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STUDYING THE COMPOSITION OF IODINE-RICH KHAUDAK AND UCHKIZIL UNDERGROUND BRINES IN THE SOUTHERN REGIONS OF UZBEKISTAN BASED ON X-RAY FLUORESCENCE ANALYZER

This study investigates the underground brines of Khaudak and Uchkizil in southern Uzbekistan, which are rich in various mineral salts, including iodine. The chemical composition of these brines and their time-dependent transformations under environmental exposure were analyzed using X-ray fluorescence spectroscopy (XRF).

The results demonstrate that iron compounds present in the brine gradually precipitate over time. The original iron content of the water was approximately 0.130%, with up to 65% of it forming sediment. Additionally, upon adding specific iodine oxidizing agents and storing the sample for one month, a reddish-brown precipitate was formed. This precipitate mainly consisted of iron and chlorine compounds, with minor contributions from iodine and similar elements. The precipitate contained 1.317% iodine, corresponding to 7.66% (21.32 mg/L) of the total iodine in the Khaudak water sample. Exposure to ultraviolet radiation caused further oxidation and volatilization of iodine into the atmosphere. These findings emphasize the importance of timely iodine separation for recovery from brines under natural and controlled conditions.

Keywords: Khaudak brine, Uchkizil brine, iron(III) chloride, iodine precipitation, X-ray fluorescence spectroscopy.

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Өзбекстанның оңтүстік аймақтарындағы йодқа бай Хаудак және Учкизил жер асты суларының құрамын рентгендік флуоресцентті анализатор негізінде зерттеу

Бұл **зерттеуде** Өзбекстанның оңтүстігінде орналасқан және йод қосылған әртүрлі минералды тұздардан тұратын Хаудак және Учкизил жер асты суларына арналған және бұл сулардың өзара ұқсастығы мен олардың уақытқа байланысты өзгерістері талданған. Сыртқы ортаның әсерінен су құрамынан алынған бірнеше сынамалар мен бөлшектердің өзгерістері анықталды.

Алынған тұзды ерітінді құрамы рентгендік флуоресцентті анализаторды талдау негізінде сипатталған. Оған сәйкес уақыт өте келе судағы темір қосылыстары шөгіндіге түсетіні анықталды. Бастапқыда судың құрамында темірдің шамамен 0,130% - ы болады және белгілі бір уақыт аралығында баяу батып кетеді. Бұл тұнбаның құрамында темірдің мөлшері шамамен 65% құрайды. Сонымен қатар, жаңадан алынған су үлгісінде йодқа арнайы тотықтырғыштарды қосып, оны 1 ай бойы сақтау арқылы йодтың белгілі бір бөлігінің тұнбаға түскені байқалды. Нәтижесінде негізінен темір мен хлордан және сирек йодпен және ұқсас элементтермен қосылыстардан тұратын қоңыр-қызыл түсті тұнба пайда болды. Тұнбаның құрамында 1,317% йод бар, ал Хаудак суындағы йодтың 7,66% (21,32 мг/л) тотығу арқылы тұнбаға түскен. Алайда, бұл су ашық ауада ультракүлгін сәуленің әсеріне ұшыраған кезде, судағы йодтың тотығуы және ауа қабатына шығуы байқалды.

Түйін сөздер: Хаудак тұзды суы, Учкизил тұзды суы, темір(III) хлориді, йод тұнбасы, рентгендік флуоресценттi талдау.

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Изучение состава йодсодержащих подземных рассолов Хаудака и Учкизила в южных районах Узбекистана на основе рентгенофлуоресцентного анализа

В настоящем исследовании рассматриваются подземные рассолы Хаудака и Учкизила, расположенные в южных регионах Узбекистана, обладающие высокой минерализацией и содержанием йода. Состав и трансформация рассолов при воздействии внешней среды исследованы с применением рентгенофлуоресцентного анализа (XRF).

