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N.B. Uralov[*], **Kh.Kh. Turayev**, **B.A. Normurodov**,
I.A. Umbarov, **F.B. Kurbonov** and **Y.Sh. Bozorov**

Termez State University, Termez, Uzbekistan
* e-mail: uralovnuriddin0091@gmail.com

X-RAY fluorescence analysis of mineral composition in Khaudak and Uchkizil groundwaters, Uzbekistan

This study focuses on the analysis of underground waters from Khaudak and Uchkizil, located in the southern region of Uzbekistan. These waters are characterized by the presence of various mineral salts, including iodine-containing compounds. The research examines the similarities between these waters, as well as their compositional changes over time under the influence of external environmental factors. Water samples collected at different intervals were analyzed to monitor variations in composition. The elemental content of the samples was determined using X-ray fluorescence (XRF) analysis, which revealed that iron compounds present in the water tend to precipitate over time. The initial iron content of the water was approximately 0.130%, with subsequent sedimentation resulting in iron-rich deposits containing up to 65% iron. In addition, a freshly collected water sample was treated with specific oxidizing agents for iodine and stored for one month. This process led to the formation of a reddish-brown precipitate primarily composed of iron and chlorine, with minor components including iodine and similar elements. The precipitate was found to contain 1.317% iodine, corresponding to 7.66% (21.32 mg/L) of the total iodine content in the Haudak water. Furthermore, exposure of the water to ultraviolet light under open-air conditions resulted in the oxidation and volatilization of iodine, indicating its sensitivity to photochemical degradation.

Key words: Khaudak, Uchkizil, iron (III)-chloride, X-ray fluorescence.
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1 Introduction

In recent years, iodine-related health issues have become increasingly prominent worldwide. A growing number of iodine deficiency disorders are being reported, both in terms of incidence and variety. One of the urgent tasks in addressing these concerns is the study and analysis of iodine-bearing surface and groundwater sources. This includes detailed examination of physical and chemical characteristics such as temperature, color, mineral composition, and density. Among these sources, Khaudak and Uchkizil groundwaters in southern Uzbekistan are of particular interest due to their high mineralization and significant iodine content.

Studies conducted in South China have shown that iodine concentrations in groundwater often exceed the World Health Organization's recommended range of 5–300 μg/l. The enrichment of iodide is especially pronounced under mildly acidic (pH ≈ 6.6) and reducing conditions (Eh ≈ 198.4 mV) [1]. Based on the results of iodine analysis in seven geo-ecological zones across China, 4 levels were recognized in groundwater with concentrations of less than 10 μg/l and greater than 300 μg/l [2]. It has been determined that iodine in water exists primarily as molecular iodine (85.6%), followed by iodide ions (3.2%), iodate (9.1%), and iodine chloride (2.1%) [3]. Microorganisms are known to play a critical role in the iodine biogeochemical cycle by mediating oxidation, reduction, volatilization, and deiodination processes. Comparative metagenomic analyses of deep groundwater in the North China Plain revealed the presence of idrABP1P2 gene clusters involved in the reduction of iodine species. Additionally, iron- and sulfur-reducing bacteria may contribute to iodide formation via reductive dissolution of iron minerals and abiotic iodate reduction [4]. In addition, the water of the Datong Basin was analyzed. According to it, the range of iodine in the groundwater of the Datong Basin was 4-2175 μg/l, and it was determined that the enrichment of iodine in the groundwater was due to the action of microorganisms. [5]. In Danish groundwater, iodine concentrations were observed in 23 groundwater wells between 2011 and 2021. The study found that iodine concentrations fluctuate over time. [6]. Concentrations of iodide (I-), iodate (IO3 -), and total iodine (TI) from natural iodine species were studied at four Danish research sites. TI concentrations in groundwater ranged from 5 to 14,500 μg/l. High TI concentrations in the four iodine specification areas were characterized by three main causes: atmospheric deposition and leaching from iodine-enriched soil due to proximity to the sea, desorption of aquifer sediments from marine organic matter and iodine-enriched soil, and the effect of iodine from minerals and residual brines leading to upward diffusion of iodine. [7]. In addition, iodide, iodine, and common iodine in the main drinking water samples were analyzed in this country. 6 regions with an average of 15 μg/l and close to 12 μg/l were identified [8]. Within the Commonwealth of Independent States (CIS), iodine-rich groundwaters have been investigated in Russia, Azerbaijan, Turkmenistan, Uzbekistan, and Ukraine. Among total proven reserves, Turkmenistan holds approximately 40% of iodine-rich groundwater, followed by Russia (34%), Azerbaijan (22%), Ukraine (3%), and Uzbekistan (1%) [9]. In most cases, high-iodine waters were drawn from wells 75–120 m deep. The combination of high pH and a reducing environment facilitates iodine enrichment, accounting for 63.2–99.3% of the total iodine content. Sediment samples from such wells contain 0.18–1.46 mg/kg of iodine, moderately correlated with total organic carbon (TOC) [10].

