with a relative efficiency of 50% and an energy resolution of 1.86 keV at the 1332 keV peak of ^{60}Co connected to a Canberra multi-channel analyzer DSA-1000, and a planar GLP36360 with the crystal dimensions 36x13 mm and an energy resolution of 585 eV at the 122 keV peak of ^{57}Co connected to an ORTEC multi-channel analyzer DSPEC LF. Detector calibration for relative detection efficiency was made with the help of a multi-gamma ray standard (^{152}Eu , ^{154}Eu , ^{155}Eu) and an isotopic source ^{133}Ba , both by Canberra.

The irradiated uranium ore samples were counted by the coaxial detector several times: after seven days (to analyze La, Sm, Yb), two weeks (Ce, Nd, Eu, Tb, Sc), and almost two months (Gd, Tm) of decay. Such a long time interval comparing with 30 days for usual rock analysis is necessary to make ^{239}Np X-ray $K_{\alpha 1}$ peak (103.4 keV) completely decayed since it turned out to be a serious spectral interference for the 103.2 keV line of ^{153}Gd . To avoid the same interference in the case of ^{153}Sm analysis intensity of its 69.7 keV line was measured.

The planar detector was used to analyze holmium in the ore samples by two measurements – after 6 days (t_1) of decay and after 15-17 days (t_2), when ^{166}Ho was considered completely decayed. ^{166}Ho count rate to calculate the corresponding REE content was found as follows:

$$J_{t_1}^{\text{Ho}} = J_{t_1}^{\Sigma} - J_{t_2}^{\text{Xe}} / k \exp(-\lambda_{\text{Xe}}(t_2 - t_1)),$$

where J^{Σ} is the sum peak (^{166}Ho and ^{133}Xe) count rate, λ_{Xe} – the ^{133}Xe decay constant, and factor k takes account of different measuring geometries. Due to a considerable spectral interference of 208.4 keV line of ^{208}Lu and 209.8 keV line of ^{239}Np the former line intensity was also counted by the planar detector after two or three weeks of decay depending on the uranium content.

Table 1 – Main nuclear parameters and interferences of the radionuclides used to calculate REE by INAA internal standard method

Radionuclide	Half-life, days	Energy, keV	Quantum yield, %	Interferences	Detector
^{59}Fe	44.5	1099.2	56.5		GX5019
^{46}Sc	83.8	889.3	99.9		GX5019
^{140}La	1.7	1596.2	95.4	U(n,f)	GX5019
^{141}Ce	32.5	145.4	48.3	U(n,f)	GX5019
^{147}Nd	11.0	91.1	28.1	U(n,f)	GX5019
^{153}Sm	1.9	69.7	4.7	^{187}W , ^{140}La	GX5019 GLP36360
^{152}Eu	4943	121.8	28.7		GX5019
^{153}Gd	240.4	103.2	21.1	^{153}Sm , ^{233}Pa , ^{239}Np	GX5019
^{160}Tb	72.3	298.6	26.1	^{233}Pa	GX5019
^{166}Ho	1.1	80.6	6.7	^{133}Xe , ^{131}I	GLP36360
^{170}Tm	128.6	84.3	2.5	^{182}Ta	GX5019
^{175}Yb	4.2	396.3	13.2		GX5019
^{177}Lu	6.6	208.4	10.4	^{239}Np	GLP36360
^{233}Pa	27.0	311.9	38.5		GX5019 GLP36360
^{131}I	8.0	364.5	81.5		GX5019

The main nuclear parameters of the analytical gamma-ray lines of the radionuclides used to calculate REE content, the interferences which were taken into account, and the detectors to count specific radionuclide intensities are summarized in Table 1. U(n,f) means the same radionuclide as a U fission product, whereas ^{133}Xe and ^{131}I by the uranium fission reaction appear as a spectral interference. Some minor interferences in an ordinary rock matrix were regarded as inessential and neglected. The partly resolved peaks were divided by the software.

Typical measuring time was 20-30 minutes and amounted to three hours in the case of Gd and Tm analysis because of their low content. Spectra collection was carried by the MAESTRO software (GLP36360) and by the special software developed in the Institute of Nuclear Physics (GX5019). No corrections for neutron self-shielding, gamma-ray self-absorption or true-coincidence effects were applied.

Unlike the approach where La, Ce, and Nd apparent contents are corrected for uranium fission products using U measured contents, the analytical line count rates of the corresponding radionuclides were corrected. With this end in view, the contribution coefficients F_i as the ratios of these analytical line intensities to the intensities of ^{239}Np full-energy peak and two long-lived uranium fission product (^{131}I , ^{140}Ba) peaks were estimated empirically. Once measured in certain time F_i values can be recalculated for any time of decay. ^{153}Sm peaks originating from ^{235}U fission after uranyl sample irradiations were indistinguishable in the spectra and this interference was therefore neglected.

The same variant of the single comparator method of standardization as described in [20] was applied, and REE content of the uranium ore samples were calculated according to the algorithm presented in [21]. $1/f$ ratio in the selected position by the results of a range of independent irradiations appeared to be $1/f = 0.031 \pm 0.002$ ($P = 0.95$, $\sigma = 0.001$). This ratio differs from the value evaluated in [21] because for the time elapsed beryllium reflectors were installed into the reactor's active zone.

Fe content of the uranium samples measured by XRF was used as the internal comparator to determine U, Th and the REEs analyzed by the coaxial detector. Since the intensity of 192.3 keV line of ^{59}Fe counted by the planar detector was very low, Th content of the samples was used as the internal comparator to analyze Lu, and Sm content – to analyze Ho.

