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Activity concentration of plutonium isotopes in bottom sediments and water in Crimean salt lakes

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Abstract
The $^{238,239+240}$Pu activity concentrations in sediments and in water in the Crimean salt lakes were studied. Activity ratio of ^{238}Pu/$^{239+240}$Pu in depth profiles of sediments was used to estimate the contribution of two main sources of man-made plutonium to the Pu sediment inventory. The most part of the plutonium was of global origin. Concentration factor of the plutonium isotopes in sediments, radiocapacity factor of the lake, the type of biogeochemical behavior of plutonium in these reservoirs and the sediment inventory of $^{238,239+240}$Pu in the lakes were evaluated.

Keywords $^{238,239+240}$Pu · Sediment · Depth distribution of plutonium · Water · Crimean salt lakes · The Black Sea

Introduction

The input of man-made radionuclides into the environment started after the humankind began using nuclear technologies for military and peaceful purposes. Accidents at nuclear facilities became one of the sources of radionuclide release into the environment [1–7]. The aquatic ecosystems play an important role in migration of radionuclides. Salt lakes occupy a special place among aquatic ecosystems, because many of them are stagnant water bodies and therefore become a long-term depot of radionuclides. However the resources of salt lakes are widely used for national economic purposes. The most intensively used lake resources are their bottom sediments being used as therapeutic and cosmetic mud. The Crimean salt lakes are also popular places of mass spontaneous tourism [8, 9]. The lake brine is used as a raw material for the extraction of common salt and other substances. *Dunaliella salina* and others living in the Crimean salt lakes [10, 11] are important raw materials for fishery enterprises. The Crimean salt lakes are promising objects for the development of aquaculture [10, 11].

The ^{238}Pu and $^{239+240}$Pu investigations in the components of the Crimean salt lakes ecosystems were not fulfilled until recently. At the same time the bottom sediments in lakes were well known to be characterized by the highest plutonium concentration factors (C_f) and to be a main depot for plutonium radionuclides in aquatic ecosystems [4–7]. For the Crimean peninsula the main sources of man-made radioactive isotopes were the global fallout from the nuclear weapons testing in open environment and the fallout from the accident at the Chernobyl nuclear power plant (ChNPP) in 1986 [3, 12]. In the early 1990s the $^{239+240}$Pu distribution on the territory of the Crimean peninsula was assessed [3, 13]. The $^{239+240}$Pu distribution was uneven and patchy. Some salt lakes located in regions with the $^{239+240}$Pu higher density of the surface contamination [14].

The aim of this study is to determine the $^{238,239+240}$Pu activity concentration in depth profiles in sediments and in the water of the Crimean salt lakes, as well as the contribution of the two main sources of the man-made plutonium input to the lake sediments and the $^{238,239+240}$Pu sediment inventory as necessary information to allow an assessment of the long-term redistribution of plutonium from water to sediments in these reservoirs.

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Material and methods

Sampling

The study of ^{238}Pu and $^{239+240}$Pu activity concentration in the depth profiles of sediments and in the water in the four lakes belonging to the four geographical groups of the Crimean lakes was carried out in 2017–2018. In the six salt lakes during this period only 0–5 cm layers of sediments were examined (Fig. 1).

The materials for the study were taken during coastal land expeditions. The groups of lakes were chosen considering the fact that these four geographical groups of the salt lakes coincide with the four groups of balneological resources of the Crimean peninsula [9, 14]. The samples of sediments and water in the seas were taken on the 72nd, 84th and 90th scientific cruises of the R/V "Professor Vodyanitsky" in 2013–2017 (Fig. 1). The sampling of surface water (in lakes: 250–760 L and in the Black Sea: 1000–2000 L) was carried out using ''MasterFlex'' peristaltic pumps. The sea bottom sediment samples were taken with the box corer (made by «Shelf», United States) with dimensions of 35 × 35 × 50 cm and the ground acrylic tubes which were equipped with a vacuum valve. Sediment cores (0–30 cm) were cut into layers of 2–5 cm thickness with a screw extruder.