Показано, что содержащиеся в воде соединения железа со временем постепенно выпадают в осадок. Исходное содержание железа составляло около 0,130%, из которых до 65% оседает. При добавлении специализированных окислителей йода и выдержке образца в течение одного месяца наблюдалось образование буро-красного осадка, преимущественно состоящего из соединений железа и хлора, а также незначительного количества йодсодержащих соединений. Выявлено, что содержание йода в осадке составило 1,317 %, что соответствует 7,66 % от общего количества йода в воде Хаудака (21,32 мг/л). Воздействие ультрафиолетового излучения приводило к дальнейшему окислению и улетучиванию йода в атмосферу. Таким образом, полученные результаты исследования подчеркивают необходимость своевременной экстракции йода из минеральных вод для предотвращения его потерь в естественных условиях.

Ключевые слова: Хаудакский рассол, Учкизильский рассол, хлорид железа (III), осаждение йода, рентгенофлуоресцентный анализ.

Introduction

Interest in underground brines has increased significantly in recent years due to their industrial and medical importance. Some of these highly mineralized waters are used for therapeutic purposes, while others serve as valuable sources for the extraction of trace elements such as iodine and bromine. Studies on iodine-rich groundwater in South China have revealed average iodine concentrations of 890 μg/L, with maximum levels reaching 6,350 μg/L—far exceeding the WHO's recommended range of 5–300 μg/L. Iodine enrichment typically occurs under acidic conditions (pH ≈ 6.6) and in low-redox environments (Eh ≈ 198.4 mV) [1]. Iodine in water exists in various forms, including molecular iodine (85.6%), iodide ions (3.2%), iodate (9.1%), and iodine chloride (2.1%) [2].

Microbial activity plays a critical role in iodine cycling through oxidation, reduction, volatilization, and deiodination processes. Recent metagenomic analyses of deep groundwater in the North China Plain have identified microbial gene clusters linked to dissimilatory iodine reduction. Iron- and sulfur-reducing bacteria can further mobilize iodine by facilitating the reductive dissolution of iron minerals [3]. In the CIS region, major iodine-rich aquifers have been reported in Turkmenistan, Russia, Azerbaijan, Uzbekistan, and Ukraine. Iodine reserves in these countries are distributed as follows: Turkmenistan (40%), Russia (34%), Azerbaijan (22%), Ukraine (3%), and Uzbekistan (1%) [4]. In studies related to sea mud, the amount of iodine was ten times higher than in water and was 0.002-0.01%, and in oil and adjacent waters it was 0.01%; the amount of iodine from mineral ore deposits is around 1% in Chilean saltpeter deposits, 280 mg/kg with phosphorites, and up to 6 mg/kg in some coal deposits. It also contains up to 0.1 mg/kg of table salt obtained from seawater, 0.25 mg/kg of rock salt, and 0.06 mg/kg of potassium salts. In this place, the lack of iodine in salt deposits is recognized by the absence of the ability of iodide ions to isoform the position of chlorine in the crystal lattice. [5]. The main amount of iodine is located in the center of this basin, with iodine concentrations ranging from 3.31 to 1890 μg l(-1). Most samples with iodine concentrations above 500 μg l(-1) were obtained from wells between 75 and 120 m deep.

High pH and reducing environment are favorable for the enrichment of iodine in groundwater, which is 63.2-99.3% of total iodine. Sediment samples from the drilled well contained 0.18-1.46 mg kg(-1) of iodine, which was moderately correlated with total organic carbon (TOC). According to experiments, iodine binds mainly to iron oxyhydroxides and organic substances in sediments. Iodine mobilization processes are proposed to involve the reductive dissolution and transformation of iron oxyhydroxide between iodide, iodine, and organic iodine caused by microbial activity under alkaline and reducing conditions [6]. The conversion of iodide to elemental iodine in an acidic solution significantly resists the formation of iodide or has almost no effect on the conversion of iodide to molecular iodine. In some versions of the study, pre-acidification of aqueous brine with metered hydrochloric or sulfuric acid is the optimal condition for this method [7]. As a continuation of the above scientific work, salt water was heated to a sufficient level, the pH level was brought to 2 to 4, and an oxidizing agent was added to form free iodine from the iodide in the water, and it was desorbed by airflow [8]. Sodium chloride, which contains iodine, is concentrated in underground saltwater using an electrodialysis device. In Japan, many types of table salt are produced industrially in this way. In this case, crystals containing sodium chloride as the main component are precipitated and separated into crystals by separating methods [9]. The results of research on the adsorption of organic iodine on activated carbon [10, 11] and similarly synthesized sorbents are also important [12-14].