External factors also influence changes in the amount of iodine in natural waters. For example, when studying the effects of ozone on iodine on the sea surface for a certain period of time, a decrease in iodine emission was detected [11]. Iodine concentrations in the solution were measured at different temperatures and at different time intervals. According to the results of the study, which contained iodine, when frozen samples were stored for a long time, it was found that the amount of iodine changed little. [12]. In some studies, however, iodide detection is achieved by oxidizing iodide to hypoiodic acid using monochloramine [13]. And the detection of iodine and total iodine in seawater by the method of differential pulse polarography brings some convenience. For example, it is convenient to determine the amount of iodine after oxidation of iodide to iodine by exposure to ultraviolet light by very low chemical processing [14]. More than a dozen chemical reagents have been applied to identify iodine and Bromine species in marine sediments. As a result, iodine was found to exist mainly in the electro-positive state in the form of N-iodoamides, while bromine was found to exist in a variety of chemical forms [15]. The effect of ultraviolet rays on iodine present in marine waters was studied based on the spectrophotometric method, and iodide was then analyzed by difference [16].

The conversion of iodide into elemental iodine is favored in acidic environments and can be enhanced by pre-acidification using hydrochloric or sulfuric acids, followed by oxidative treatment to release free iodine into the air [17, 18]. In practical applications, electrodialysis is employed to concentrate iodized sodium chloride from brines, a process used in Japan to produce various types of iodized table salt [19]. The synthesis of polyaniline nitrocellulose (PANS) was carried out using aniline (AN) and nitrocellulose (NS). This substance was used as the main product for ion exchange membranes. As a result, research is ongoing on the use of PANS as a new ion exchange membrane for the separation of iodine from saline waters. [20], The crystal structure and Hirshfeld surface analysis results of the newly synthesized complex compound $[Cd(OPD)_3SO_4]\ H_2O$ derived from o-phenylenediamine (OPD) are being studied, and the sorption properties are being analyzed. [21].

Currently, the composition of sorbents obtained as a result of numerous syntheses is carried out using analyzers operating on the basis of luminescence properties. For example, the interaction of charged particles – protons, nitrogen, oxygen and carbon ions – with LiF single crystals was studied using the luminescence method. [22]. The properties of the thermally stimulated luminescence (TSL) peak in the phase transition temperature region were also studied through the spectral-luminescent properties of ammonium halide crystals. [23].

This study focuses on the Khaudak and Uchkizil underground waters located in the Surkhandarya region of Uzbekistan, which are known to contain elevated concentrations of iodine alongside other mineral salts. These waters offer a unique opportunity to explore the physicochemical mechanisms underlying iodine retention and mobilization under natural and induced conditions. To this end, water samples were collected and analyzed for baseline parameters, including temperature, color, mineral composition, and density. The effects of environmental exposure and oxidative treatments on the chemical composition of the waters were examined using X-ray fluorescence (XRF) spectroscopy.

2 Materials and methods

Polyethylene containers with a capacity of 5 liters and 1 liter for storing brine samples and forming sediment; 100 ml beakers and flasks for separating the formed sediment; Büchner funnel, filter paper and vacuum pump for filtering the sediment; Glass beaker, drying cabinet for drying the separated sediment; Thermometer with a range of 0-100 °C for monitoring the temperature of the initial water sample and the conditions during sediment formation; Areometer for determining the density of brine; 30% solution of iron (III) chloride for oxidizing the iodide compound contained in brine; X-ray fluorescence analyzer for analyzing the composition of water and sediment; When iron (III) chloride is used as an oxidant to separate iodine from brine, a selective oxidation process for iodine occurs, since most iodine-containing groundwater sources also contain bromine along with iodine, and iron (III) chloride cannot oxidize bromine, and iodine can be separated separately. After determining the amount of ions in brine, it is possible to draw clear conclusions about the method by which iodine can be separated.

The X-ray fluorescence analysis. EDX-8100P Energy Dispersive X-Ray Fluorescence Spectrometer (SHIMADZU). This spectrometer with ranges of detectable elements from C to U. The X-ray tube consists of a Rh-anode and operates at a voltage of 4-50 kV. The given range of concentrations is from ppm to 100%.

3 Experimental part

Khaudak water is extracted from the ground from a depth of about 3,000 meters, and Uchkizilwater from a depth of about 400 meters. They are mainly present at temperatures of 70-80 °C and 25-30 °C, respectively, at the time they are mined underground. A solution of a brown-reddish suspension is formed when the water of the tank drops from its initial temperature to room temperature (15-25 °C) and gradually forms a precipitate for 5-6 days. The Uchkizil water will not be significantly hot when mined, and the color of the solution will also be clear and close to transparency. Therefore, brown-reddish sediment from it falls less often. Below, a brown-reddish sediment obtained over 30 days from Uchkizil water was separated and dried, and the spectra obtained from the X-ray fluorescent analyzer (Figure 1) and images in a scanning electron microscope (Figure 2) obtained at a particle size of 10 µm were given (Table 1).