Results and discussion

All the three ways of La, Ce, and Nd analytical line count rate correction were compared with the help of OSO 528 and OREAS 100a and led to almost the same contents of these REEs. Relative bias was about 1% in the case of Nd determination, <2%, and <3% in the cases of Ce and La determination, correspondingly. However, taking account of that ^{140}Ba count rate may be rather low and ^{239}Np count rate is much more affected by neutron flux spectrum composition than fission products, the ^{131}I -based correction was considered as more reliable. The results of light lanthanide analysis in the two uranium-rich CRMs using this way of correction are presented in Table 2.

As for the uranium-enriched CRMs, the measured Nd contents using the ^{131}I -based correction differed by 1% comparing with the results of initial CRM analysis, by 3% and by (3-4)% in the cases of Ce and La determination; however the assessments can be different if compared with the certified values in Table 3. To demonstrate that the loss of accuracy wasn't too high Figure 1 is adduced revealing how the correction factor $K_{\text{La,Fe}}$ for La analysis by INAA ISM was defined [20]. Only the CRMs contributing to $K_{\text{La,Fe}}$ evaluation within $\pm 5\%$ of the relative bias including 07110 and GBPG-1 (the green area) were selected. Two dark dots show deviations of La determination in the same CRMs when they were highly enriched with uranium.

The values of La, Ce, and Nd count rate corrections came up to (70-85)%. Despite the recommendation to reduce decay time of ^{140}La analysis in order to decrease its production from the reaction $^{235}\text{U}(n,f)^{140}\text{Ba} \rightarrow ^{140}\text{La}$ [11], the results after a fortnight decay (when ^{140}La decay curve becomes exponential-like) appeared to be more correct than after a week of decay, although the correction is higher. That is why two weeks of decay was implemented subsequently to analyze the uranium ore samples.

Table 2 – REE content of the uranium-rich CRMs, ppm

CRM	Element	U/REE ratio	Certified value	Measured value	Count rate correction, %
OSO 528	La	0.847	444 ± 33	439 ± 44	23.8
	Ce	0.481	782 ± 69	768 ± 77	12.1
	Nd	0.985	382 ± 25	384 ± 39	18.5
	Ho	20.3	18.5 ± 0.7	18.5 ± 2.7	44.7
	Lu	66.7	5.64 ± 0.23	5.80 ± 0.70 *	-
Oreas 100a	La	0.519	260 ± 9	267 ± 27	13.0
	Ce	0.292	463 ± 20	464 ± 47	7.9
	Nd	0.888	152 ± 8	152 ± 15	19.2
	Ho	28.1	4.81 ± 0.14	4.82 ± 0.78	59.6
	Lu	59.7	2.26 ± 0.11	2.33 ± 0.24	-

* Th content of OSO 528 is not certified and measured by INAA using GX5019 detector

Table 3 – REE content of the uranium-enriched CRMs, ppm

CRM	Element	U/REE ratio	Certified value	Measured value	Count rate correction, %
GBPG-1 (+U)	La	25.8	53.0 ± 8.0	52.2 ± 8.3	81.0
	Ce	13.1	104 ± 11	100 ± 14	75.8
	Nd	31.2	43.8 ± 7.3	44.3 ± 9.8	85.5
	Lu	4406	0.31 ± 0.05	0.32 ± 0.04	-
GBW 07110 (+U)	La	18.4	62.5 ± 2.5	57.8 ± 8.6	76.4
	Ce	9.8	117 ± 7	113 ± 16	70.7
	Nd	24.3	47.2 ± 2.5	47.3 ± 9.5	82.7
	Lu	2340	0.49 ± 0.04	0.51 ± 0.06	-

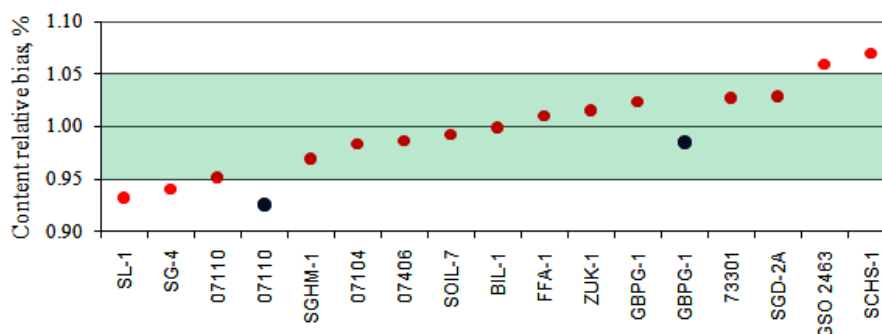


Figure 1 – Changing of La determination accuracy after 07110 and GBPG-1 CRMs were enriched with high uranium contents

Holmium measured contents of OSO 528 and OREAS 100a differed from the certified values by only 1% with ¹⁶⁶Ho count rate corrections amounted to 60%. Ho determination in the uranium-enriched CRMs appeared impossible due to very high U/Ho ratios. Lu determination in all CRMs as described above revealed the relative bias no more than 4%.

So, the CRM analysis made possible to demonstrate that the INAA ISM being used with the proposed way of uranium fission product corrections allows maintaining the precision of the method under the conditions of high U/REE ratios.

