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M.M. Burkitbayev[1], U.Zh. Dzhusipbekov[2], G.O. Nurgalieva[2], Z.K. Bayakhmetova[2], D. Duisenbai[2], Sh.N. Nazarkulova[1*], I.V. Matveyeva[1, 3], Ye.Yu. Yarovaya[1]

[1]Al-Farabi Kazakh National University, Almaty, Kazakhstan
[2]JSC A.B. Bekturov Institute of Chemical Sciences, Almaty, Kazakhstan
[3]Kazakh-British Technical University, Almaty, Kazakhstan
*e-mail: sholpan.nazarkulova@kaznu.edu.kz
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Assessment of natural radionuclides content in humate-containing fertilizers

Abstract. Humic substances (HS) possess significant physiological activity and play a crucial role in enhancing soil properties and plant productivity. This study is devoted to the determination of the content of natural radionuclides in humate-containing samples obtained from natural raw materials of Kazakhstan. Five samples, including both solid and liquid fertilizers, were prepared and analyzed. Key parameters such as elemental composition, content of carboxyl and phenolic groups, and the yield of free humic acids were determined. The concentrations of uranium-238, uranium-234, thorium-232, thorium-230, thorium-228, polonium-210, and lead-210 isotopes were measured using alpha-spectrometric and alpha-beta radiometric methods. Results indicated that the liquid fertilizer exhibited the lowest radionuclide activities, while the solid samples showed varying levels of radioactive isotopes, with the highest activity recorded in sample A2. Nonetheless, the specific activities of all measured radionuclides complied with the national radiation safety standards (SanPiN 2.6.1.2523-09), confirming the fertilizers' safety for agricultural use. This work highlights the importance of radiation monitoring in fertilizers and the potential of humate-containing materials as environmentally friendly agricultural enhancers. The findings contribute to the development of sustainable agricultural practices through the safe utilization of natural organomineral fertilizers derived from local raw materials.
Keywords: Humic substances, natural radionuclide, fertilizers, uranium series, activity concentration.

Introduction

Fertilizers can increase crop yields by an average of 50-80% by eliminating nutrient deficiencies in the soil. Nutrients include macronutrients (nitrogen, phosphorus, potassium), mesonutrients (sulfur, calcium, magnesium) and micronutrients (iron, molybdenum, zinc, boron, cobalt, copper, manganese). These elements are part of fertilizers, divided into 5 main types: nitrogen, potassium, phosphorus, micronutrients and complex or compound mineral fertilizers [1].

Humic substances (HS) are characterized by high physiological activity due to their structural features and physicochemical properties [2-5]. Humic substances activate the metabolism and reproduction of beneficial soil microflora, enhance the protective mechanism of plants against the effects of unfavorable physical, chemical and biological factors, and contribute to increased productivity of agricultural crops and product quality. In addition, HS have a complex effect on the soil, improving its physical, chemical and biological properties, maintaining the organo-mineral balance of soils and promoting the restoration and increase of soil fertility. Liquid humate-containing fertilizers containing a balanced set of biologically active humic substances, macro- and microelements have high biological efficiency and minimal negative impact on the ecosystem and are used to stimulate physiological processes in plants, increase the yield and quality of agricultural products, increase the absorption of nutrients and plant resistance to chemical, physical and biological stress [5-8].

Along with increasing crop yields, the use of fertilizers has negative consequences, such as soil pollu-

tion and potential contamination of agricultural products, and the entry into the food chain of elements with high toxicity associated with the specificity of specific fertilizers, for example, phosphorus fertilizers contain heavy metals, radioactive isotopes of some metals, and potassium fertilizers often contain impurities of chlorine and a radioactive isotope of potassium, while excessive use of nitrogen fertilizers causes the accumulation of nitrates in plants [9]. Thus, an important characteristic of any fertilizer, both mineral and organic, is quality, which implies a minimum content of pollutants in the composition and control of fertilizers for their content.

The uranium industry has played a significant role in the economy of modern Kazakhstan, which has been a major source of uranium for more than 50 years [10]. The long-term and intensive activities of the uranium industry have left a legacy of environmental pollution [11]. The well-developed uranium industry affects the radio ecological situation of the region [12], where three isotopes of uranium occur in nature. These include U-238, U-235, and U-234. The potential harmfulness of radionuclides is based on their long half-lives and chemical behavior [13].