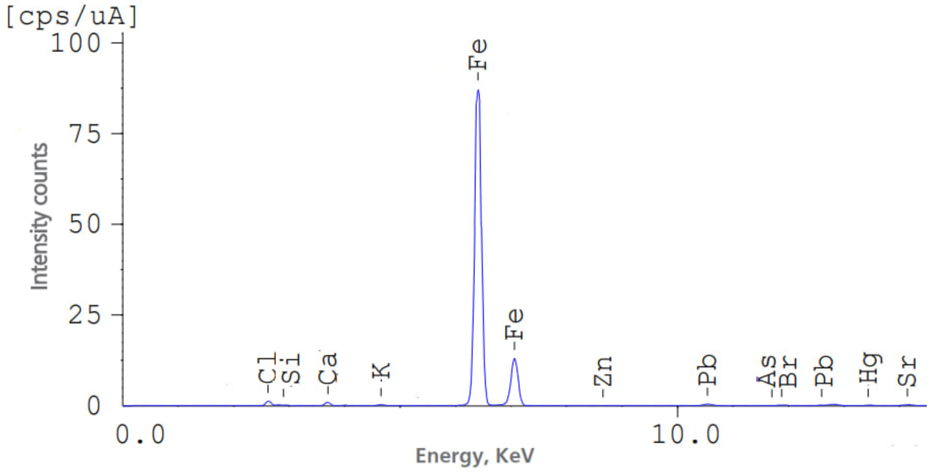

Figure 1 – X-ray fluorescence analyzer spectrum of sediment taken from Uchkizil water.

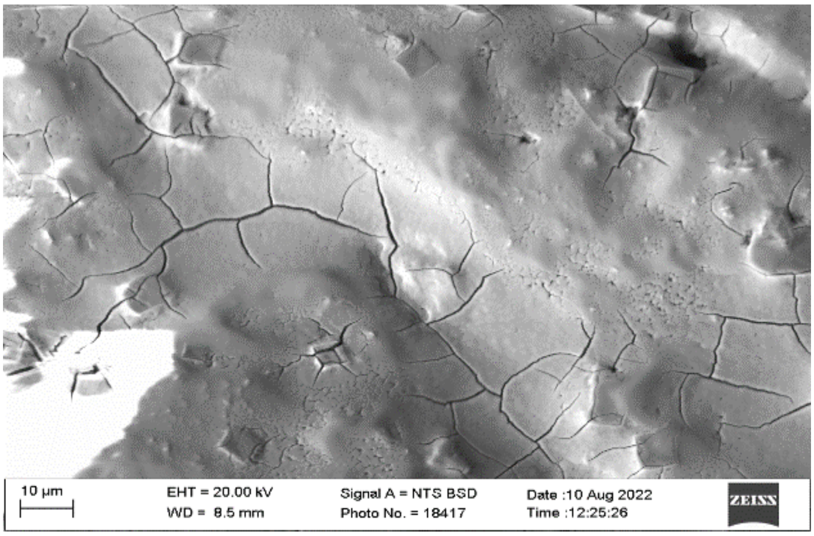

Figure 2 – SEM image of the sediment taken from Uchkizil water.

Table 1 – Quantitative result of the X-ray fluorescence analyzer of the sediment obtained from Uchkizil water.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Fe	77.398 %	[0.193]	Quan-FP	FeKa	320.6805
Cl	14.894 %	[0.369]	Quan-FP	ClKa	0.6529
Ca	3.336 %	[0.097]	Quan-FP	CaKa	1.8473
Si	3.227 %	[0.369]	Quan-FP	SiKa	0.1098
Pb	0.332 %	[0.012]	Quan-FP	PbLb1	0.1781
As	0.197 %	[0.071]	Quan-FP	AsKb	0.3581
K	0.197 %	[0.034]	Quan-FP	K Ka	0.0250
Sr	0.183 %	[0.010]	Quan-FP	SrKa	2.5602
Br	0.121 %	[0.005]	Quan-FP	BrKa	0.1427
Zn	0.096 %	[0.009]	Quan-FP	ZnKa	0.0305
Hg	0.020 %	[0.006]	Quan-FP	HgLa	0.0061

It can be seen from this table that there is no iodine in the precipitate, and iron, chlorine, and calcium can be mentioned as the main elements. It was observed that certain water-soluble compounds of iron precipitate when brought from 70-80 °C to room temperature (15-25 °C) or hydrolyze and form water-insoluble compounds and sink to the bottom of the solution. The solution left after the separation of the brown-reddish sediment that fell under the water of the Khaudak is clear, it was dried at a temperature of 20-30 °C under the influence of sunlight and examined in an X-ray fluorescent analyzer (Figure 3, Table 2).

Below, Uchkizil water was left in the open air, under the influence of sunlight, for 1 week after it was taken from the ground. Then it was analyzed in an X-ray fluorescence analyzer (Fig. 4, Table 3).

This table lists chlorine as the main constituent and calcium as the main mass.

After extracting the water from the underground, it was left in the open air for 2 weeks under the influence of sunlight. Sample 2. Then it was analyzed in an X-ray fluorescence analyzer (Fig. 5, Table 4).