Table 4 - Comparison of La, Ce, and Nd analysis in the uranium ore samples by INAA ISM and X-ray fluorescence methods

Sample number	La content, ppm		Ce content, ppm		Nd content, ppm		U content, ppm	U/REE ratio			Count rate correction, %		
	INAA	XRF	INAA	XRF	INAA	XRF		U/La	U/Ce	U/Nd	La	Ce	Nd
1	2	3	4	5	6	7	8	9	10	11	12	13	14
1748	11.3 ± 1.8	12.2 ± 4.4	28.5 ± 3.9	29.7 ± 8.3	11.3 ± 2.1	12.0 ± 4.4	204 ± 24	17	6.9	17	81.9	66.7	80.3
3258	10.7 ± 2.6	12.1 ± 4.4	27.3 ± 4.0	29.8 ± 8.3	12.1 ± 2.8	12.9 ± 4.4	377 ± 45	31	13	29	91.2	77.4	86.1
1685	6.8 ± 3.0	5.8 ± 2.7	20.1 ± 3.5	17.4 ± 4.4	9.1 ± 3.2	7.7 ± 2.7	426 ± 51	73	24	55	95.7	86.3	91.9
8109	12.9 ± 2.4	12.8 ± 4.4	31.4 ± 4.3	32.4 ± 8.3	13.9 ± 2.6	14.3 ± 4.4	257 ± 31	20	7.9	18	86.9	70.0	80.9
282216	12.6 ± 2.1	12.0 ± 4.4	29.9 ± 4.0	27.4 ± 8.3	11.4 ± 2.0	11.4 ± 4.4	205 ± 25	17	7.5	17	82.0	62.6	77.6
282035	9.9 ± 2.5	10.5 ± 4.4	25.5 ± 3.8	24.3 ± 8.3	10.1 ± 2.6	10.2 ± 4.4	310 ± 37	29	13	30	91.7	78.4	88.0
7108	12.2 ± 2.0	10.4 ± 4.4	28.9 ± 4.0	26.8 ± 8.3	11.8 ± 2.2	11.2 ± 4.4	196 ± 23	19	7.3	18	83.2	67.9	80.5
3407	10.4 ± 1.6	11.2 ± 4.4	23.8 ± 3.5	25.4 ± 8.3	11.3 ± 2.5	12.7 ± 4.4	226 ± 27	20	8.9	18	80.1	77.4	85.0

1	2	3	4	5	6	7	8	9	10	11	12	13	14
1315	21.2 ± 4.3	23.5 ± 8.3	41.7 ± 6.0	38.0 ± 8.3	16.2 ± 3.7	17.4 ± 4.4	445 ± 53	19	12	26	88.6	75.1	85.9
440529	6.9 ± 1.2	6.9 ± 2.7	17.5 ± 2.6	16.5 ± 4.4	7.9 ± 1.8	8.1 ± 2.7	191 ± 23	28	12	24	84.0	77.9	85.7
267962	27.1 ± 3.6	26.9 ± 8.3	59.7 ± 7.9	57.5 ± 13	24.6 ± 3.6	26.4 ± 8.3	169 ± 20	6.3	2.9	6.4	54.1	47.6	62.5
4002	13.6 ± 2.0	12.5 ± 4.4	29.4 ± 4.1	27.5 ± 8.3	12.4 ± 2.3	11.8 ± 4.4	216 ± 26	17	7.8	18	75.0	71.6	80.6
5605	10.6 ± 2.5	10.2 ± 4.4	23.0 ± 3.6	23.1 ± 8.3	11.9 ± 3.0	11.4 ± 4.4	312 ± 37	31	13	27	90.7	81.8	87.6
310	44.3 ± 5.9	45.3 ± 8.3	110 ± 14	107 ± 23	46.6 ± 6.5	45.2 ± 8.3	198 ± 24	4.4	1.9	4.4	52.7	36.4	50.8
32029	28.7 ± 4.0	26.8 ± 8.3	72.2 ± 9.5	74.2 ± 13	27.8 ± 4.1	27.2 ± 8.3	258 ± 31	9.6	3.5	9.5	65.1	46.7	65.0
217	13.1 ± 2.0	12.5 ± 4.4	27.9 ± 3.8	28.1 ± 8.3	11.9 ± 2.1	13.5 ± 4.4	170 ± 20	14	6.1	13	76.1	67.0	77.1
2598	17.9 ± 2.5	19.4 ± 4.4	41.9 ± 5.6	43.0 ± 8.3	18.6 ± 2.9	18.5 ± 4.4	271 ± 32	14	6.3	15	69.1	55.2	68.5
2468	19.6 ± 2.7	19.4 ± 4.4	47.6 ± 6.5	43.9 ± 8.3	17.1 ± 3.4	18.3 ± 4.4	387 ± 46	20	8.8	21	65.3	66.9	82.3
2337	13.6 ± 2.0	12.4 ± 4.4	35.2 ± 4.6	31.8 ± 8.3	12.9 ± 2.0	13.1 ± 4.4	173 ± 21	13	5.4	13	77.1	49.7	68.5
3226	12.4 ± 1.8	11.9 ± 4.4	31.8 ± 4.3	28.6 ± 8.3	12.9 ± 2.2	11.6 ± 4.4	182 ± 22	15	6.4	16	71.7	62.4	76.6

Table 4 - (continuation). Comparison of La, Ce, and Nd analysis in the uranium ore samples by INAA ISM and X-ray fluorescence methods