Determining the content of radioactive elements in fertilizers helps assess radiation safety and compliance of fertilizers with sanitary rules and hygienic standards [14-18]. Due to usage of fertilizers can increase the number of radionuclides in the soil and groundwater and their subsequent entry into the human body. Broad meanings of radionuclide activity concentrations vary among countries, and there is no specific legislation defining maximum permissible limits for radioisotopes in fertilizers.

Materials and methods

Sample A1 was obtained by interaction of the tetrahydrate ammonium heptamolybdate [$(NH_4)_6Mo_7O_{24}$.$4H_2O$)] with 1% sodium humate solution at a temperature of 40°C for 60 min at a ratio of the initial components of 0.5:100 (weight/volume). Ammonium molybdate was purchased from LLC Scientific and Production Firm “Baltic Manufacture” (St. Petersburg, Russia).

Sample A2 was obtained from brown coal of the Oikaragay deposit (Almaty region) using 2% NaOH (1:8 weight/volume) for 1 hour at 25°C with vigorous stirring and its precipitation by adding hydrochloric acid to pH 1-2. The precipitate was filtered, washed with distilled water until a negative reaction for chloride ion, and dried in a drying cabinet at a temperature of 70-80°C.

Sample A3 was extracted from brown coal of the Oikaragay deposit (Almaty region). The extraction process was carried out with a 2% NaOH solution (1:8 weight/volume) for 1 hour at 25°C with vigorous stirring. The suspension was centrifuged, and the filtrate was dried in a vacuum oven at 105°C.

Sample A4 is brown coal from the Oikaragay deposit in the Almaty region.

Sample A5 was obtained by extraction from brown coal of the Oikaragay deposit (Almaty region) with a 2% solution of NH_4OH (1:8 weight/volume for 1 hour at 25°C with vigorous stirring. The suspension was centrifuged, and the filtrate was dried in a vacuum oven at 105°C.

In humate-containing samples were determined the release of free humic acids by chemical analysis [19], the amount of carboxyl groups and phenolic hydroxyls was determined using the calcium acetate and barite methods [20]. The elemental composition of humate-containing samples was determined using a Thermo analyzer FlashSmart (Thermo Scientific company, USA). The molybdenum content was determined using an inductively coupled plasma atomic emission spectrometer of the iCAP PRO XP Duo brand (Thermo Fisher Scientific, USA). The characteristics of humate-containing samples are given in the Table 1.

Table 1 – Characteristics of humate-containing samples

Sample	Elemental composition, on a dry ash-free basis, %					Content of acidic groups, on a dry ash-free basis, mg-eq/g		Yield of free humic acids based on dry ash-free basis, %
	C	H	N	O	Mo	COOH	Phenolic hydroxyl	
A1	54.51	3.80	5.23	35.64	0.27	1.76	0.70	38.40
A2	60.1 4	4.14	1.4 1	28. 32	-	1. 18	1.67	81.07
A3	42.40	3 .02	1.2 9	30.6 4	.	2 .12	2.87	74.10
A4	53.61	4.21	1.43	28.92	.	0.33	2.40	55.00
A5	55.63	3.44	1.57	33.32	-	1.30	0.75	54.12

Determination of uranium isotopes in solid humate-containing and liquid organomineral fertilizers by the alpha-spectrometric method was carried out in three stages:

1. Decomposition of the sample (in the case of liquid samples, the concentration method was used);

2. Preliminary radiochemical preparation of samples;

3. Alpha spectrometric determination of uranium isotopes.

Decomposition of solid humate-containing samples was carried out by treating them with solutions of strong acids after roasting at high temperatures to remove organic substances. Concentration of uranium isotopes in liquid fertilizers was carried out by evaporation in a glass beaker over low heat, preventing the sample from boiling.

Preliminary radiochemical preparation of samples included concentration of uranium isotopes from a solid sample, extraction separation from interfering radionuclides, and preparation of a counting sample by electrolytic method.