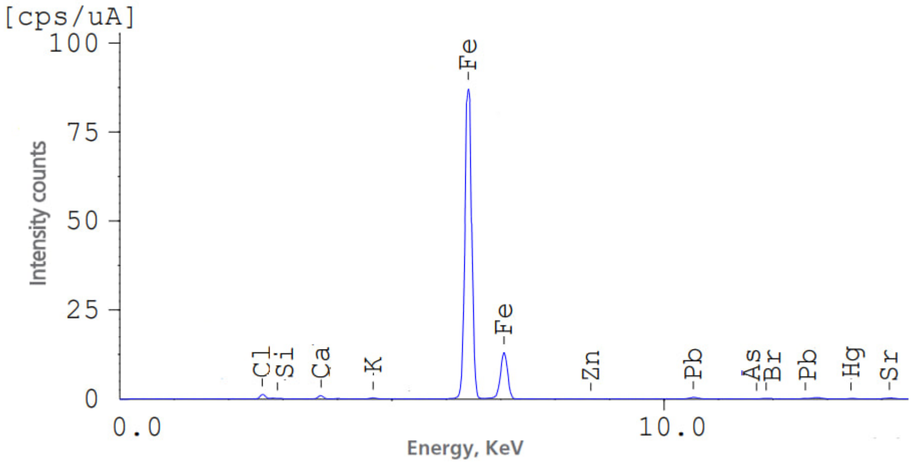

Figure 3 – X-ray fluorescence analyzer spectrum of salt obtained as a result of evaporation of Khaudak water.

Table 2 – Quantitative result of X-ray fluorescence analyzer of the salt obtained as a result of the evaporation of water from in Khaudak.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	70.093 %	[0.833]	Quan-FP	ClKa	2.2786
Ca	26.982 %	[0.429]	Quan-FP	CaKa	3.5882
K	1.273 %	[0.101	Quan-FP	K Ka	0.0399
Sr	0.703 %	[0.009]	Quan-FP	SrKa	10.1298
Br	0.514 %	[0.011]	Quan-FP	BrKa	0.6417
S	0.235 %	[0.054]	Quan-FP	SKa	0.0980
Fe	0.130 %	[0.006]	Quan-FP	FeKa	0.1543
Zn	0.070 %	[0.008]	Quan-FP	ZnKa	0.0229

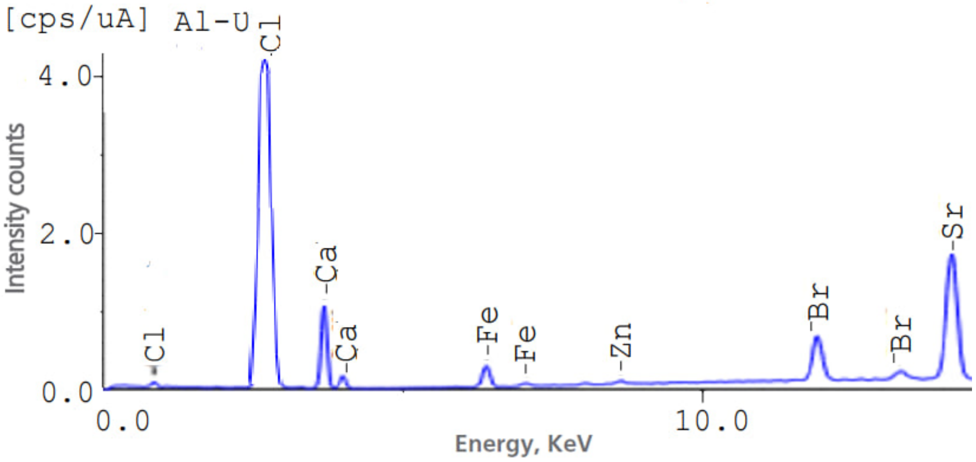

Figure 4 – X-ray fluorescence analyzer spectrum of 1-week-old water of Uchkizil water, sample 1.

Table 3 – Quantitative X-ray fluorescence analyzer result of Uchkizil water, sample 1.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	11.715 %	[0.155]	Quan-FP	ClKa	1.8506
Ca	2.110 %	[0.039]	Quan-FP	CaKa	2.6479
Fe	0.073 %	[0.002]	Quan-FP	FeKa	1.0001
Sr	0.125 %	[0.002]	Quan-FP	SrKa	20.0853
K	0.064 %	[0.007]	Quan-FP	K Ka	0.0176
Br	0.047 %	[0.001]	Quan-FP	BrKa	0.6858
Zn	0.006 %	[0.001]	Quan-FP	ZnKa	0.0221
H_2O	85.860 %	[------]	Balance	--------	--------