Sample number	La content, ppm		Ce content, ppm		Nd content, ppm		U content, ppm	U/REE ratio			Count rate correction, %		
	INAA	XRF	INAA	XRF	INAA	XRF		U/La	U/Ce	U/Nd	La	Ce	Nd
2464	15.9 ± 2.4	17.6 ± 4.4	37.5 ± 5.2	40.2 ± 8.3	15.5 ± 3.2	17.4 ± 4.4	305 ± 37	17	7.6	17	78.0	71.2	83.9
315	17.7 ± 3.1	17.1 ± 4.4	46.5 ± 6.7	43.2 ± 8.3	15.4 ± 4.8	17.3 ± 4.4	525 ± 63	31	12	30	84.3	76.7	90.6
325	10.9 ± 2.3	12.9 ± 4.4	33.6 ± 5.1	33.1 ± 8.3	13.2 ± 4.4	13.3 ± 4.4	454 ± 54	35	14	34	89.2	81.1	91.3
2504	26.9 ± 3.7	24.9 ± 8.3	69.3 ± 9.1	60.8 ± 13	25.0 ± 3.8	23.3 ± 8.3	222 ± 27	8.9	3.7	9.5	60.1	48.6	67.7
32040	11.4 ± 2.2	12.7 ± 4.4	27.1 ± 4.2	29.3 ± 8.3	11.9 ± 3.4	12.1 ± 4.4	446 ± 54	35	15	37	87.5	82.1	89.5
323	10.8 ± 2.5	9.9 ± 2.7	28.7 ± 4.7	27.1 ± 8.3	9.8 ± 3.9	10.4 ± 4.4	406 ± 49	41	15	39	90.4	84.8	93.0
4009	17.3 ± 2.7	15.6 ± 4.4	34.7 ± 9.5	33.0 ± 8.3	14.1 ± 2.3	14.8 ± 4.4	202 ± 24	13	6.1	14	78.6	57.6	73.0
28104	14.8 ± 2.7	13.8 ± 4.4	37.3 ± 5.1	32.4 ± 8.3	14.1 ± 2.7	14.6 ± 4.4	273 ± 33	20	8.4	19	86.4	66.7	81.1
2505	17.9 ± 2.7	18.7 ± 4.4	44.9 ± 5.9	45.7 ± 8.3	17.1 ± 2.6	18.2 ± 4.4	257 ± 31	14	5.6	12	78.2	51.3	69.1
281912	14.3 ± 2.3	12.1 ± 4.4	33.4 ± 4.5	31.1 ± 8.3	12.4 ± 2.2	12.6 ± 4.4	209 ± 25	17	6.7	17	81.6	62.2	78.2
282219	6.4 ± 1.9	6.9 ± 2.7	17.4 ± 2.6	17.1 ± 4.4	6.5 ± 1.8	7.4 ± 2.7	196 ± 23	28	12	26	93.4	79.1	89.1
274820	10.1 ± 1.9	8.9 ± 2.7	24.4 ± 3.3	21.8 ± 8.3	9.8 ± 1.8	9.8 ± 2.7	181 ± 22	20	8.3	18	87.4	67.5	80.8
1409	21.0 ± 2.9	20.6 ± 8.3	41.8 ± 5.5	42.5 ± 8.3	17.8 ± 2.5	18.8 ± 4.4	118 ± 14	5.7	2.8	6.3	66.8	42.6	58.1
57111	14.3 ± 2.4	12.7 ± 4.4	32.0 ± 4.3	28.9 ± 8.3	11.8 ± 2.1	12.6 ± 4.4	197 ± 24	16	6.8	16	83.7	62.1	78.1
282923	12.2 ± 2.3	12.5 ± 4.4	28.7 ± 3.9	26.8 ± 8.3	9.0 ± 1.9	9.7 ± 2.7	179 ± 21	14	6.8	18	87.0	67.2	84.0

Table 5 - Rare earth element contents of the uranium ore samples by INAA and X-ray fluorescence methods, ppm