The samples were isolated and purified by extraction with tributyl phosphate in toluene. The nitric acid solution containing uranium isotopes, concentrated in a smaller volume, was transferred to a separatory funnel, freshly purified TBP solution in toluene was added and extracted for 5 minutes.

Next, uranium was re-extracted, for which the organic phase was washed with distilled water. The combined aqueous re-extract was evaporated to dryness, moistened with concentrated nitric acid, and again evaporated to dryness to remove traces of organic matter.

The preparation of the counting sample was carried out by the method of electrolytic deposition of uranium isotopes on a steel disk. To achieve the best peak resolution on the alpha-spectrogram, it is necessary that the uranium isotopes be applied to a polished steel substrate in a thin, uniform layer. For this purpose, the dry residue containing uranium isotopes was dissolved in a mixture of HNO_3, Trilon B, NH_4Cl, $(NH_4)_2C_2O_4$. The pH of the solution was maintained at 7-8. Then, the solution containing uranium isotopes was transferred in bulk to an electrolytic cell and the electrodeposition of purified isotopes was carried out at a constant current of 1 A.

To determine thorium isotopes in the liquid sample, radiochemical preparation was carried out, which included:

- concentration of thorium isotopes,
- separation and isolation of thorium isotopes (separation of macrocomponents, polonium, radium, plutonium, uranium),
- preparation of a counting sample.

For solid samples, radiochemical preparation consisted of:

- decomposition,
- separation and isolation of thorium isotopes (separation of macrocomponents, polonium, radium, plutonium, uranium),
- preparation of a counting sample.

The method is selective and is focused on determining only the isotopes of thorium-232, thorium-230 and thorium-228. The possible interfering effect of radionuclides with similar alpha radiation energies (uranium-234, uranium-238, polonium-210, radium-226, plutonium-238, americium-241) was eliminated during the radiochemical preparation of samples.

The separation and extraction of thorium isotopes from accompanying interfering alpha-emitting natural radionuclides with similar alpha-radiation energies (isotopes of uranium, radium, polonium, etc.) was carried out on ion-exchange resins. The ion-exchange resins were pre-treated, including obtaining a resin of a certain fraction, removing interfering components, and bringing the resin to the required pH value.

The preparation of the counting sample was carried out by electrolytic deposition on a low-background disk at a constant current (I = 1 A).

Losses during analysis were monitored by adding a thorium-234 solution to the sample, followed by comparison of the integral beta particle count rate from the working preparation (counting sample) and from a specially prepared comparison sample of identical geometry.

Uranium and thorium isotopes were measured using the alpha-spectrometric method on the Alpha – analyst alpha -spectrometer from Canberra with the appropriate software. This method allows identifying an isotope by energy and intensity, determining its activity and content in the sample. For this purpose, the device was pre-calibrated by energy and efficiency. Background values were determined before the measurement.

The alpha spectrometer “Canberra” (USA) is a specialized spectrometric system designed for detailed analysis of alpha-emitting radionuclides (in particular, uranium and thorium isotopes). The device includes a silicon semiconductor detector of the PIPS (Passivated Implanted Planar Silicon) type, placed in a vacuum chamber, which ensures low background and high energy resolution (no worse than 20-25 keV). The advantage of the design is high sensitivity when measuring low-energy alpha quanta (from 3 to 8 MeV), which is especially important when study-

ing natural radionuclides in organo-mineral matrices. The counting sample was prepared by applying a thin (up to 100 μg/cm²) precipitant to a disk carrier, followed by drying and stabilization of the layer. The spectrum clearly showed peaks of alpha decay of U-238, U-234, Th-232 isotopes and their daughter products, which allowed for qualitative and quantitative analysis at the level of Bq/kg units.