Methods of the $^{238, 239+240}$Pu determination in the natural samples

The $^{238,239+240}$Pu radioisotopes were determined in the samples by well-known techniques [15, 16]. Samples of sediments were dried at 80 °C and incinerated in a muffle furnace at 550 °C. The radioactive tracer ^{242}Pu was added to the ashed samples for determination of the Pu chemical yield. Then samples were leached, followed by purification and separation of plutonium by the use of the two-step ion-exchange chromatography [14, 16]. Plutonium in the water samples was pre-concentrated by co-precipitation with manganese dioxide (MnO_2) and $Fe(OH)_3$. First the pH of the water sample was adjusted to 2 with adding HNO_3 and spiked ^{242}Pu as a chemical yield tracer. After adding $KMnO_4$ (5 g per every 20 L of a sample) the sample was left to stand for 0.5–1 h. Then the pH of the sample was adjusted to 9 with adding 10 M NaOH and saturated solution $MnCl_2$ was added to co-precipitate Pu with MnO_2. In a day or 2 days the sample supernatant was siphoned from the precipitate. The pH of the precipitate was adjusted to 0–1 and dissolved with 30% solution of H_2O_2 and was added saturated solution of $KMnO_4$ (5 g). The sample was left to stand for 0.5–1 h and the second co-precipitation with MnO_2 (with adding 10 M NaOH to the pH 9) was carried out. In a day or 2 days the precipitate was separated from the solution by decantation and filtration through filter paper using a Buchner funnel and a Bunsen flask connected to a vacuum pump. The precipitate was dissolved in 2 M HNO_3 with the addition of H_2O_2 in a heat-resistant glass. The solution was heated and brought to a boil on a hot plate to decompose hydrogen peroxide. Then plutonium was precipitated with $Fe(OH)_3$ by adding 50–100 mg of $FeCl_3$, 1 g of $NaNO_2$ and concentrated ammonia solution to pH 8–9. In a day or 2 days the precipitate was separate from the solution with centrifugation (3000 rpm). The precipitate was dried and then dissolved with concentrated HNO_3 3–5 ml and adding 50 ml 8 M HNO_3 immediately before anion-exchange column chromatography. Further the water and sediments samples were treated using two-stage ion-exchange chromatography with anion-exchange resin Bio-RAD AG1x4 and AG1x8. Activity of alpha–emitters ^{238}Pu and $^{239+240}$Pu, ^{242}Pu was measured after electrodeposition of Pu onto stainless steel discs. The measurements were carried out on the semiconductor alpha-spectrometric complex "EG & G ORTEC OCTETE PC" using the MAESTRO software model A65-B32. Background of detectors in the spectrum picks of the measured alpha-emitting plutonium isotopes was 2×10^{-6}–4×10^{-6} Bq, detection limit = 2×10^{-5} Bq per sample, the samples were measured 250,000–550,000 s. The uncertainties of ^{238}Pu and $^{239+240}$Pu activity concentrations included 1σ counting errors of net counts of these radioisotopes and radiotracer (^{242}Pu) combined with uncertainties of radiotracer addition and weighting procedure.

The belonging of $^{238,239+240}$Pu to the global fallout or to the Chernobyl fallout was determined by the value of the ^{238}Pu/$^{239+240}$Pu activity ratio by the generally accepted method [3, 7, 17, 18]. To characterize the role of sediments