Brines from Khaudak and Uchkizil in Uzbekistan are characterized by exceptionally high mineralization levels (up to 300 g/L) and significant iodine content (>20 mg/L), making them promising for iodine extraction. This study aims to analyze the compositional features of these brines using XRF spectroscopy and evaluate iodine transformation and stability under environmental conditions [15].

Methods

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%.

Experimental part

Khaudak water is extracted from the ground from a depth of about 3,000 meters, and three-red water 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).

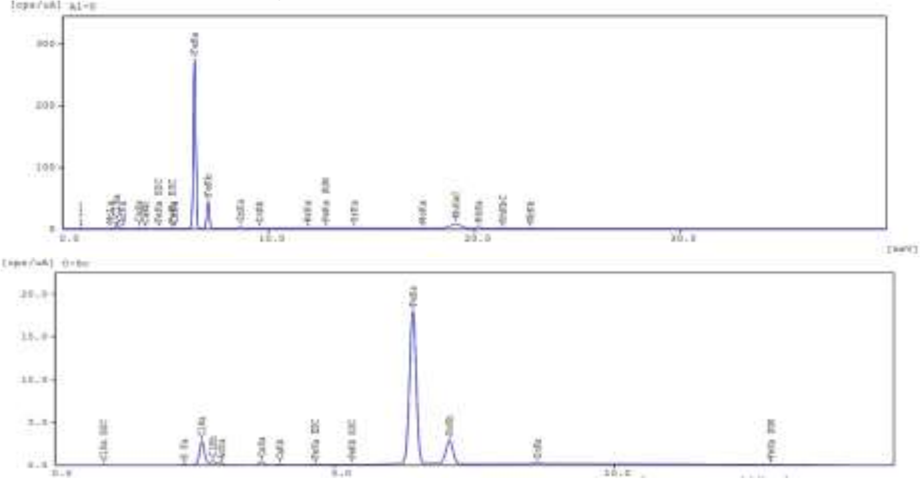

Figure 1 – X-ray fluorescence analyzer spectrum of sediment taken from Uchkizil water

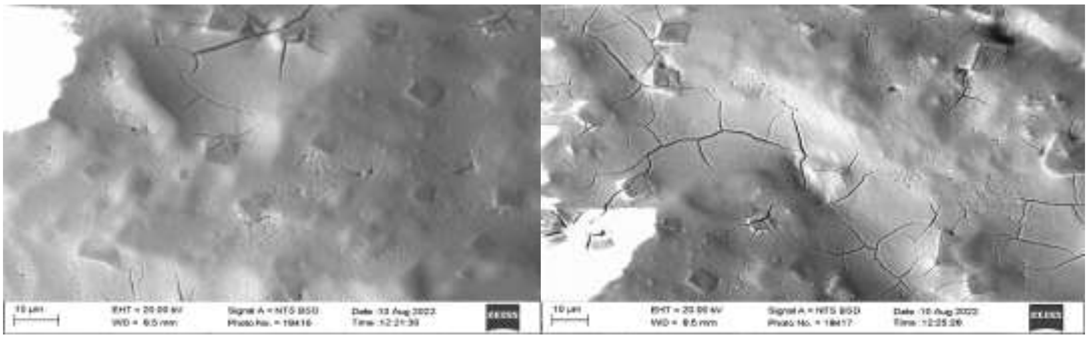

Figure 2 – SEM image of the sediment taken from Uchkizil water

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).

