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OPTIMIZATION OF THE METHOD FOR INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INAA) OF NATURAL OBJECTS

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Abstract. Experimentally, optimization of the time modes of neutron activation analysis of samples of natural media was carried out, based on the nuclear physical characteristics of the studied and “interfering” radionuclides. The detection limit and accuracy of element determination were chosen as a criterion for optimizing the method.

Keywords: neutron activation analysis, optimization of time modes, limit of determination, gamma radiation, gamma spectrometry, half-life, radionuclide activity, element isotope, analysis error, radioactive decay, analytical peak, statistical error, photo peak area, “interfering” radionuclides.

Introduction. Modern problems of ecology, as well as control and protection of the environment, require a comprehensive study of the content, distribution, distribution, accumulation, forms of occurrence and migration of elements in atmospheric air, sediments,

natural and waste waters, soils and other natural objects [1].

When solving the problem of control and protection of the natural environment by physical and chemical methods, it is necessary to determine 50-60 elements, with a detection limit of 10-7-10-14 g, with samples of 0.1-1.0 g. Among the analytical methods, nuclear -physical methods and mainly neutron activation analysis (NAA), characterized by a low detection limit, multiple elements and high productivity.

Currently, there are a number of works on optimization of temporary modes of INAA [2], where multiple irradiation, “cooling” and measurement are proposed. In our case, it was necessary to conduct a highly sensitive mass analysis for a certain number of toxic anthropogenic and natural elements in a large number of samples. It should be emphasized that thanks to the automation of the aerosol sampling process, we were able to collect relatively high samples on the filters, which in turn influenced the INAA methodology for atmospheric aerosols, namely, the measurement time of long-lived isotopes was reduced by 2.5-3 times, with an increase in the number of elements being determined and reducing the analysis error.

Materials. Using the example of the analysis of atmospheric aerosols, we will consider the optimal INAA scheme. The task of optimizing the time modes of analysis is to identify such conditions for conducting INAA that ensure the simultaneous determination of the content of the maximum number of elements with the smallest error. Methods for optimizing the INAA of natural samples are reduced to the study of certain functions and are based on solving the equation of the following form:

$$a = f(\bar{x}, \bar{p}, \bar{q}) \qquad (1)$$

where, a is the optimization function (detection limit, error, etc.);

$\bar{x}$ - a set of optimized parameters - factors (irradiation, cooling and measurement time);

$\bar{p}$ - a set of nuclear-physical data determined by the sample matrix (mass of isotopes, their atomic weights, reaction cross-section, decay constants, etc.);

$\bar{q}$ - a set of characteristics of the analytical complex equipment (intensity of activating radiation, recording efficiency of the equipment, etc.).

As a rule, due to the complex nature of function (1), optimization of analysis conditions is carried out on an electronic computer (computer) or experimentally.

We carried out optimization of the time modes of analysis experimentally, based on the nuclear physical characteristics of the studied and interfering radionuclides. The detection limit and accuracy of element determination were chosen as optimization criteria for INAA. The proposed optimal INAA scheme using atmospheric aerosols as an example is presented in Fig. 1.