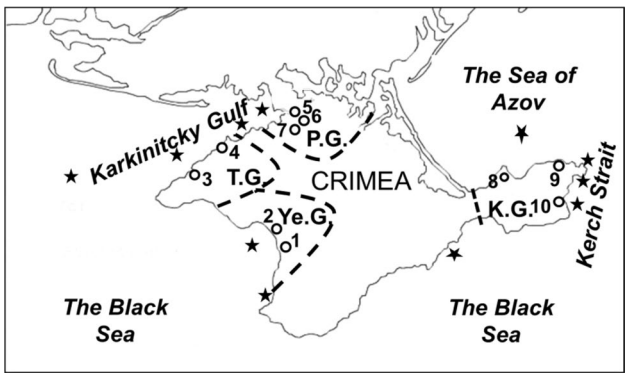

Fig. 1 Sampling location: (numbered circles)—investigated Crimean salt lakes: (1)—Kyzyl–Yar, (2)—Sasyk–Sivash, (3)—Dzharylhach, (4)—Bakalskoe, (5)—Krasnoye, (6)—Kiyatskoe, (7)—Kirleutskoe, (8)—Aktashskoe, (9)—Chokrakskoe and (10)—Tobechikskoe from four geographical groups (dashed line): Ye.G.—Yevpatoriya group, T.G.—Tarkhankut group, P.G.—Perekop group and K.G.—Kerch group and (black stars)—sampling stations in the adjacent coastal areas of the Black Sea and the Sea of Azov

in the redistribution of plutonium radioisotopes in the lakes, concentration factors of plutonium (C_f ($^{239+240}$Pu)) in sediments and factors of radiocapacity of lakes (F_r ($^{239+240}$Pu)) were calculated according to the well-known approaches [12, 19].

Results and discussion

$^{239+240}$Pu in the lakes' and sea water

The $^{239+240}$Pu activity concentration of water in four Crimean salt lakes varied from 0.8 ± 0.3 to 16.5 ± 1.3 mBq m^{-3} (Fig. 2). The difference in the levels of $^{239+240}$Pu activity concentration in the water of lakes may be associated with the spottiness of radioactive fallouts [14], with different water supply sources of the lakes as sources of secondary plutonium contamination of the lakes [9, 14, 20]. But at present, only plutonium radioisotopes scattered in the environment serve as a source of plutonium input into the Crimean salt lakes. Therefore, the size of specific catchment area of the lakes (a ratio of a lake catchment basin area to a lake area) could influence the level of the plutonium concentration in water of the lakes because of different amount of substances introduced into a lake with surface water and with river runoff. The lowest value of the specific catchment area is typical for Lake Kirleutskoe—5, for lakes Dzharylgach and Kyzyl-Yar it was much higher (104 and 41, respectively) and for Lake Aktashskoe—17 [9].

The values of the $^{239+240}$Pu activity concentrations in water of the lakes were on the average 13 times higher than in the Black Sea water [12]. The catchment area of the Black Sea is about 5 [3] and it is many times less than the specific catchment area of the

lakes adjacent to the sea areas. This may lead to the formation of lower concentrations of plutonium in the water in the Black Sea in comparison with the lakes' water especially in lakes Dzharylgach and Kyzyl-Yar at present period (Fig. 2). In addition, the dilution capacity of the Black Sea as a deep body of water is much greater than that of the stagnant and shallow salt lakes. But as it was shown earlier [14], the average $^{239+240}$Pu activity concentrations in sediments of the lakes in the upper layer was 2 times lower than those in the Black Sea sediments. These facts may indicate the specific features of the redistribution of plutonium in salt lakes. The retention of the increased plutonium radioisotopes activity concentrations in the water of the salt lakes could also be facilitated by the stirring up of bottom sediments as a result of wind mixing in shallow salt lakes and subsequent partial remobilization of plutonium radioisotopes from sediments into water. It is likely that the redistribution of plutonium from water to sediments in lakes was influenced by internal conditions, one of which maybe was a high salinity of lake waters [14]. But it is known that high salinity had a multidirectional effect on the redistribution of various elements from water to other components of aquatic ecosystems [6, 21]. So, these questions requires further study to clarify the reasons for the increased $^{239+240}$Pu activity concentration radioisotopes in the salt lake water in comparison with the $^{239+240}$Pu activity concentration in the Black Sea water at present period.