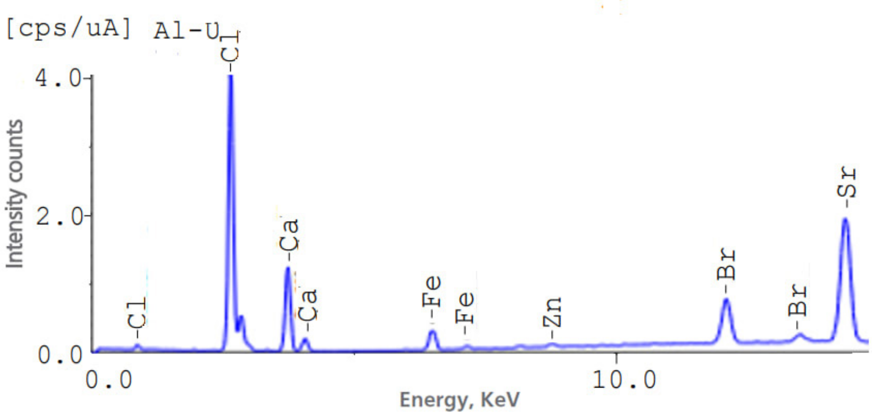

Figure 5 – The X-ray fluorescence analyzer spectrum of 2-week-old water of Uchkizil water, sample 2.

Table 4 – Quantitative X-ray fluorescence analyzer result of Uchkizil water, sample 2.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	11.754 %	[0.155]	Quan-FP	ClKa	1.8559
Ca	2.120 %	[0.039]	Quan-FP	CaKa	2.6555
Sr	0.125 %	[0.002]	Quan-FP	SrKa	20.0113
Fe	0.071 %	[0.002]	Quan-FP	FeKa	0.9690
K	0.062 %	[0.007]	Quan-FP	K Ka	0.0170
Br	0.048 %	[0.001]	Quan-FP	BrKa	0.7012
Zn	0.005 %	[0.001]	Quan-FP	ZnKa	0.0202
H_2O	85.815 %	[------]	Balance	--------	--------

As can be seen from this table, little change has occurred compared to the value of iron in the previous table.

Uchkizil water was left in the open air for 6 weeks after being extracted from the ground, under the influence of sunlight. Sample 3. Then it was analyzed in an X-ray fluorescence analyzer (Fig. 6, Table 5).

The underground water of Khaudak was kept for 4 weeks in an open environment under the influence of light, then the analysis of the analyzer was carried out (Fig. 7, Table 6).

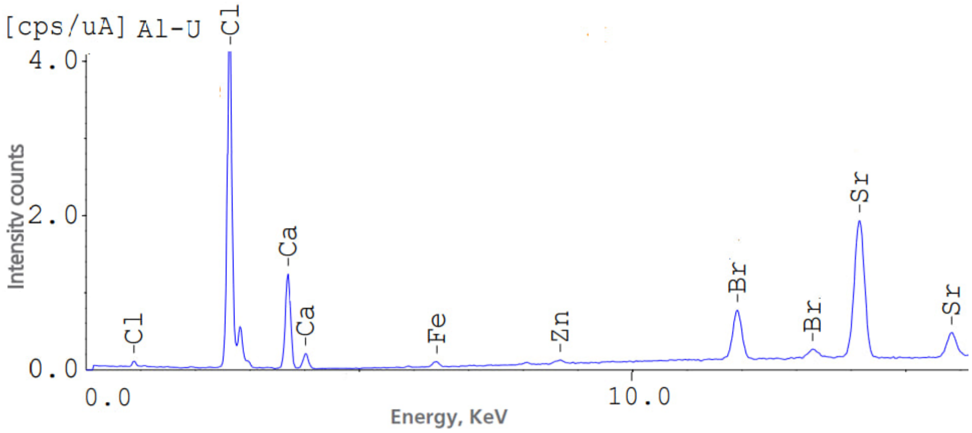

Figure 6 – The spectrum of Uchkizil water 2 weeks in an X-ray fluorescent analyzer, sample 3.

Table 5 – Uchkizil water, quantitative result of sample 3 from X-ray fluorescent analyzer.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	12.099 %	[0.157]	Quan-FP	ClKa	1.9070
Ca	2.171 %	[0.040]	Quan-FP	CaKa	2.6739
Sr	0.124 %	[0.002]	Quan-FP	SrKa	19.7715
K	0.056 %	[0.006]	Quan-FP	K Ka	0.0151
Br	0.048 %	[0.001]	Quan-FP	BrKa	0.6904
Fe	0.016 %	[0.001]	Quan-FP	FeKa	0.2160
Zn	0.006 %	[0.001]	Quan-FP	ZnKa	0.0214
H_2O	85.481 %	[------]	Balance	--------	--------