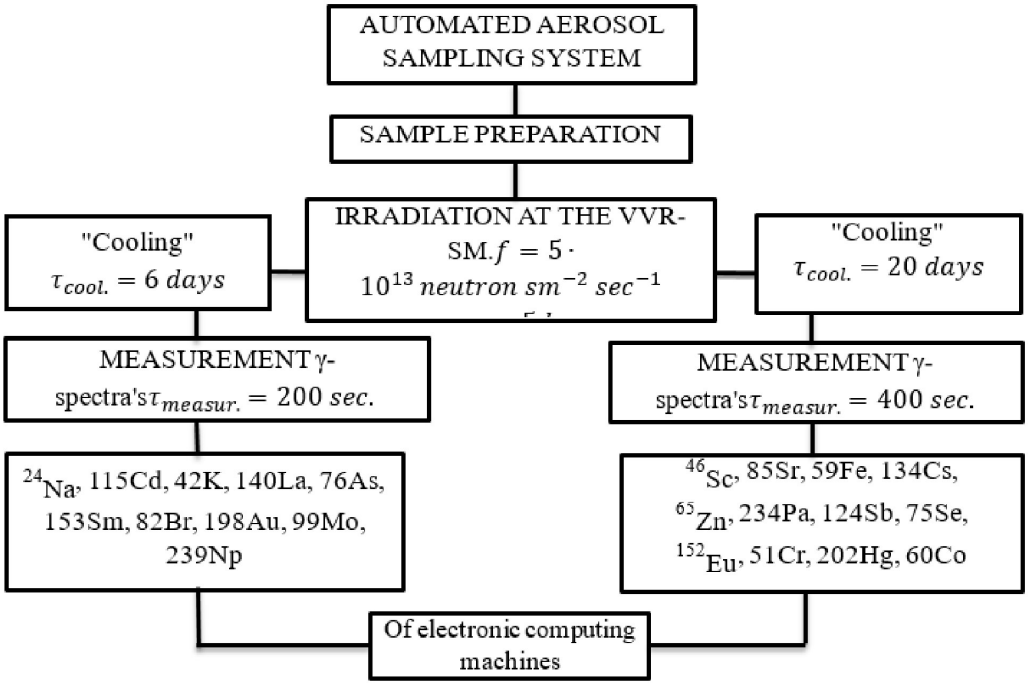

Fig.1. INAA scheme for urban atmospheric aerosols

The determination of Mo, Sm, Au and As in soils and the dissolved phase of water is difficult due to the “interfering” activity of 24Na and 82Br. To increase the sensitivity and accuracy of determining these elements, we resorted to varying the neutron spectrum. It is known that the activation cross section of some elements has a resonant character in the above-thermal region of the neutron spectrum [2]. When irradiated in a nuclear reactor, using a cadmium filter, the flux of thermal neutrons at the location of the sample is suppressed and activation will be carried out by resonant neutrons of a wide spectrum of energy. The determination of these elements was carried out by irradiating the test sample in a cadmium channel for 5 hours in a flow of f = 5·1013 neutron cm-2 sec-1. After “cooling” for 6 days for medium-lived and 20 days for long-lived radionuclides, measurements were carried out along the corresponding gamma lines on an installation containing two spectrometric paths with Ge(Li) detectors with a volume of 80 cm3 and an ISKRA-minicomputer. 226 using the SOSNAA program [3], developed by employees of the activation analysis laboratory of the Institute of Nuclear Physics of the Academy of Sciences of the Republic of Uzbekistan. The resolution of the equipment in the region of the 60Co peak (1332.5 keV) was 3.2 keV.

Mathematical processing of the obtained spectrometric information included spectrum smoothing, automatic search for peaks, determination of the areas of photo peaks and background, and calculation of the statistical error of determination.

In Fig. Figures 2-3 show some characteristic gamma spectra of the samples of natural environmental objects we studied. The overlap of gamma lines of different radionuclides leads to difficulties in interpreting spectroscopic information.

Such difficulties arose in the case of the following pairs of radionuclides: 134Cs - 124Sb, 153Sm - 239U, 65Zn - 46Sc, 203Hg - 75Se, 46Sc - 110mAg and 85Sr - 65Zn. In the first case, we proceeded as follows. Although the 603 keV line has almost one hundred percent yield of gamma quanta for both 134Cs and 124Sb, to determine these radionuclides we used

pure lines 796 keV and 1691 keV with lower yields of gamma quanta (30 % and 50 %, respectively), which was quite enough with our samples. In other cases, where the second radionuclide in each pair was interfering with the first, the ratio of the two characteristic lines of the second radionuclide in its standard was used to determine its contribution to the total peak with the first radionuclide in the test sample.