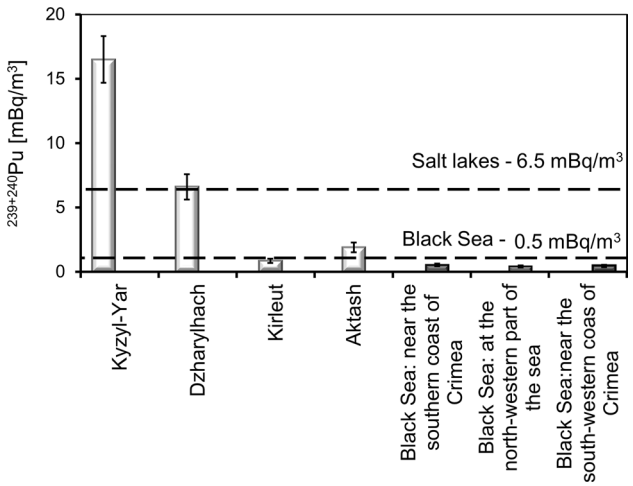

Fig. 2 The $^{239+240}$Pu in water of the Crimean salt lakes (white histogram) and in the adjacent coastal areas of the Black Sea (gray histogram), where the dashed lines are the average $^{239+240}$Pu activity concentrations in the water of lakes and the Black Sea

$^{238,239+240}$Pu in upper layer of the lakes' sediments

The average $^{239+240}$Pu activity concentrations that were determined in upper layers of sediments in 10 salt lakes in 2016–2018 are shown in Fig. 3. Both sandy and silt sediments were found in the four lakes. It was revealed, that $^{239+240}$Pu activity concentrations were significantly lower in sandy sediments than in the silt ones of the same lakes (Fig. 3).

To characterize accumulative ability of sediments the C_f ($^{239+240}$Pu) in sediments were calculated according to the data presented in Figs. 2 and 3. The C_f ($^{239+240}$Pu) by the silt sediment ranged from 2.4 to 8.8×10^4, in the sandy sediment varied from 3.7 to 5.6×10^3. In the adjacent areas of the Black Sea C_f ($^{239+240}$Pu) by the silt and sandy bottom sediment varied within 1.1×10^6–2.2×10^6 and 3.7×10^4–1.4×10^5, respectively [12]. The role of sediments in the redistribution of plutonium from water in the lakes was quantitatively assessed using the F_r ($^{239+240}$Pu) [12, 19]. It shows what percentage of plutonium radioisotopes from their total amount in the reservoir accumulated in bottom sediments [12]. The F_r ($^{239+240}$Pu) for the salt lakes reached 99% for silt and sandy sediment which is due to the high

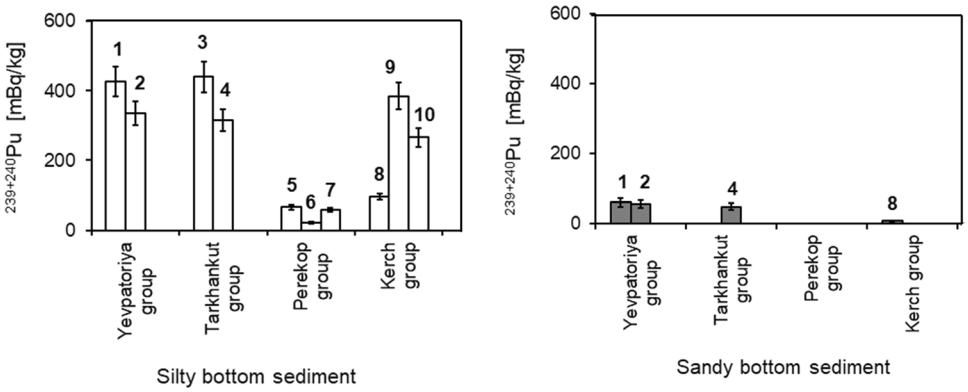

Fig. 3 $^{239+240}$Pu activity concentration in silt and sandy sediments (0–5 cm layer) of ten Crimean salt lakes from four groups: (1)—Kyzyl–Yar, (2)—Sasyk–Sivash, (3)—Dzharylhach, (4)—Bakalskoe, (5)—Krasnoye, (6)—Kiyatskoe (7)—Kirleutskoe, (8)—Aktashskoe, (9)—Chokrakskoe, (10)—Tobechikskoe