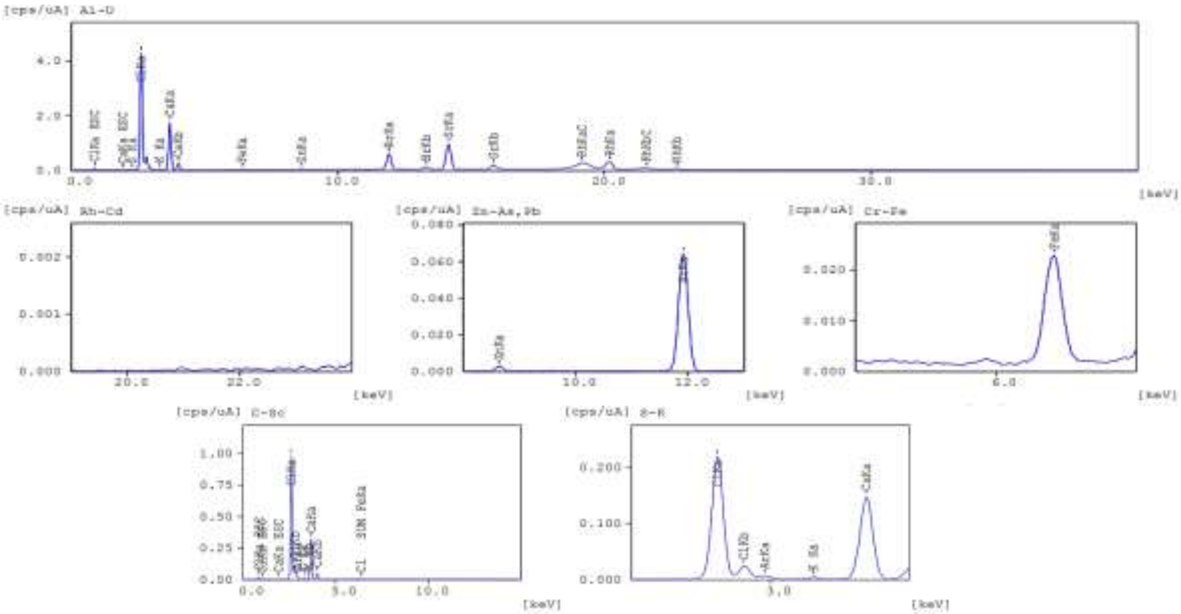

Figure 3 – X-ray fluorescence analyzer spectrum of salt obtained as a result of evaporation of Khaudak water

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

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).

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)

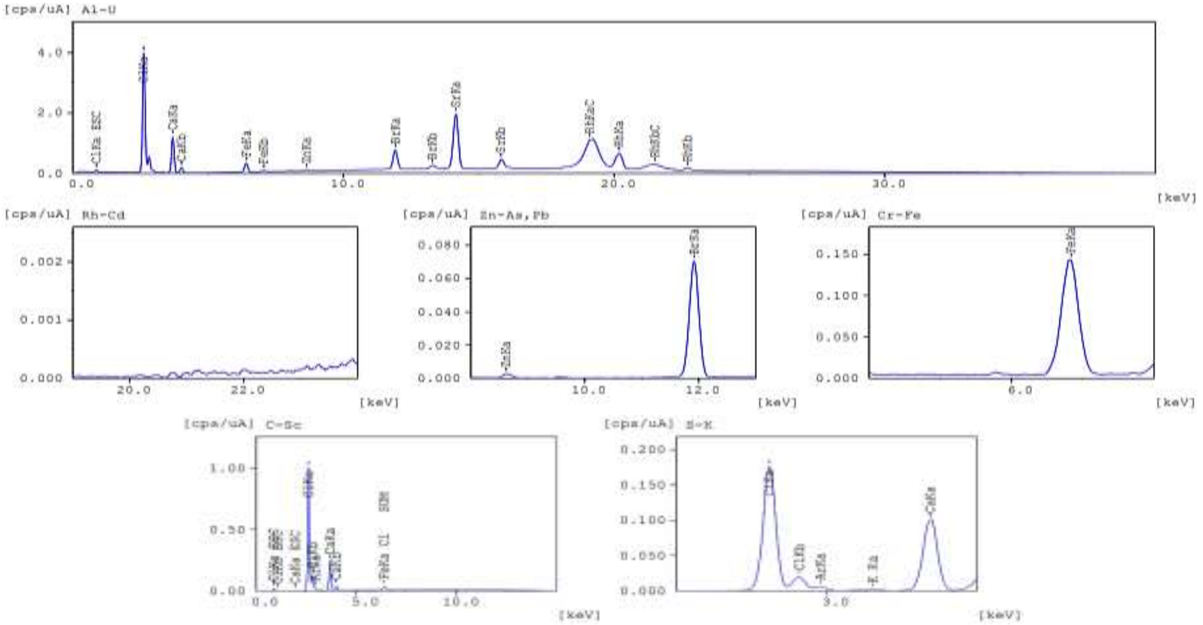

Figure 4 – X-ray fluorescence analyzer spectrum of 1-week-old water of Uchkizil water, sample 1.