To determine polonium-210 and lead-210 isotopes in solid humate-containing and liquid organomineral fertilizers by the alpha-beta radiometric method, a variant of simultaneous spontaneous electrochemical precipitation of Po-210 and Bi-210 (= Pb-210) in radiochemically pure form on substrates made of stainless corrosion-resistant Ni – Ti steel was implemented. In this case, the precipitation of Pb-210 on Fe is practically excluded, and its determination was carried out by beta radiation of daughter Bi-210. Radionuclides are leached from 5 g of air-dry crushed sample with a mixture of HNO_3, HCl and H_2O_2, after which $HClO_4$ is added to the filtrate and the solution is evaporated to wet salts to remove traces of HNO_3. The salts are dissolved in 0.5 M HCl and then Po-210 and Bi-210 are deposited on one side of the steel substrate for 2 hours. Isotope measurements were carried out in the range of 10-36 hours after spontaneous currentless deposition on an alpha-beta radiometer with a silicon detector for measuring low activities UMF-2000 (JSC NPP Doza, Moscow, Russia), as recommended in the methodology (MI No. FR 15382).

The UMF-2000 radiometer is the device equipped with a low-background counter with a protection system from external radiation and an automated recorder of counting information. The design of the radiometer includes a removable cassette with a sample, located in a fixed geometry opposite the counting device. Due to its high sensitivity (detection threshold of about 0.05 Bq/cm² with a 10-minute exposure) and ease of use, the UMF-2000 is optimal for this work. Its use is especially justified when it is necessary to confirm data obtained by the spectrometric method, as well as for preliminary screening of the radioactivity level.

The combined use of two methods ensured the reliability of measurements, expanded the analytical range (in terms of registration energies) and made it possible to obtain substantiated data on the distribution of natural radionuclides in the studied fertilizers. The relevance of using these devices in this work is due to their high reliability, representativeness of the results and compliance with modern requirements for radiometric control of substances of natural origin.

Results and discussion

The results of radiometric analysis of five samples of humate-containing preparations are presented in Figures 1-7. The highest activity values of uranium and thorium radionuclides were recorded in sample A4, where the activity of U-238 reached 202±14 Bq/kg, and U-234 – 245±15 Bq/kg. At the same time, the minimum levels of these radionuclides were observed in sample A1, which suggests significant differences in the original mineral composition of the raw materials or in the concentration processes during production.