Sample number	Sc	Y	Pr	Sm	Eu	Gd	Tb	Ho	Tm	Yb	Lu
1748	2.40 ± 0.24	20.9 ± 8.3	5.3 ± 1.4	2.93 ± 0.35	0.585 ± 0.059	2.80 ± 0.56	0.381 ± 0.038	0.55 ± 0.19	0.236 ± 0.042	1.52 ± 0.15	0.223 ± 0.027
3258	2.21 ± 0.22	18.6 ± 4.4	5.7 ± 2.7	3.05 ± 0.37	0.663 ± 0.066	2.66 ± 0.54	0.507 ± 0.051	0.78 ± 0.38	0.237 ± 0.043	1.47 ± 0.15	0.290 ± 0.035
1685	2.73 ± 0.27	14.7 ± 4.4	3.5 ± 1.4	2.30 ± 0.28	0.544 ± 0.055	2.36 ± 0.48	0.348 ± 0.035	< 1	0.203 ± 0.037	1.31 ± 0.13	0.213 ± 0.026
8109	4.80 ± 0.48	17.5 ± 4.4	6.2 ± 2.7	3.43 ± 0.41	0.806 ± 0.081	2.75 ± 0.55	0.498 ± 0.050	0.67 ± 0.22	0.312 ± 0.056	1.63 ± 0.16	0.258 ± 0.031
282216	3.71 ± 0.37	15.1 ± 4.4	5.8 ± 2.7	3.24 ± 0.39	0.632 ± 0.063	2.92 ± 0.59	0.567 ± 0.057	1.3 ± 0.3	0.478 ± 0.086	2.69 ± 0.27	0.436 ± 0.052
282035	2.96 ± 0.30	11.4 ± 4.4	5.6 ± 2.7	2.72 ± 0.33	0.635 ± 0.064	2.22 ± 0.45	0.385 ± 0.039	0.88 ± 0.30	0.243 ± 0.044	1.38 ± 0.14	0.239 ± 0.029
7108	1.71 ± 0.17	12.1 ± 4.4	5.0 ± 1.4	2.55 ± 0.31	0.543 ± 0.055	2.16 ± 0.43	0.307 ± 0.031	0.92 ± 0.24	0.161 ± 0.029	1.02 ± 0.10	0.212 ± 0.025
3407	1.96 ± 0.20	11.4 ± 4.4	6.9 ± 2.7	2.73 ± 0.33	0.564 ± 0.057	2.52 ± 0.51	0.304 ± 0.030	1.0 ± 0.3	0.192 ± 0.035	1.17 ± 0.12	0.183 ± 0.022
1315	3.67 ± 0.37	27.2 ± 4.4	6.7 ± 2.7	4.58 ± 0.56	1.15 ± 0.12	4.42 ± 0.89	0.705 ± 0.071	< 2	0.384 ± 0.069	2.90 ± 0.29	0.611 ± 0.073
440529	1.29 ± 0.13	13.8 ± 4.4	3.5 ± 1.4	3.10 ± 0.37	0.422 ± 0.042	5.5 ± 1.1	1.39 ± 0.14	1.8 ± 0.5	0.96 ± 0.17	6.60 ± 0.66	0.99 ± 0.12
267962	13.9 ± 0.14	35.4 ± 8.3	10.0 ± 2.7	5.95 ± 0.71	1.29 ± 0.13	5.6 ± 1.2	0.905 ± 0.091	1.6 ± 0.4	0.489 ± 0.088	3.18 ± 0.32	0.465 ± 0.056
4002	2.09 ± 0.21	15.1 ± 4.4	5.8 ± 2.7	3.25 ± 0.39	0.640 ± 0.064	2.74 ± 0.55	0.377 ± 0.038	0.52 ± 0.27	0.210 ± 0.038	1.23 ± 0.13	0.205 ± 0.025
5605	2.17 ± 0.22	13.8 ± 4.4	5.5 ± 2.7	2.67 ± 0.32	0.689 ± 0.069	2.38 ± 0.48	0.335 ± 0.034	1.3 ± 0.4	0.229 ± 0.041	1.20 ± 0.12	0.248 ± 0.030
310	16.6 ± 0.17	47.4 ± 8.3	14.6 ± 4.4	10.7 ± 1.3	2.19 ± 0.22	9.2 ± 1.9	1.54 ± 0.15	2.9 ± 0.6	0.77 ± 0.14	4.34 ± 0.44	0.620 ± 0.074
32029	13.0 ± 1.3	38.1 ± 8.3	10.0 ± 2.7	5.82 ± 0.70	1.16 ± 0.12	4.49 ± 0.90	0.890 ± 0.089	2.2 ± 0.6	0.56 ± 0.10	3.27 ± 0.33	0.486 ± 0.058
217	2.11 ± 0.21	10.6 ± 4.4	6.5 ± 2.7	2.80 ± 0.34	0.681 ± 0.068	2.05 ± 0.41	0.331 ± 0.033	0.50 ± 0.20	0.156 ± 0.028	0.97 ± 0.10	0.156 ± 0.019
2598	2.23 ± 0.22	20.1 ± 8.3	6.7 ± 2.7	4.24 ± 0.51	0.749 ± 0.075	3.78 ± 0.76	0.615 ± 0.062	1.2 ± 0.3	0.269 ± 0.048	2.52 ± 0.25	0.470 ± 0.056
2468	4.68 ± 0.47	36.6 ± 8.3	7.0 ± 2.7	5.38 ± 0.65	0.845 ± 0.085	4.09 ± 0.82	0.681 ± 0.068	1.5 ± 0.4	0.389 ± 0.070	2.55 ± 0.26	0.404 ± 0.048
2337	2.58 ± 0.26	15.9 ± 4.4	4.6 ± 1.4	4.49 ± 0.54	0.565 ± 0.057	3.82 ± 0.77	0.709 ± 0.071	1.2 ± 0.3	0.507 ± 0.091	2.45 ± 0.25	0.569 ± 0.068
3226	1.93 ± 0.19	21.2 ± 8.3	4.7 ± 1.4	3.40 ± 0.41	0.643 ± 0.065	3.18 ± 0.64	0.545 ± 0.055	1.2 ± 0.3	0.322 ± 0.058	2.03 ± 0.21	0.310 ± 0.037
2464	2.84 ± 0.28	27.2 ± 8.3	6.4 ± 2.7	3.93 ± 0.47	0.647 ± 0.065	3.12 ± 0.63	0.687 ± 0.069	1.2 ± 0.3	0.472 ± 0.085	2.93 ± 0.30	0.490 ± 0.059
315	5.12 ± 0.51	22.7 ± 8.3	7.5 ± 2.7	4.42 ± 0.53	0.99 ± 0.10	4.14 ± 0.83	0.626 ± 0.063	1.4 ± 0.7	0.381 ± 0.069	2.16 ± 0.22	0.357 ± 0.043
325	3.30 ± 0.33	15.0 ± 4.4	5.1 ± 2.7	3.36 ± 0.40	0.714 ± 0.072	3.38 ± 0.68	0.688 ± 0.069	< 2	0.512 ± 0.092	3.28 ± 0.33	0.486 ± 0.058
2504	3.69 ± 0.37	33.7 ± 8.3	9.4 ± 2.7	6.47 ± 0.78	1.39 ± 0.14	6.0 ± 1.2	1.08 ± 0.11	1.8 ± 0.4	0.539 ± 0.097	3.38 ± 0.34	0.499 ± 0.060
32040	1.79 ± 0.18	18.0 ± 4.4	4.4 ± 1.4	3.41 ± 0.41	0.599 ± 0.060	3.20 ± 0.64	0.513 ± 0.051	< 2	0.273 ± 0.049	1.66 ± 0.17	0.262 ± 0.031
323	2.95 ± 0.30	13.3 ± 4.4	5.2 ± 2.7	3.09 ± 0.37	0.741 ± 0.074	2.45 ± 0.49	0.444 ± 0.045	< 2	0.248 ± 0.045	1.41 ± 0.14	0.258 ± 0.031
4009	4.01 ± 0.40	16.4 ± 4.4	6.3 ± 2.7	4.75 ± 0.57	0.716 ± 0.072	2.76 ± 0.56	0.470 ± 0.047	-	0.247 ± 0.044	1.29 ± 0.13	0.198 ± 0.024
28104	4.30 ± 0.43	17.5 ± 4.4	5.9 ± 2.7	3.92 ± 0.47	0.715 ± 0.072	3.03 ± 0.61	0.623 ± 0.062	1.1 ± 0.4	0.358 ± 0.064	2.07 ± 0.21	0.326 ± 0.039
2505	2.09 ± 0.21	23.7 ± 8.3	7.6 ± 2.7	4.18 ± 0.50	0.914 ± 0.092	4.11 ± 0.83	0.645 ± 0.065	0.64 ± 0.24	0.286 ± 0.051	1.61 ± 0.16	0.240 ± 0.039