C_f ($^{239+240}$Pu) of bottom sediments, as well as the shallow depth of the lakes (0.9–2.0 m). Since 99% of the plutonium was redistributed into sediments, the type of biogeochemical behavior of plutonium was characterized as a pedotropic type [12]. Therefore, in the salt lakes, bottom sediments serve as the main depot of plutonium radioisotopes. The activity concentrations of ^{238}Pu in upper layer (0–5 cm) of the lakes' sediments were significantly lower than of $^{239+240}$Pu and they changed from 39 ± 13 to 1 ± 0.5 mBq/kg. In Lake Kiyatskoe it was below detection limit.

Depth distribution of ^{238}Pu and $^{239+240}$Pu in bottom sediment

The depth distribution of the plutonium radionuclides obtained in 30 cm sediment cores differed in the four lakes of each group of the Crimean salt lakes (Fig. 4). The activity ratios of ^{238}Pu/$^{239+240}$Pu in the global fallout and the Chernobyl radioactive fallout are known to be differed by more than an order of magnitude and in 1986 at the Crimea latitudes they were equal to 0.03 and 0.47 respectively [22, 23]. In the upper layer of sediments (0–5 cm), a significant excess of the ^{238}Pu/$^{239+240}$Pu values above 0.03 was observed only in the two lakes (Fig. 4). Therefore, only in these layers the presence of plutonium with Chernobyl origin was found. Its percentage was equal in the sediments of Lake Sasyk-Sivash 23%, and in Lake Dzharylgach—4% of the total plutonium content in the layer.

In the deeper layers in sediments the ^{238}Pu/$^{239+240}$Pu activity ratio was about 0.03 and indicated the plutonium with global fallout origin (Fig. 4). In the Lake Chokrakskoe the $^{239+240}$Pu or ^{238}Pu profiles were destroed and had no any maxima of $^{239+240}$Pu in 0–30 cm layer of sediments of this lake (Fig. 4). The sediments of a shallow Lake Chokrakskoe were characterized both mixing by wind and sporadic impact during warm season by many spontaneous tourists covering themselves with the silt sediments, they also collected mud in containers of 3–5 L and take it home and in this way they jumbled sediments. These processes could lead to mixing of the sediments in the 0–30 cm layer in the lake and the formation of an almost uniform vertical distribution of the plutonium radioisotopes in sediments. (Figure 4). The ^{238}Pu/$^{239+240}$Pu activity ratio over the whole depth of the investigated profile was quite low because the Chernobyl origin part of Pu may has been mixed and redistributed over the 0–30 cm layer of the sediments (Fig. 4). To estimate the percentage of plutonium with Chernobyl origin in the plutonium inventory in this lake, it is necessary to take samples from a floating craft in the remote central region of the lake, where perhaps there is not anthropogenic influence. In other three lakes the $^{239+240}$Pu maxima were observed at different depths in the sediments (7–22 cm). This is probably due to the different sedimentation rates in the lakes [18]. The highest $^{239+240}$Pu activity concentration (2.1 Bq kg^{-1}) was recorded in the 10–15 cm sediment layer in Lake Sasyk–Sivash from the Yevpatoriya group. In this and other layers the ^{238}Pu/$^{239+240}$Pu activity ratio indicated the plutonium radioisotopes with global fallout origin (Fig. 4). An exception was the 7–12 cm layer in Lake Kirleutskoe, where the ratio was 0.1, which indicated the presence of plutonium with Chernobyl origin and its percentage was about 16%. This position of the maximum of plutonium with Chernobyl origin can be explained by the higher sedimentation rate in this lake, compared with other studied lakes. The decrease in the intake of plutonium with Chernobyl origin in the recent years is obviously associated with the cessation of the water supply from the Dnieper River via the North-Crimean Canal to Lake Kirleutskoe [14]. The more exact explanation on this point can be given by the further study of the $^{238,239+240}$Pu vertical distribution in the cores of the sediments in the salt lakes with dividing the sediment cores into thinner layers (for example 0.5–1 cm) and taking into account the sedimentation rate in the each particular lake.