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	--------	--------

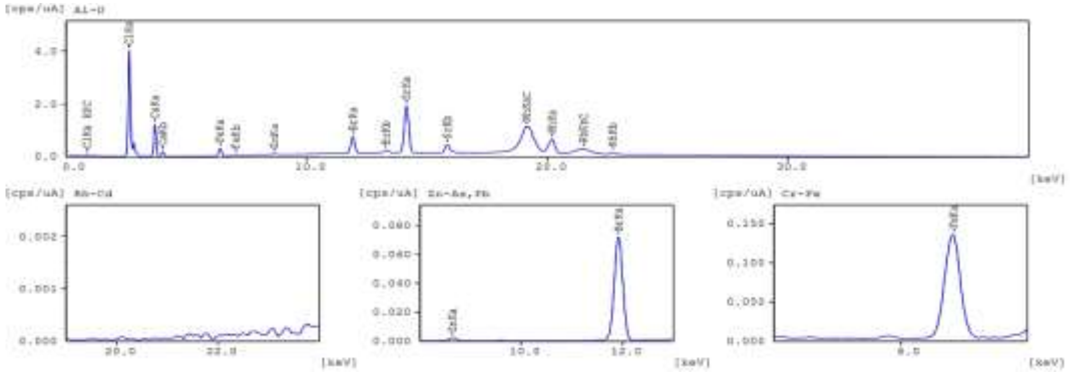

Figure 5 – The X-ray fluorescence analyzer spectrum of 2-week-old water of Uchkizil water, sample 2.

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	--------	--------

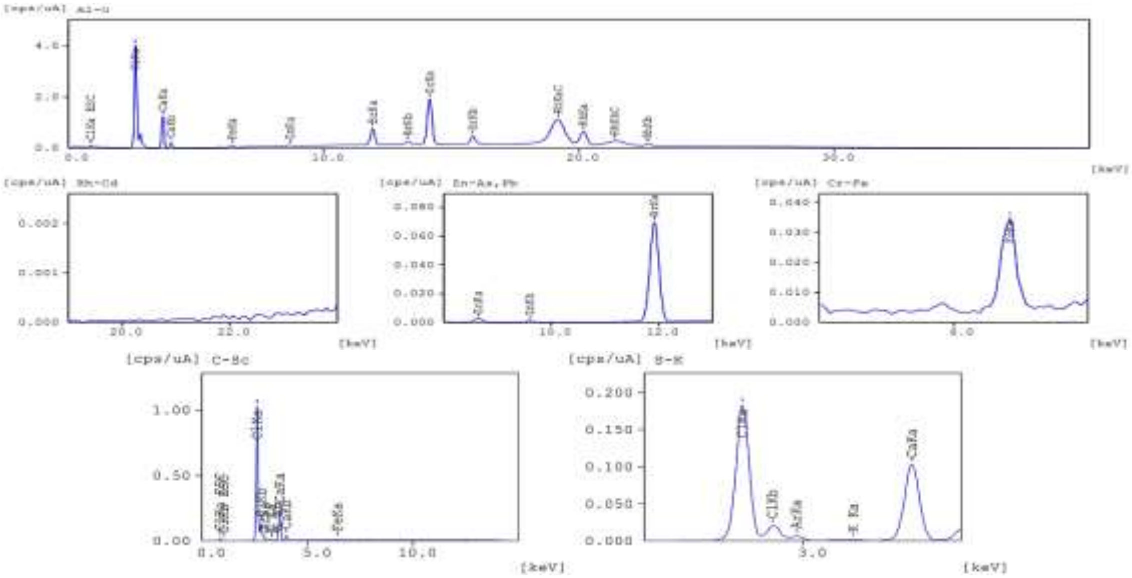

Figure 6 – The spectrum of Uchkizil water 2 weeks in an X-ray fluorescent analyzer, sample 3.