Table 5 (continuation)											
Sample number	Sc	Y	Pr	Sm	Eu	Gd	Tb	Ho	Tm	Yb	Lu
281912	2.16 ± 0.22	16.0 ± 4.4	6.6 ± 2.7	3.36 ± 0.40	0.737 ± 0.074	3.13 ± 0.63	0.525 ± 0.053	1.0 ± 0.4	0.267 ± 0.048	1.88 ± 0.19	0.260 ± 0.031
282219	3.36 ± 0.34	14.1 ± 4.4	4.1 ± 1.4	2.20 ± 0.26	0.554 ± 0.056	2.14 ± 0.43	0.459 ± 0.046	1.4 ± 0.4	0.354 ± 0.064	2.16 ± 0.22	0.338 ± 0.041
274820	3.18 ± 0.32	16.3 ± 4.4	5.2 ± 2.7	2.67 ± 0.32	0.615 ± 0.062	2.41 ± 0.48	0.464 ± 0.047	0.85 ± 0.26	0.281 ± 0.051	1.62 ± 0.16	0.242 ± 0.029
1409	7.53 ± 0.75	19.2 ± 4.4	8.0 ± 2.7	3.94 ± 0.47	0.814 ± 0.082	3.36 ± 0.67	0.553 ± 0.056	0.89 ± 0.20	0.327 ± 0.059	2.11 ± 0.21	0.326 ± 0.039
57111	3.92 ± 0.39	15.9 ± 4.4	6.0 ± 2.7	2.98 ± 0.36	0.692 ± 0.069	2.51 ± 0.50	0.454 ± 0.046	1.3 ± 0.3	0.296 ± 0.053	1.88 ± 0.19	0.323 ± 0.039
282923	4.15 ± 0.42	17.8 ± 4.4	4.5 ± 1.4	2.88 ± 0.35	0.631 ± 0.063	2.76 ± 0.55	0.596 ± 0.060	1.6 ± 0.4	0.418 ± 0.075	2.56 ± 0.26	0.410 ± 0.049

La, Ce, and Nd contents of the uranium ore samples measured by two analytical techniques, as well as their uranium contents measured by INAA ISM, are presented in Table 4. Experimentally evaluated uranium to light lanthanide ratios and the values of count rate corrections of the corresponding radionuclides are adduced too. Table 5 summarizes the results of other REE analysis by INAA and XRF.

REE contents of the uranium ore sample turned out to be very low – from one third (Sc, La, Gd) to one (Pr) of their average crustal contents. Among the three lanthanides produced by uranium fission, U to La ratios were the highest (up to 73) with the analytical line count rate corrections coming up to 95%. The other ratios reached 24 for Ce and 55 for Nd with count rate corrections going to 86% and 93%, correspondingly. ^{166}Ho intensity corrections were also very high – from 82% to 95%. Moreover, in several cases when U content of the samples exceeded 400 ppm Ho analysis was impossible and only the upper limits were evaluated. Holmium determination in sample 4009 failed due to very high W content, about 0.095% by XRF.

The results of La, Ce, and Nd analysis by INAA and XRF were compared. Since the uranium ore samples were not selected at random, a nonparametric method of mathematical statistics should be used. The strict Mann-Whitney U -test for independent samples insensitive to variable distributions was employed. All three calculated values of the standard normal variable (0.52 is the maximum, $n = 35$) did not exceed its critical value 1.96 ($P = 0.95$) for the two-tailed test, hence the analytical results by two methods were admitted as statistically indistinguishable. Average values of La, Ce, and Nd contents of the uranium ore samples measured by INAA and XRF differed by 1% to 3.5%, while the pair-wise comparison showed discrepancy not more than 15%. Correlation between two assessments of light lanthanide contents of the uranium ore samples is depicted in Figure 2.