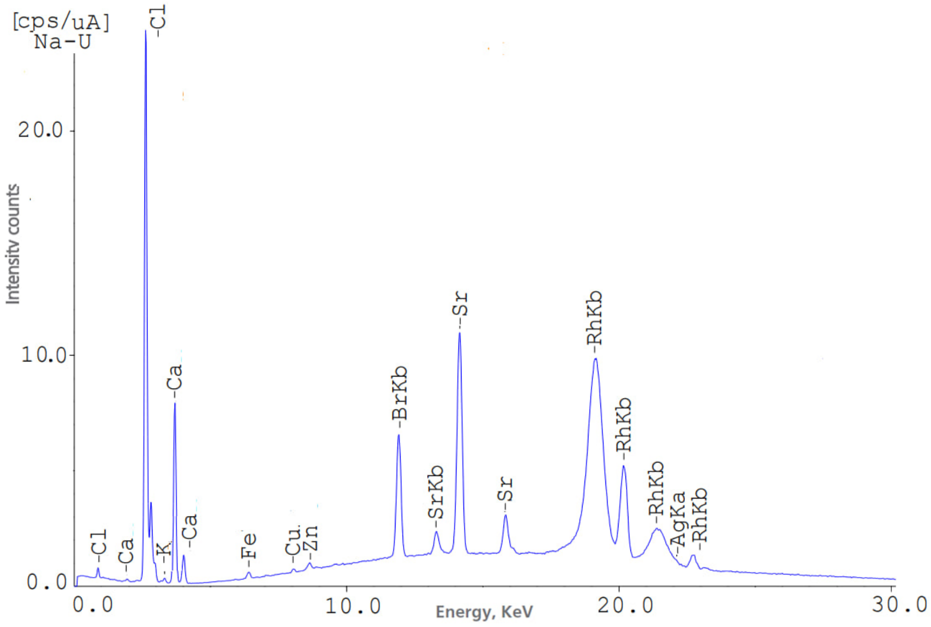

Figure 7 – X-ray fluorescence analyzer spectrum of Khaudak 1-month-old water.

Table 6 – Quantitative result of X-ray fluorescence analyzer of Khaudak 1 month water.

Analyte	Result (ppm)	Result (%)	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	133682.9 ppm	13.368	[823.663]	Quan-FP	ClKa	128.4750
Ca	17710.98 ppm	1.771	[184.954]	Quan-FP	CaKa	47.1744
K	829.649 ppm	0.083	[94.152]	Quan-FP	K Ka	1.1207
Sr	662.721 ppm	0.066	[4.040]	Quan-FP	SrKa	106.8234
Br	426.566 ppm	0.043	[3.461	Quan-FP	BrKa	53.5927
Fe	71.311 ppm	0.0071	[6.599]	Quan-FP	FeKa	2.0086
Ag	52.328 ppm	0.0052	[10.472]	Quan-FP	AgKa	2.1917
Zn	41.250 ppm	0.0041	[4.122]	Quan-FP	ZnKa	2.6987
Cu	18.408 ppm	0.0018	[4.054]	Quan-FP	CuKa	1.0050
H_2O	84.650 %	84.650	[------]	Balance	--------	--------

It can be seen that the values in this table are very close to the elements and their amounts in the above Uchkizil water.

There is an oil production area around Khaudak water, and the following indicators were obtained when analyzing the composition of the water separated from this oil (Figure 8, Table 7).

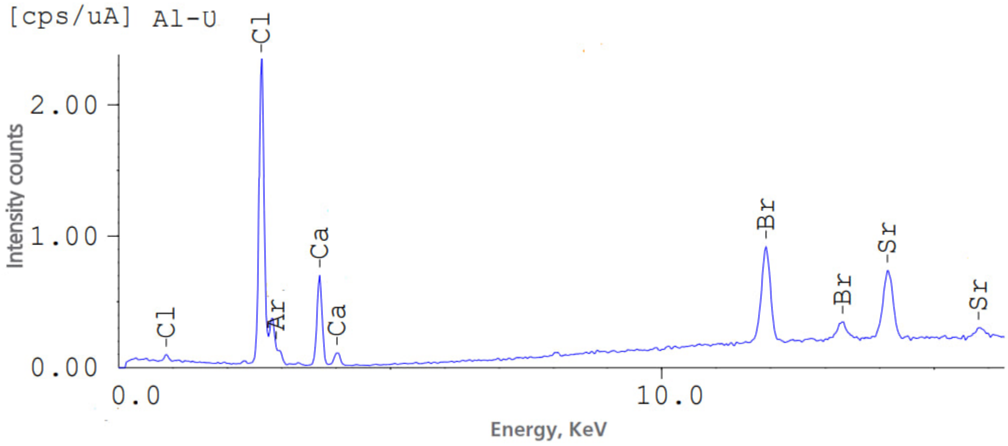

Figure 8 – Spectrum of oil water in X-ray fluorescence analyzer.

Table 7 – Quantitative result of oil water from X-ray fluorescence analyzer.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Cl	5.189 %	[0.092]	Quan-FP	ClKa	1.0113
Ca	0.695 %	[0.017]	Quan-FP	CaKa	1.5505
K	0.031 %	[0.003]	Quan-FP	K Ka	0.0148
Br	0.030 %	[0.001]	Quan-FP	BrKa	0.7932
Sr	0.021 %	[0.001]	Quan-FP	SrKa	5.6960
P	0.004 %	[0.011]	Quan-FP	P Ka	0.0014
H_2O	94.030 %	[------]	Balance	--------	--------

4 Results and discussion

The contents of Uchkizil, Khaudak underground waters, which are rich in various mineral salts, and water separated from Khaudak oil were studied. According to it, the change and stability indicators of the amount of Ca, Fe, Br and Sr in Uchkyzil water based on 3 samples are shown in Fig. 9.