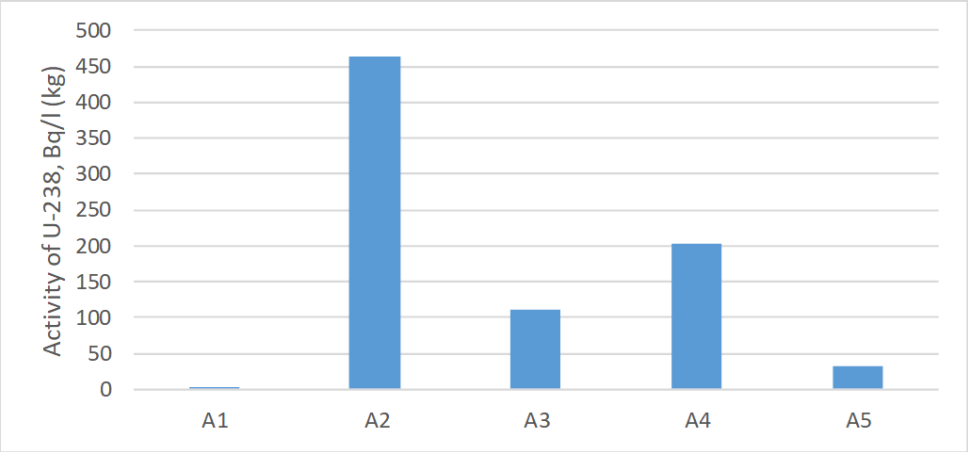

Figure 1 – Content of uranium-238 isotope in the analyzed samples

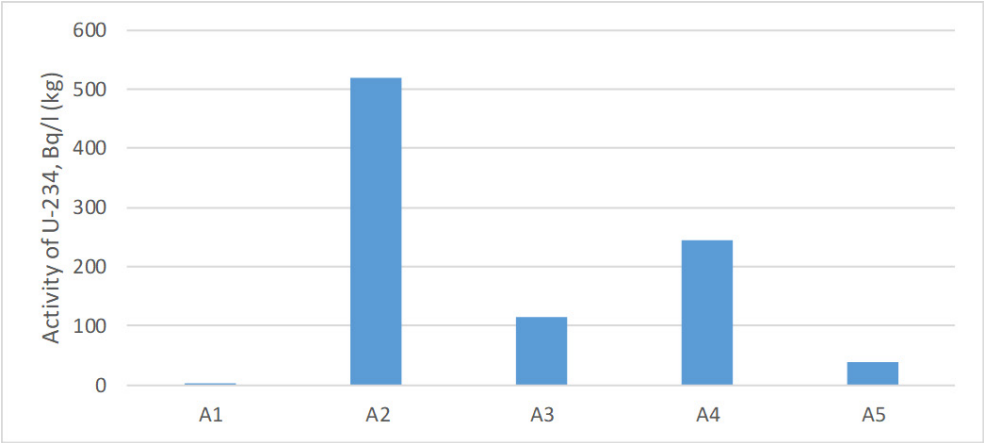

Figure 2 – Content of uranium-234 isotope in the analyzed samples

A similar pattern is also characteristic of radionuclides of the thorium series: the highest activity of Th-232 and its daughter isotopes (Th-230 and Th-228) was recorded in samples A2 and A4, which indicates the presence of thorium-containing components of natural origin in these samples.