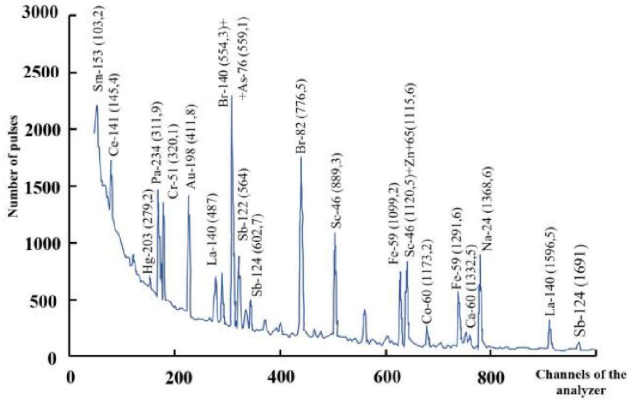

Fig.2. Gamma spectrum of atmospheric aerosol:
тirrad.= 5$hours$; $\tau_{cool.} = 6\ days$; $\tau_{measur.} = 200\ sec.$

The contribution of the second radionuclide found in this way was subtracted from the indicated peak to determine the contribution of the first.~

It should be noted that the successful use of INAA in monitoring the quality of the natural environment requires a comprehensive study and assessment of the influence of various sources of errors, as well as an assessment of the metrology of the developed methods.

The detection limit of INAA elements is defined as [4]

$$C_{min} = \frac{k\sqrt{I_f \cdot C_x}}{I_x} \qquad (2)$$

where, is the detection limit of the desired element; C_{min}
C_x- content of the element under study in the sample:
I_x- useful activity (area under the photo peak) of the element being determined;
I_f- interfering activity (base area under the peak);
k- confidence factor (3÷10).

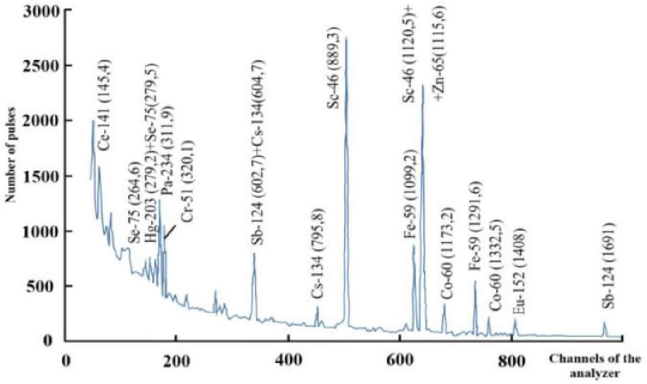

Fig.3. Gamma spectrum of atmospheric aerosol:
тirrad.= 5$hours$; $\tau_{cool.} = 20\ days$; $\tau_{measur.} = 400\ sec.$

The concentration of elements is determined by the formula:

$$C_{xi} = [I_{\text{обр}} \cdot m_{\text{ЭТ},i}] / [I_{\text{ЭТ},i}\, V], \qquad (3)$$

where, is the concentration of the i-th element; C_{xi}

$I_{\text{обр.}}$- area of the analytical peak of the i-th element in the breakdown;

$m_{\text{ЭТ},i}$ - mass of the i-th element in the comparison sample;

$I_{\text{ЭТ},i}$ - area of the analytical peak of the i-th element in the comparison sample;

V- volume of water or pumped air or mass of soil.

Taking (3) into account, it is easy to show that

$$C_{min} = \frac{k\sqrt{I_f \cdot m_{et}}}{I_{et} \cdot V} \qquad (4)$$

A generally accepted way to assess the correctness of a method is to use certified reference materials to assess and control the reproducibility and correctness of analytical results [5].

The similarity of the physicochemical composition of standard samples and the analyzed natural objects results in the same magnitude of contributions to the error made due to the influenceinterferingreactions, self-shielding effect, etc. ExceptIn addition, the difference in the geometric shapes of the standard and the sample is minimal, which also reduces the measurement errors of naturalsamples. As standard samples foranalysisaerosols, soils, waters and precipitation, certified standard samples SV, SOV1-SOV5 and SP1-SPZ were used.