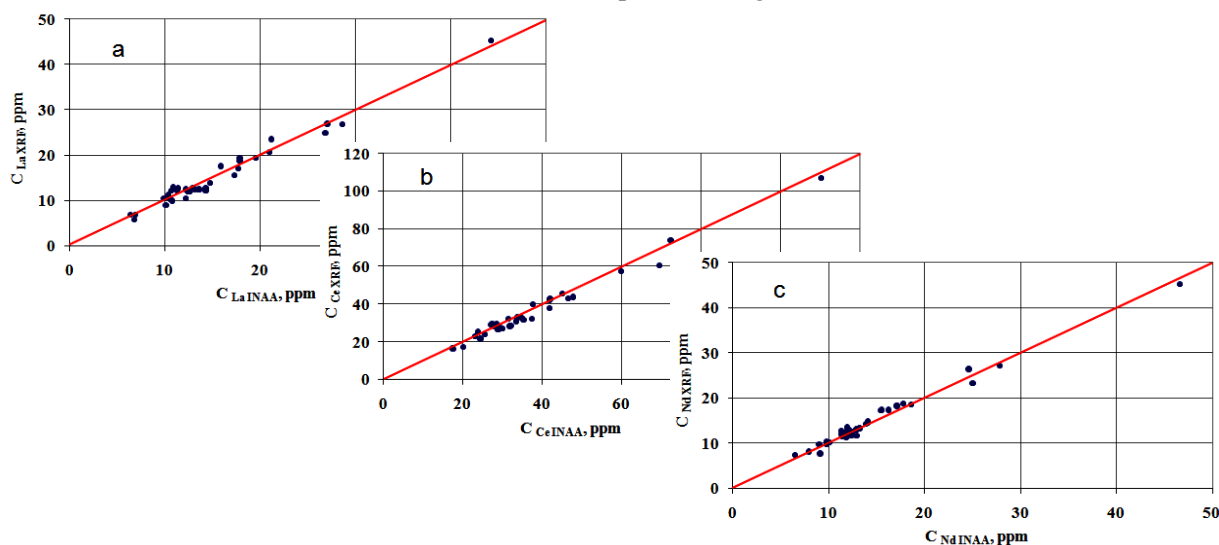


Figure 2 – Relationship between La (a), Ce (b), and Nd (c) contents of uranium ore samples by INAA and XRF

The uncertainty of REE contents by XRF corresponds to the prescribed values for every percentage interval according to the certified analytical technique and, as it was marked before, is independent of the uranium content. Unlike this, gamma-ray analytical peaks being highly impeded by the spectral interferences from the uranium fission products, INAA uncertainty sharply increases according to a hyperbolic law with the uranium content, when two close values are subtracted to receive corrected count rates. That is why uncertainty of these INAA results excites a special interest.

Main components of the uncertainty by INAA ISM were considered: Fe internal standard determination by XRF (8% for Fe contents (0.5-1)%), the methodical uncertainty of the comparator model (up to 10%), statistical uncertainties of Fe and other element counting, uncertainties of the contribution coefficients F_i evaluating. The full (enhanced) uncertainty was assessed according to the rules of error summing for correlated and uncorrelated random numbers.

Thus the evaluated enhanced uncertainty of La determination in the uranium ore samples varied from 13% to 39%, Ce – from 13% to 17%, and Nd – from 14% to 40% ($P = 0.95$). The assessed limit of La, Ce, and Nd quantitative determination by INAA (30% of uncertainty) corresponded to $\approx (91-94)\%$ correction of the analytical line intensities, and the limit of semi-quantitative determination (50% of uncertainty) corresponded to $\approx (95-97)\%$ correction. Since in the cases of high spectral corrections other sources of uncertainties become insignificant, 90% and 95% corrections may be accepted as the limits of quantitative (QL) and semi-quantitative (SQL) analysis. Larger correction gives only qualitative results (Figure 3).

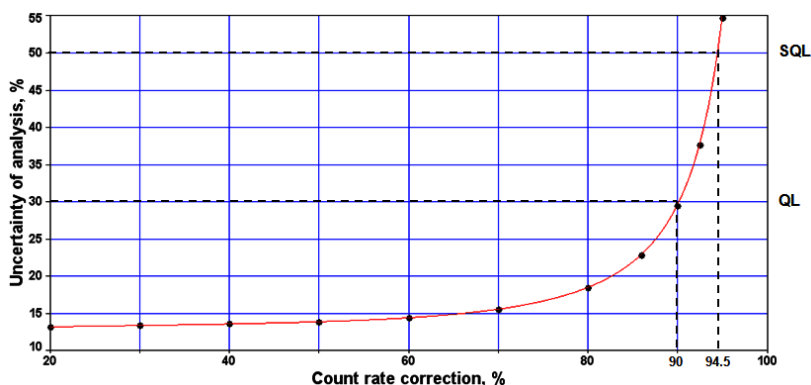


Figure 3 – Limits of Nd quantitative and semi-quantitative analysis depending on count rate correction

In spite of the planar detector resolves the sum peak ^{166}Ho and ^{133}Xe up to the background, ^{166}Ho count rate to calculate holmium content of the uranium ore samples according to the expression above turned out to be very sensitive to spectral treatment. Even a scrupulous approximation of the corresponding spectra parts is carried out (Figure 4), the uncertainty of holmium analysis by INAA evaluated in the same way varied from 21% to 55%. For more than a half from thirty five uranium ore samples holmium analysis appeared as not a quantitative one due to very high uranium fission product interference which is substantially higher than in case of light lanthanide determination. That is why holmium contents of the samples can be considered on the whole as approximate and should be treated together with the contents of other REEs only.

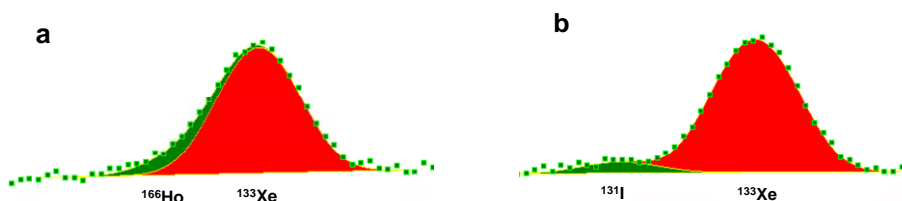


Figure 4 – A part of gamma-ray spectra treatment to calculate Ho content after 6 days (a) and 16 days (b) of decay, sample 3226

As INAA of the uranium ore samples for the rare earths which are not encumbered by the uranium fission products presented a standard procedure, it does not require a discussion.