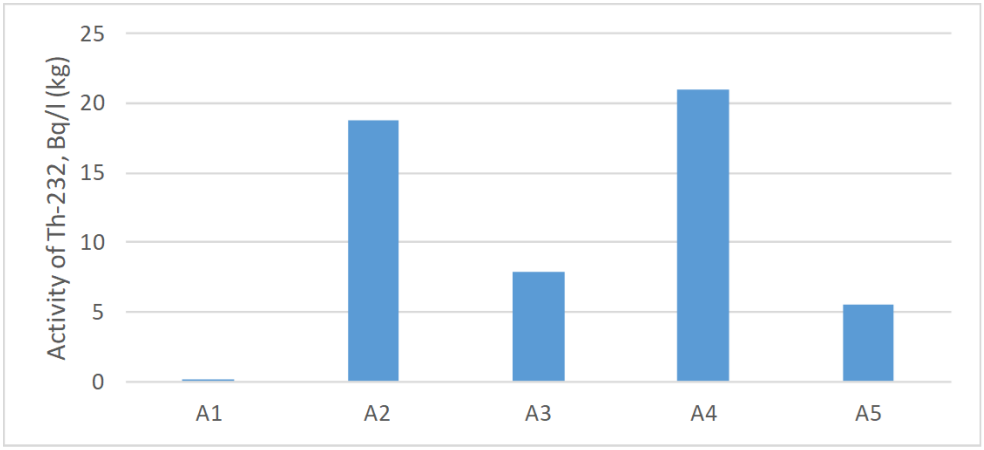

Figure 3 – Content of thorium-232 isotope in the analyzed samples

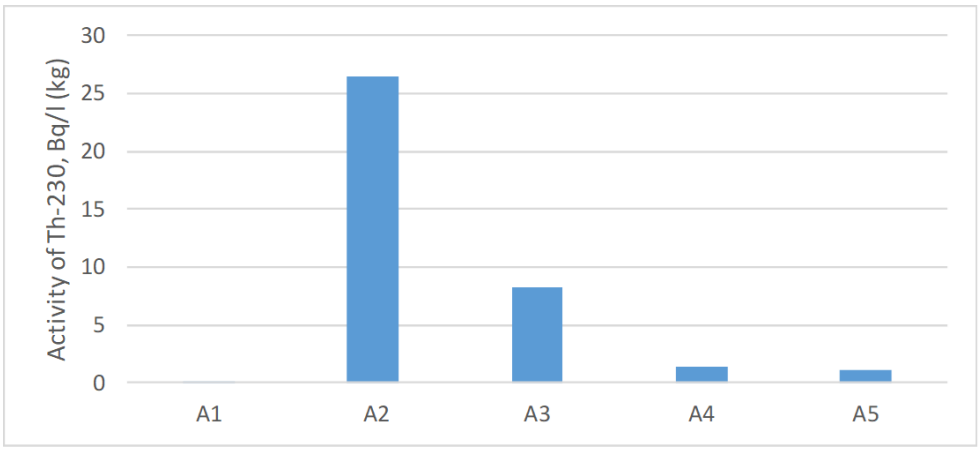

Figure 4 – Content of thorium-230 isotopes in the analyzed samples

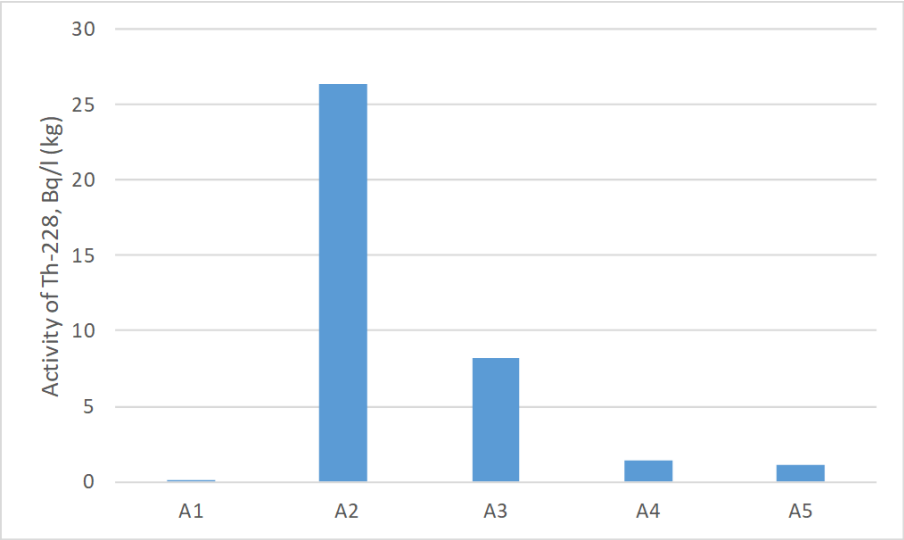

Figure 5 – Content of thorium-228 isotopes in analyzed samples

The radioactivity of Po-210 and Pb-210 also correlates with the concentration of uranium. Their values are highest in the samples A2 (119 and 513 Bq/kg, respectively) and A3 (112 and 483 Bq/kg), which indicates the enrichment of these products with radionuclides from the uranium-radium series. Probably, during the processing of organomineral components, sorption accumulation of these radionuclides occurred, especially in the presence of humic acids, which have a high complexing capacity [21].

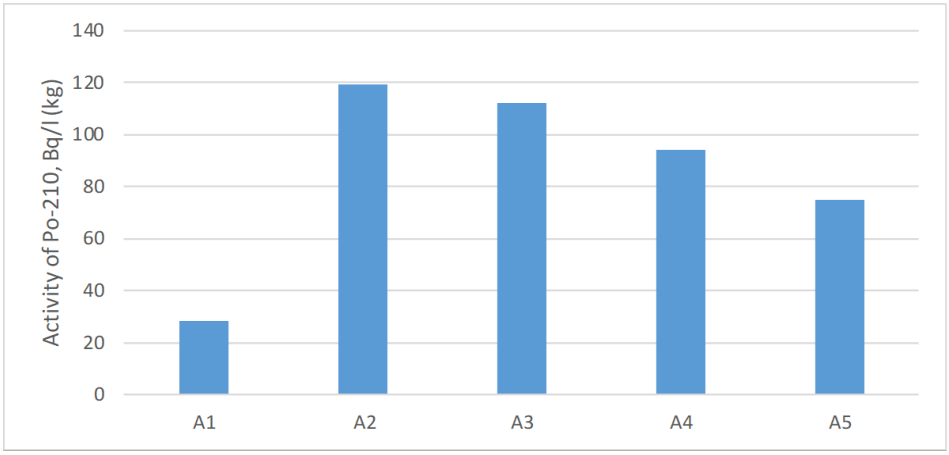

Figure 6 – Content of polonium-210 isotope in samples

Comparison of the radionuclide composition with the expected content of humic substances shows that higher concentrations of U-238, Th-232 and Po-210 are characteristic of fertilizers containing an active organic matrix. This confirms the role of humic acids as factors enhancing the migration and retention of radionuclides in the solid phase.

Thus, it can be concluded that the content of natural radionuclides in humate-containing fertilizers is determined not only by the origin of the raw materials, but also by the degree of complexation with humic substances. These results are consistent with previously published data [22], where the influence of organic matter on the behavior of radionuclides in the soil-plant system was emphasized. Such a dependence is especially significant when assessing environmental risks when using fertilizers based on natural organomineral components.