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	--------	--------

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).

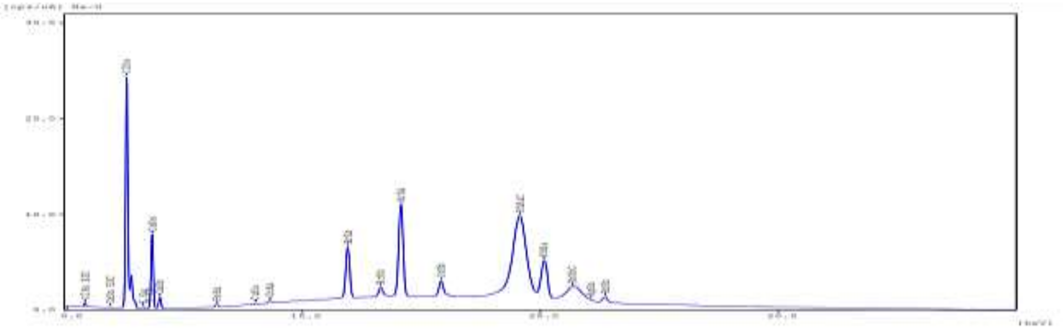

Figure 7 – X-ray fluorescence analyzer spectrum of Khaudak 1-month-old water

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).

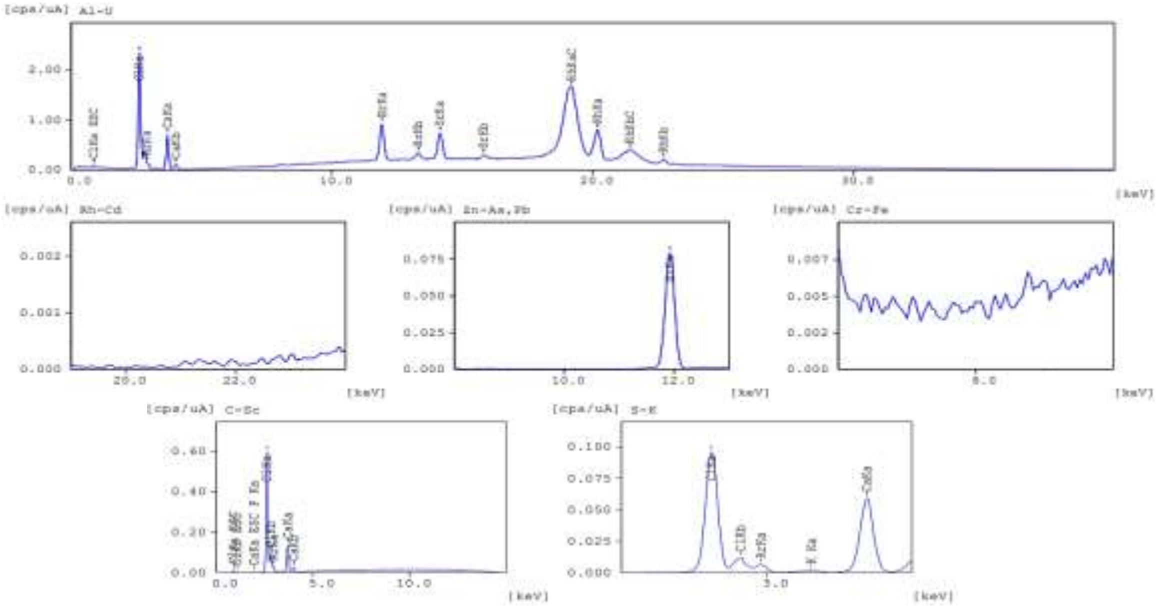

Figure 8 – Spectrum of oil water in X-ray fluorescence analyzer.

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	--------	--------

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.