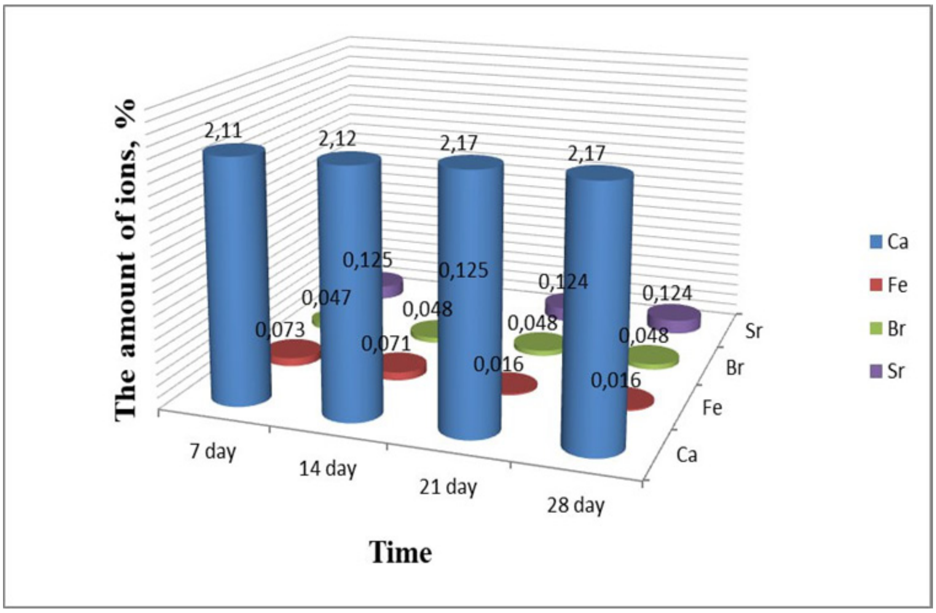

Figure 9 – Quantitative diagram of elements Ca, Fe, Br and Sr in the water of Uchkizil.

In this diagram, it can be seen that the concentration of Br and Sr compounds in the waters presented based on 3 samples has not changed for different periods. If we explain that strontium salts dissolve well in water and do not form precipitates, we can see that Br is stable to the effects of external environmental factors, such as the sun, ultraviolet rays, and air. It can be seen that the amount of Fe has decreased by a small amount.

A sample of 5 l of Haudak water was taken and a 30% Fe (III) chloride solution was added to it, shaken for 5 minutes, and kept for 1 month. As a result, a brown-red precipitate was formed, the main part of which consists of iron and chlorine, and a smaller part of compounds with iodine and similar elements. The composition of this precipitate was imaged in an X-ray fluorescence analyzer (Fig. 10, Table 8).

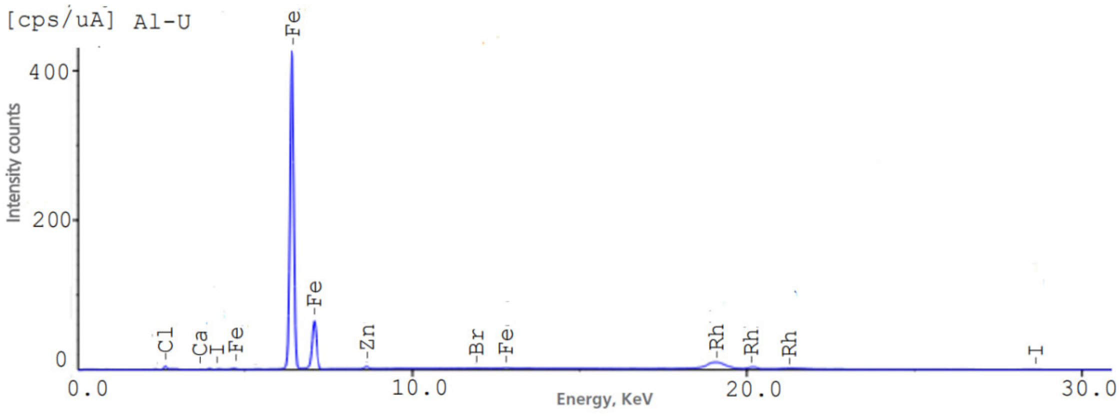

Figure 10 – The spectrum of the sediment in the X-ray fluorescence analyzer.

Table 8 – Quantitative result of precipitation obtained from X-ray fluorescence analyzer.