Inventories of the $^{238+239+240}$Pu in the sediment of the four lakes were estimated by the use of the plutonium radioisotopes activity concentration in the profiles (0–30 cm layer) of the lakes' bottom sediments and taking into account the cumulative weight of these bottom sediments layers

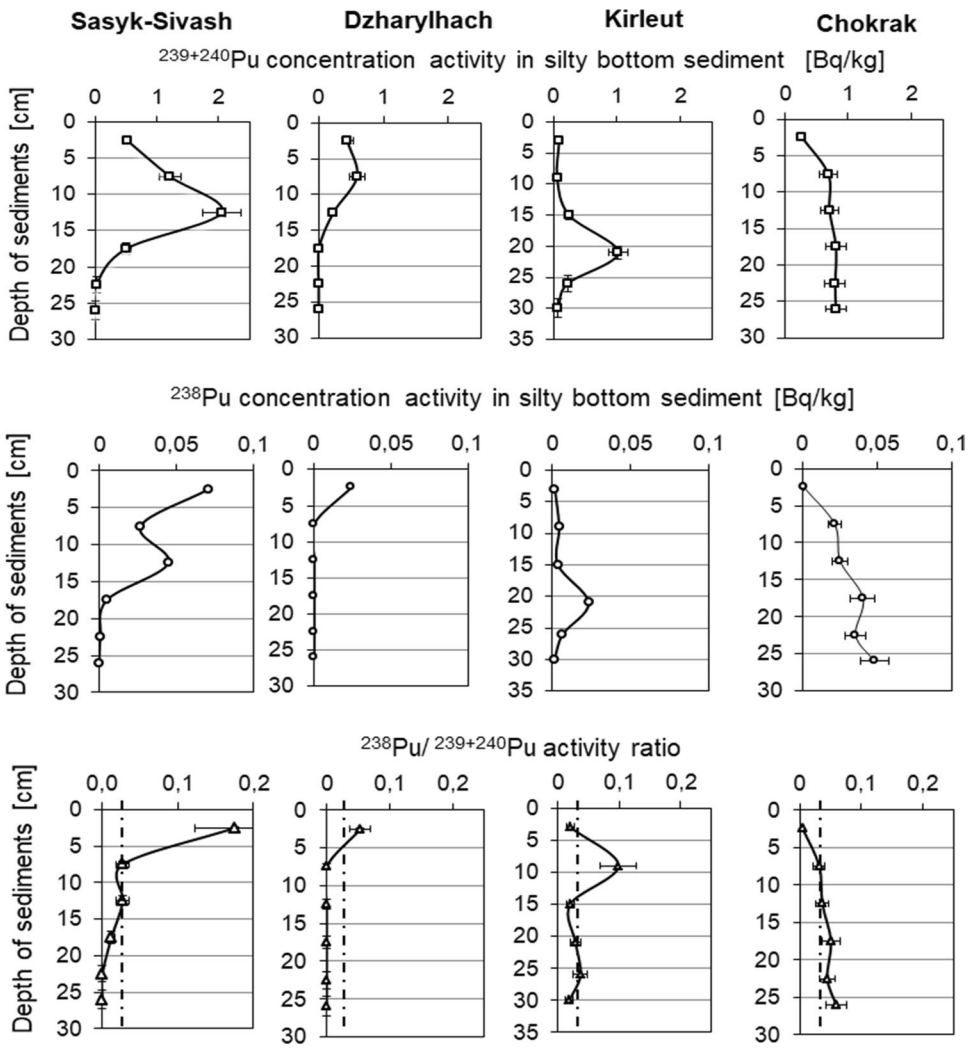

Fig. 4 Depth distribution of $^{239+240}$Pu, ^{238}Pu and their activity ratio ^{238}Pu/$^{239+240}$Pu in bottom sediment of four lakes from each studied geographical group of Crimean salt lakes, where the dash-dotted line is the average ^{238}Pu/$^{239+240}$Pu activity ratio in the global radioactive fallout for 40–50° N latitude band