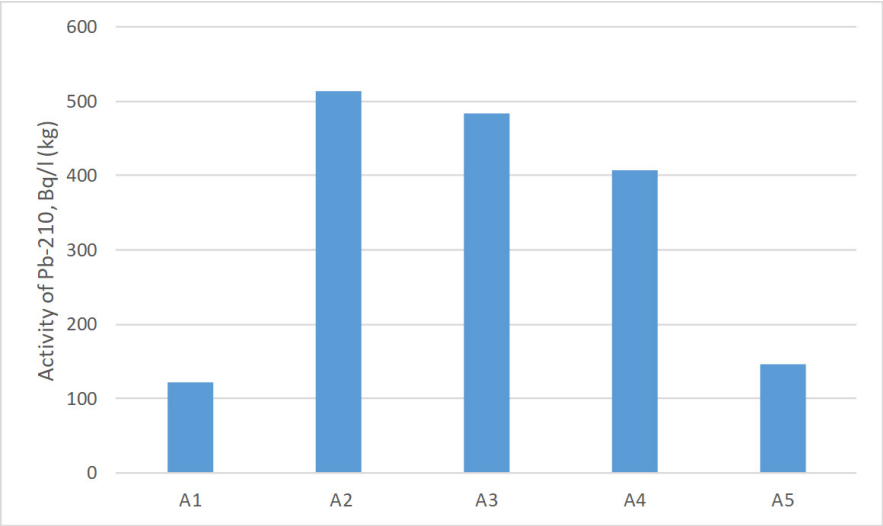

Figure 7 – Content of lead-210 isotope in samples

To assess the reliability of the experimental data obtained on the activity of radionuclides in samples of humate-containing fertilizers, statistical processing of the results was carried out. The main calculation indicators were: arithmetic mean, standard deviation, confidence interval, and variation coefficient. The processing was performed for all studied radionuclides: U-238, U-234, Th-232, Th-230, Th-228, Po-210, and Pb-210.

The arithmetic mean was calculated using the formula:

$$\bar{x} = (1/n) \cdot \sum x_i \quad (1)$$

where $\bar{x}$ is arithmetic mean, x_i is individual values, n is number of measurements.

The standard deviation (unbiased) was defined as:

$$s = \sqrt{[(1 / (n - 1)) \cdot \sum (x_i - \bar{x})^2]} \quad (2)$$

where s is standard deviation, $\bar{x}$ is average value.

Confidence interval (at 95% significance level):

$$CI = \bar{x} \pm t * (s / \sqrt{n}) \quad (3)$$

where CI is confidence interval, t is Student's coefficient, s is standard deviation, n is number of measurements.

The coefficient of variation (in percent) is calculated using the formula:

$$CV = (s / \bar{x}) \cdot 100\% \quad (4)$$

where CV is coefficient of variation, s is standard deviation, $\bar{x}$ is average value.

These formulas were used to analyze the distribution of radiometric measurement results, including to assess the variability of the content of the studied radionuclides in samples of humate-containing preparations. They provided an objective quantitative interpretation of the data and allowed statistical comparison between different preparations. Table 2 presents the statistical parameters for all the studied radionuclides.

Table 2 – Statistical parameters of all studied radionuclides

Radionuclide	Average value	Standard deviation	Confidence interval (95%)	Coefficient of variation (%)
U-238	4224.20	8940.41	±7836.61	211.65
U-234	5106.85	10851.44	±9511.71	212.49
Th-232	10.67	8.91	±7.81	83.46
Th-230	7.44	11.07	±9.71	148.79
Th-228	7.44	11.07	±9.71	148.79
Po-210	85.60	36.45	±31.95	42.58
Pb-210	334.40	186.74	±163.68	55.84

The presented data show that the greatest variability was demonstrated by U-238 and U-234 radionuclides, which may be due to the characteristics of the initial raw materials and the uneven distribution of uranium in the humic matrix. The lowest values of the variation coefficient are observed for Po-210 and Pb-210, which indicates a more uniform distribution of these radionuclides across the samples, probably due to stable sorption bonds with organic matter. Such differences in statistical parameters must be taken into account when assessing the radiation safety of humate-containing fertilizers, especially in the context of long-term impact on the soil-plant system and potential migration of radionuclides in agrobiocenoses.