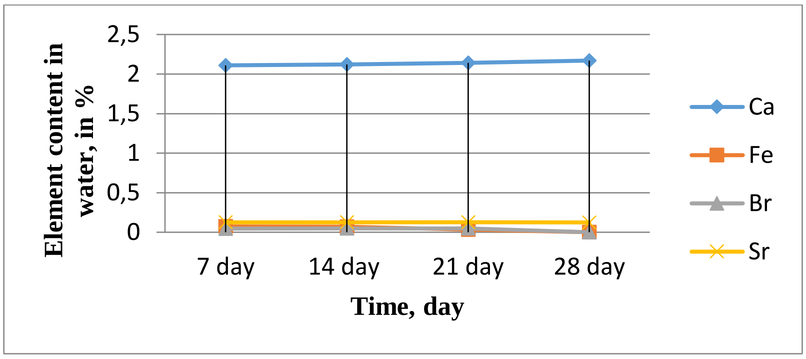

Figure –. Quantitative diagram of elements Ca, Fe, Br and Sr in the water of Uchkizil

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).

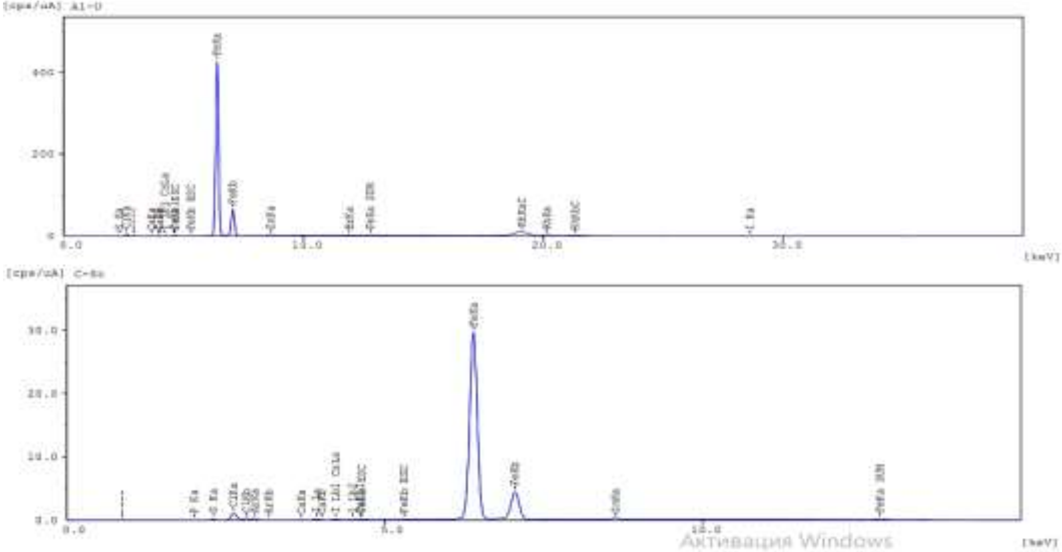

Figure 9 – The spectrum of the sediment in the X-ray fluorescence analyzer.

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.

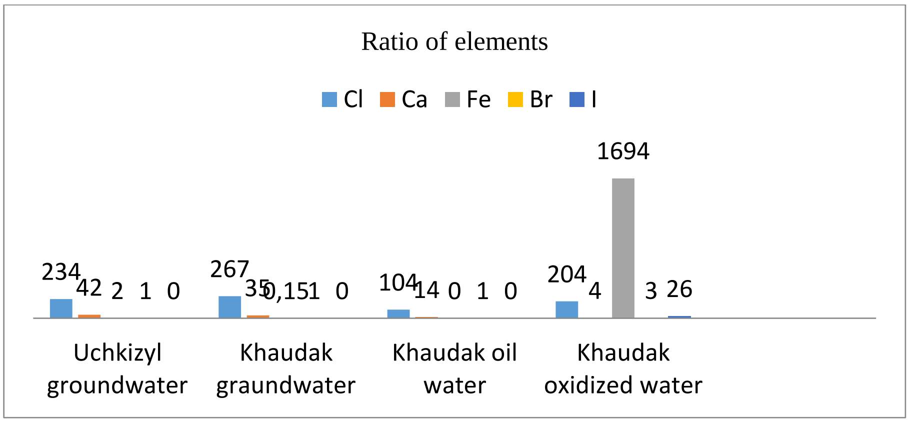

Figure 11 – Quantitative description of water samples and sediment content obtained from them.

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.

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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