The discrepancy between our and recommended data averages 5-25%. Long-term and routine analysis of approved domestic and foreign reference materials indicates good reproducibility and correctness of INAA. The productivity of INAA of natural objects according to the proposed scheme is 500-600 element determinations per shift.

Conclusions:

1) The recommended optimal time regimes ensured the achievement of detection limits comparable to the literature data with a shorter duration of irradiation and “cooling” of samples, which is important in geophysical and environmental studies;

2) According to the proposed optimal scheme, the concentrations and forms of occurrence of more than twenty elements in adjacent natural environments are determined with a detection limit of 10-6-10-13 g/g, a relative standard deviation of 0.05-0.25;

Analysis of the metrological parameters of INAA allows us to conclude that there is a low detection limit, satisfactory reproducibility and accuracy, which favors the use of the method in quality control of various natural environments.

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ISOTHERM OF AMMONIA ADSORPTION IN ZEOLITE CaA (M-22)

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Abstract:

Objective. The article presents experimentally obtained values of the adsorption isotherm of ammonia molecules in synthetic zeolite CaA (M-22) at a temperature of 303 K. Isotherm values were measured using an improved microcalorimeter connected to a universal high vacuum apparatus. The differential values of the free energy were calculated from the equilibrium values of the pressure. In CaA (M-22) zeolite, a regular relationship between the amount of adsorption and energy properties of ammonia molecules, as well as the sorption mechanism from the initial area of adsorption to the area of condensation heat of ammonia and the regularity of ammonia molecules filling the volume of zeolite were determined. Under experimental conditions, the adsorption capacity of this zeolite for ammonia was found to be 10.5 mmol/g in 1 g of zeolite. It was determined that 40% of the total adsorption is sorbed up to the equilibrium pressure of 1 torr, 50% up to 6 torr pressure, 60% up to 24 torr pressure, 65% up to 50 torr pressure and 100% at 467 torr pressure. The adsorption isotherm was recharacterized by the three-state equation of micropore volumetric saturation theory (VMOT) and it was shown that the theoretically calculated values were in perfect agreement with the experimentally obtained values.

Methods. The adsorption isotherm was measured with high accuracy (enthalpy 0.2 μJ, small values of pressure with an accuracy of 10^{-5} torr) and stability using a system consisting of a Tiana-Calve type DAK-1-1A differential automated microcalorimeter connected to a universal high-vacuum device. The adsorption-calorimetric method used in the research allows obtaining molar thermodynamic characteristics, as well as revealing the detailed mechanisms of adsorbent-adsorbate and adsorbate-adsorbate sorption processes. Adsorption measurements and dosage of adsorbate were performed using a high-vacuum adsorption device. The device allows dosing of adsorbate by gas-volume and volume-liquid methods. B627 diaphragm baratron was used to measure the equilibrium pressure up to $10^{-5} \div 0{,}8$ torr, U manometer was used to measure the pressures at R>0.8 torr. The adsorption-calorimetric method allows to study nano-, micro-, mesostructured adsorbents and their surface-active surfaces, to reveal in detail the main thermodynamic properties and mechanisms of adsorption processes in which adsorbents occur.

Results. Adsorption of ammonia molecules on CaA (M-22) zeolite at a temperature of 303 K from the area of small saturations to the heat of condensation of ammonia from the initial area to the saturation pressure was found that the enthalpy values of ammonia adsorption in this zeolite are almost 40-50 kJ/mol higher than the adsorption enthalpy of polar and non-polar molecules of different nature. During the adsorption process, it was found that ammonia molecules interact with Na^+ and Ca^{2+} cations in S_I and S_{II} positions of zeolite.

Conclusion. The results of adsorption-calorimetric research obtained on the basis of experience allow to obtain the main thermodynamic functions of the studied systems, which are necessary for the

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