Conclusion

Very low REE contents prevent from regarding the new sites under investigation of the Chu-Sarysu uranium provinces as a source of accompanying rare-earth primary product. However, taking the study as an example, the possibility and restrictions of REE analysis under the conditions of high U/REE ratios by XRF and INAA were investigated. The modified RLP-21T makes possible express determination of light lanthanides in the uranium ore samples with the acceptable accuracy but without any efforts which must be exerted in case of INAA ISM application. Since a new alternative of high-sensitive determination of light lanthanides in any rock samples by energy dispersive XRF appeared, it seems that INAA method should be intended to analyze heavier REE which can be accessible to XRF only in concentrates.

Acknowledgements

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И. Ю. Силачѐв

РГП «Институт ядерной физики», Алматы, Казахстан

АНАЛИЗ РЕДКОЗЕМЕЛЬНЫХ МЕТАЛЛОВ В УРАНОВОМ СЫРЬЕ НЕЙТРОННО-АКТИВАЦИОННЫМ И РЕНТГЕНОФЛУОРЕСЦЕНТНЫМ МЕТОДАМИ

Аннотация. Одним из приоритетных направлений экономики Республики Казахстан в XXI веке является развитие редкоземельной отрасли цветной металлургии. Высоким содержанием редкоземельных металлов (РЗМ) характеризуются некоторые урановые месторождения РК. Для определения РЗМов в минеральном сырье наиболее подходят неdestructивные методы – инструментальный нейтронно-активационный (ИНАА) и рентгенофлуоресцентный (РФА) методы анализа. Нейтронно-активационное определение ряда РЗМов в урановой руде существенно затруднено продуктами деления урана, образующими прямые (La, Ce и Nd) и спектральные интерференции. Анализ РЗМов в геологических образцах методом РФА обычно обладает недостаточной чувствительностью и ограничен в основном легкими лантаноидами.

В данной работе на примере образцов руды Шу-Сарысувской урановорудной провинции рассмотрена возможность метода ИНАА для определения РЗМов в условиях значительных интерференций, вызванных высоким содержанием урана. Рентгенофлуоресцентный анализ легких лантаноидов выполнен на уровне кларковых содержаний с помощью модифицированного энергодисперсионного спектрометра РЛП-21Т по К-серии их характеристического рентгеновского излучения. Сравнение содержания La, Ce и Nd в образцах урановой руды методами РФА и ИНАА с использованием критерия Манна-Уитни показало, что различие в результатах статистически незначимо. На основании анализа бюджета неопределенности оценена максимальная величина коррекции скорости счета аналитических линий, соответствующая пределу количественного и полуколичественного определения La, Ce и Nd методом ИНАА. Сделан вывод о предпочтительности метода РФА для экспрессного анализа легких лантаноидов в горных породах.

Ключевые слова: нейтронно-активационный анализ, рентгенофлуоресцентный анализ, редкоземельные металлы, урановая руда

И.Ю. Силачѐв

«Ядролық физика институты» РМК, Алматы, Қазақстан

НЕЙТРОН-АКТИВАЦИЯЛЫҚ ЖӘНЕ РЕНТГЕНФЛУОРЕСЦЕНТТІК ӘДІСТЕРІ АРҚЫЛЫ УРАНДЫҚ ШИКІЗАТТАҒЫ ЖЕРДЕ СИРЕК КЕЗДЕСЕТІН МЕТАЛДАРДЫ ТАЛДАУ

Аннотация. XXI ғасырдағы Қазақстан Республикасы экономикасының басым бағыттарының бірі түсті металлургияның жерде сирек кездесетін саласын дамыту болып табылады. ҚР кейбір уран кен орындары жерде сирек кездесетін металдардың (ЖСМ) жоғары құрамымен сипатталады. Минералдық шикізатта ЖСМ-дың болуын анықтау үшін декұрылымдық емес әдістер – аспаптық нейтрон-активациялық (АНАТ) және рентгенфлуоресцентті талдау (РФТ) әдістері ең қолайлы болып саналады. Уран кеніндегі бірқатар ЖСМ-ды нейтрон-активациялық анықтау тіке (La, Ce және Nd) және спектрлік интерференциялар түзілетін уран бөлу өнімдерімен айтарлықтай қиындық тудырады. Геологиялық үлгілердегі ЖСМ-ды РФТ әдісі арқылы талдау әдетте сезгіштігі жеткіліксіз және негізінен жеңіл лантаноидтермен шектеледі.

Осы жұмыста шалғай жатқан Шу-Сарысу уран кені кендері үлгілері ретінде уранның жоғары мөлшерімен туындаған елеулі интерференциялар жағдайында ЖСМ-ды анықтау үшін АНАТ әдісінің мүмкіндігі қарастырылған. Жеңіл лантаноидтерді рентгенфлуоресцентті талдау олардың сипаттамалық рентгендік сәулеленуінің К-топмасы бойынша РЛП-21Т модификацияланған энергия дисперсиялық спектрометрі көмегімен кларктық құрамы деңгейінде орындалды. Манна-Уитни критерийін пайдалана отырып, РФТ және АНАТ әдістерімен уран кені үлгілеріндегі La, Ce және Nd мөлшерін салыстыру нәтижелердегі айырмашылық статистикалық тұрғыдан болашы екенін көрсетті. Белгісіздік бюджетін талдау негізінде АНАТ әдісі арқылы La, Ce және Nd сандық және жартылай сандық анықтау шегіне сәйкес аналитикалық сызықтарды санау жылдамдығын түзетудің максималды мәні бағаланды. Тау жыныстарындағы жеңіл лантаноидтерді жедел талдауда РФТ әдісінің артықшылығы жөнінде қорытынды жасалды.

Түйін сөздер: нейтрон-активациялық талдау, рентгенфлуоресцентті талдау, жерде сирек кездесетін металдар, уран кені.

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