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Fe	84.717 %	[0.243	Quan-FP	FeKa	3046.5512
Cl	10.196 %	[0.160]	Quan-FP	ClKa	11.9197
S	1.614 %	[0.066]	Quan-FP	SKa	1.5363
I	1.317 %	[0.047]	Quan-FP	I Ka	7.3613
Zn	0.977 %	[0.037]	Quan-FP	ZnKa	23.7076
P	0.701 %	[0.104]	Quan-FP	P Ka	0.2426
Ca	0.211 %	[0.027]	Quan-FP	CaKa	0.5385
Br	0.135 %	[0.026]	Quan-FP	BrKa	5.9278
Cs	0.131 %	[0.086]	Quan-FP	CsLa	0.7456

The precipitated part is 1.24 g and we can see that it consists of 1.317% I iodine. This value is 7.66% of the total iodine in water (21.32 mg/l).

Based on the analytical analysis presented above, the quantitative ratios of certain elements in Uchkizil, Khaudak and Khaudak oil waters oxidized based on $FeCl_3$ salt of Khaudak water are presented in the diagram in Fig. 11.

In this picture, it can be seen that the amount of iodine in various water samples containing iodine has decreased or completely disappeared due to the passage of time due to external factors. Only if the obtained water sample is oxidized in a short time or separated using special sorbents, it becomes important for practice.

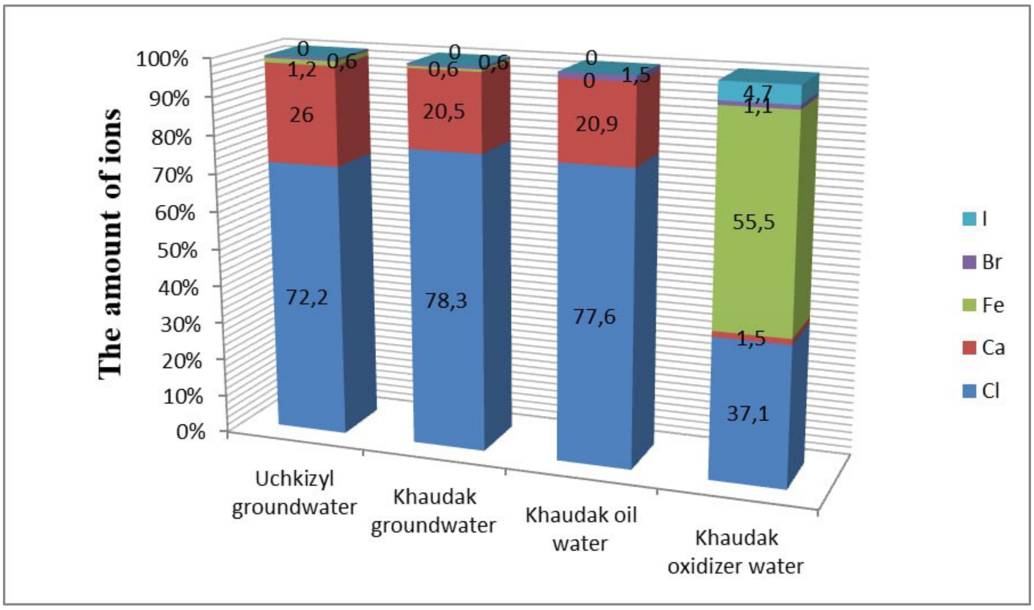

Figure 11 – Quantitative description of water samples and sediment content obtained from them.

5 Conclusion

In this study, several groundwater samples containing iodine were studied by X-ray fluorescence analysis. It has been proven that certain salts in water form precipitates due to the decrease in solubility as a result of lowering the temperature to room temperature, and they form precipitates as a result of hydrolysis. In this case, it was determined that iron salts have a high precipitation forming ability. It has been proven that iodine contained in waters with high mineral content is oxidized and released into the air under the influence of ultraviolet rays contained in sunlight and air oxygen. It was found that iron compounds in salt water settle over time.

As a result of adding special oxidants for iodine to the water of the newly obtained sample, it was observed that iodine precipitated over a certain period of time. The reasons for the relatively high amount of iodine in the mud at the bottom of the water, given in the above literature, have been confirmed. 7.66% of the iodine in the water containing 21.32 mg/l was precipitated by oxidation.

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Information about authors:

Uralov Nuriddin Bekmuradovich, PhD doctorate at the Faculty of Chemistry, Termez State University, (Termez, Uzbekistan), e-mail: uralovnuriddin1991@gmail.com

Turaev Khayit Khudaynazarovich, Doctor of Chemical Sciences, Professor at the Faculty of Chemistry, Termez State University, (Termez, Uzbekistan), e-mail: hhturayev@rambler.ru

Normurodov Bakhtiyor Abdullayevich, DSc associate professor at the Faculty of Chemistry, Termez State University, (Termez, Uzbekistan), e-mail: normurodovbakhtiyor@gmail.com

Umbarov Ibragim Amanovich, Doctor of technical sciences, Professor at the Faculty of Chemistry, Termez State University, (Termez, Uzbekistan), e-mail: umbarov@mail.ru

Kurbonov Fakhriddin Bobomurotovich, Lecturer at the Faculty of Chemistry of Termez State University, (Termez, Uzbekistan), e-mail: bekzur22@gmail.com