(Table 1). The sediment inventory of $^{238+239+240}$Pu in the lakes differed from each other and it varied from 86.5 to 196.2 Bq/m^2 (Table 1). The maximum of the $^{238+239+240}$Pu sediment inventory was found in Lake Sasyk-Sivash, which is located in the zone of a maximum radioactive fallout [14]. In general, the plutonium sediment inventories in the Crimean salt lakes were lower by more than one order of magnitude than those in the adjacent coastal Black Sea areas, where the inventory in the upper layer of the sediment varied from 1800 to 6900 mBq/m^2. The obtained results make it possible to characterize the salt lakes in terms of the low content of the plutonium radioisotopes as favorable reservoirs for tourism, balneological and raw material use, as well as for the development of aquaculture.

Table 1 The sediment inventories of $^{238+239+240}$Pu in 0–30 cm layer in the four salt lakes (calculated on 2016)

Location (Lake)	Inventory of $^{238+239+240}$Pu [Bq/m^2]	Area of the lake [km^2]	Total inventory of $^{238+239+240}$Pu in the lake [GBq]
Sasyk–Sivash	196 ± 27	75.3	14.6 ± 2.0
Dzharylgach	87 ± 10	8.3	0.7 ± 0.1
Kirleutskoe	125 ± 40	12.5	2.6 ± 0.8
Chokrakskoe	154 ± 17	8.5	1.3 ± 0.1

Conclusions

This study was one of the few attempts to investigate the features of the processes of long-term redistribution of the technogenic $^{238,239+240}$Pu in the Crimean salt lakes. Summarizing the obtained results, it can be noted that in the salt lakes plutonium exhibited a pedotropic type of biogeochemical behavior, but in comparison with the adjacent areas of the Black Sea, some differences in the plutonium radioisotopes redistribution have been revealed: (a) the $^{239+240}$Pu activity concentrations in water in the lakes were on the average 13 times higher than those in the seawater; (b) the level of the plutonium radioisotopes in the upper layer of sediments of the salt lakes was on the average 2 times lower than in similar sediments of the Black Sea; (c) the concentration factors of plutonium radioisotope in the sediments of the salt lakes were two orders of magnitude lower than those in the Black Sea. It was suggested that different density of radioactive fallouts, the water supply sources of the lakes and the size of the specific catchment area, the depth of the water bodies could influence the formation of different levels of the $^{239+240}$Pu activity concentrations in the water of the lakes and in the sea. But the difference was not only in the levels of the plutonium radioisotopes activity concentration in water and sediments of the lakes and the sea. The plutonium concentration factors in sediments also differed in these reservoirs. This difference cannot be explained by geographic conditions. It is likely that this difference may be due to the peculiarities of the biogeochemical processes in the lakes. For unambiguous answers to this issue a further study of the plutonium behavior in the salt lakes and the identification of the leading factors affecting its redistribution in the salt lakes is required. It seems important to consider the complex of biogeochemical processes in the salt lakes as a result of the interaction of natural and anthropogenic processes and conditions in the catchment area of the lakes and in their ecosystems. In the Crimean salt lakes, the $^{238,239+240}$Pu concentrations were not high. But the levels of these concentrations were sufficient to use the $^{238,239+240}$Pu as radiotracers. The method of radiotracers provides an opportunity to study the natural processes and their parameters (for example, the sedimentation rate, the mater accumulation rate) in the lakes and on their basis, to determine the values of parameters characterizing the processes of plutonium redistribution in the ecosystem of the salt lakes. These parameters include, for example, the $^{238,239+240}$Pu sedimentation fluxes from water to sediments in the present period and retrospectively. Retrospective assessments of them can be made based on the $^{238,239+240}$Pu activity concentration in the depth profile in sediments. This is the task of subsequent research.

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