Such results are consistent with reference data indicating a significant influence of the organic component of fertilizers on the distribution of natural radionuclides. Further research is expected to examine the contribution of specific fractions of humic substances to the retention of uranium and thorium series, using chromatographic fractionation methods and surface radiospectral analysis.

Conclusion

The minimum activity of uranium isotopes was determined in the liquid sample (A1), which indicates a low level of radiation contamination in this sample. However, in the solid humate-containing samples, a higher activity of both uranium isotopes is observed compared to the liquid sample. Specifically, the activity of uranium-238 varies from 463±22 Bq/kg in sample A2 to 33±3 Bq/kg in sample A5, and for the uranium-234 isotope, these values are 519±22 Bq/kg in A2 and 38±3 Bq/kg in A5. Thus, it can be concluded that the humate-containing solid samples have a higher radiation activity than the liquid ones. Also, the minimum values of the activity of thorium isotopes were recorded in the liquid sample (A1). However, the highest content of the thorium-232 isotope was recorded in sample A4, which indicates a higher concentration of this isotope in this sample. As for the thorium-230 and thorium-228 isotopes, the maximum activity was detected in sample A2, which also confirms a certain variability in the content of radionuclides in different types of samples.

For the isotopes polonium-210 and lead-210, the minimum values were established in the liquid sample A1, where the activity was 75±6 Bq/l for polonium-210 and 147±11 Bq/l for lead-210. At the same time, in solid humate-containing samples, the maximum activity of these isotopes is observed in sample A2, where it is 119±9 Bq/kg for polonium-210 and 513±36 Bq/kg for lead-210. These results confirm the high concentration of these radionuclides in solid samples, especially in humate-containing ones.

The specific activity of natural radionuclides in the studied humate-containing samples complies with the requirements of paragraph 5.3.6 of Sanitary rules and regulations 2.6.1.2523-09 “Radiation Safety Standards (NRB-99/2009)”, which means compliance with radiation norms and standards. Thus, the use of these fertilizers for their intended purpose is permitted without restrictions on the radiation factor, which makes them safe for use in agriculture.

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Conflict of interest

The authors declare that they have no conflicts of interest.

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Information about authors:

Mukhambetkali Burkitbayev – Doctor of Chemical Sciences, Professor of Al-Farabi KazNU (Almaty, Kazakhstan, e-mail: mukhambetkali.burkitbayev@kaznu.edu.kz).

Umirzak Zhumasilovich Dzhusipbekov – Doctor of Technical Sciences, Professor, JSC «A.B. Bekturov Institute of Chemical Sciences» (Almaty, Kazakhstan, e-mail: omirzak1952@gmail.ru).

Gulzipa Oryntayevna Nurgalieva – Doctor of Chemical Sciences, JSC «A.B. Bekturov Institute of Chemical Sciences» (Almaty, Kazakhstan, e-mail: N_gulzipa@mail.ru).

Zamira Kenesbekovna Bayakhmetova – Candidate of Chemical Sciences, JSC «A.B. Bekturov Institute of Chemical Sciences» (Almaty, Kazakhstan, e-mail: zamirabkz@mail.ru).

Dulat Duisenbai – Junior Researcher, JSC «A.B. Bekturov Institute of Chemical Sciences» (Almaty, Kazakhstan, e-mail: dulat_211@mail.ru).

Sholpan Nurlanovna Nazarkulova – PhD, Senior Lecturer of Al-Farabi KazNU (Almaty, Kazakhstan, e-mail: Sholpan.nazarkulova@kaznu.edu.kz).

Ilona Valeriyevna Matveyeva – PhD, Associate Prof. of Kazakh-British Technical University (Almaty, Kazakhstan, e-mail: ilona.matveyeva@kaznu.edu.kz).

Yelena Yuriyevna Yarovaya – PhD, Senior Lecturer of Al-Farabi KazNU (Almaty, Kazakhstan, e-mail: yarovayayu@mail.ru).