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ASSESSMENT OF HEAVY METAL AND RADIONUCLIDE CONTAMINATION IN WATER SAMPLES TAKEN FROM NAKHCHIVAN REGION OF AZERBAIJAN

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Abstract: This study was conducted to determine major cation (Ca, K, Mg, and Na), heavy metal (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, V and Zn) and radionuclide (U, Th) contents of the water samples taken from the four different sampling point in the Nakhchivan Autonomous Republic. Elemental concentrations were determined by inductively coupled plasma mass spectrometer system. Measured concentrations of the dissolved metal elements (except for As, Mg, and Na) in investigated water samples from Aras River and Shirarkh Canal were under the limit values presented in World Health Organization (WHO) guideline. Results of the analysis showed that all measured metal concentrations (dissolved) in investigated Arpachay River water samples also were below WHO guideline limit value. Determined radionuclide (U and Th) concentrations in all investigated samples were also below suggested values of the WHO guideline. Heavy metal pollution index (HPI), non-cancer risk (HQ) and cancer risk (CR) were calculated for investigated water samples. Calculated HPI values in this research classified investigated water samples as mediumly polluted with heavy metals. The carcinogenic risk of As through ingestion exceeded the tolerable risk of $1x10^{-4}$ indicating that the usage of these river waters as a drinking water for a long period could raise the probability of cancer. Calculated HQ values are bigger than 1 in Aras River and Shirarkh Canal waters which means using these waters for drinking purposes raises the potential risk for human health.

Key words: Azerbaijan, Aras River, heavy metals, radionuclides, pollution, cancer risk

1. Introduction

Metal contamination of riverine ecosystems has become a serious environmental problem due to the rapid urbanization and industrialization [1-3]. Metal contaminants enter into river water through anthropogenic sources such as long-term disposal of untreated and partially treated industrial effluents containing toxic metals, and indiscriminate use of metal-containing fertilizers and pesticides in agricultural fields. Main anthropogenic sources of heavy metal contamination are mining, disposal of untreated and partially treated effluents contain toxic metals, as well as metal chelates from different industries and indiscriminate use of heavy metal-containing fertilizer and pesticides in agricultural fields [4-6]. Metal contaminants are a major cause of concern for the aquatic environment because of their toxicity, abundance, persistence, and subsequent accumulation in aquatic habitats [7,8]. Heavy metals have toxic properties, leading to adverse effects on human health even in small doses. The effects of exposure to these environmental pollutants on human health are well known. Toxicity due to heavy metals can result in significant illness and reduced quality of life [9,10]. Recognition of the importance of trace metal concentrations evidence in natural waters and or environment is growing in pollution monitoring studies.

Nowadays, globally, heavy metals have been taken into consideration owing to their toxicity, ability to accumulate in the biota and adverse health effects even at low concentrations. Investigation was conducted to determine major cation (Ca, K, Mg and Na), heavy metal (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, V, U,) and radionuclide (U, Th) contents of the water samples taken from the four different sampling point in the Nakhchivan Autonomous Republic. Nakhchivan is a semi-desert region that is separated from the main portion of Azerbaijan by Armenia. The Zangezur Mountains make up its border with Armenia while the Aras River defines its border with Iran. The Aras River supplies water for agricultural needs and the hydroelectric dam generates power for both Azerbaijan and Iran.

2. Materials and Methods

The Aras River, the second largest river after the Kura River of the South Caucasus approximate length of 1072 km, is the common boundary between Turkey, Iran, Azerbaijan, and Armenia, and finally reaches to the Caspian Sea. Originating from Binaguldaq Mountains in Turkey, Aras River lies within a water catchment of around 100220 km^2. The average maximum flow within the study area is 1100 m^3/sec at Aras Dam and 2600 m^3/sec at Muqan Dam. However, the mentioned values may descend to 32 and 180 m^3/sec in arid seasons [11]. This river supplies potable water to tens of cities and villages, irrigation water to thousands of hectares of irrigation land, water to fish husbandry projects and water to numerous industrial units [12]. Accordingly, river water quality is exposed to a potential threat caused by agricultural, industrial, and residential land uses. Recently, the rapid growth of industrial and agricultural activities within the Aras River basin in Iran, Armenia, and Azerbaijan have been adversely affected by the river ecosystem. The total annual flow from Armenia to Azerbaijan through the Aras River and its tributaries (Arpa, Vorotan, and Vokhchi) is estimated at about 5.62 km^3, and from the Islamic Republic of Iran is estimated at 7.5 km^3.

Water samples were collected from the Aras River (two points), Arpachay River (one point) and Shirarkh Canal (one point) using standard polyethylene water sampler, which was rinsed a few times with river water from the sampling point before representative sampling from 15-30 cm below the water surface. Two hundred milliliters of water were filtered through a 0.45μm membrane filter using a plastic filtration assembly without a pump. A few drops of high-purity nitric acid were added to the filtrate to adjust to pH < 2. Samples were stored at 4 °C during transportation to the laboratory. Between each sampling, the water sampler was soaked with10% v/v nitric acid and rinsed with ultrapure water. All plastic-ware sample bottles, pipette tips, filtration unit, and flasks were soaked in 10% v/v HNO_3 for 24 hours and rinsed with ultra-pure water before being used. Milli-Q ultra-pure water (resistivity 18.2 MΩ·cm, pH (5.5-6.5)) was used throughout, and in all laboratory operations. In the laboratory, by adding an appropriate volume of nitric acid, the acid concentrations of the samples were adjusted to approximately 1% (v/v) nitric acid solution. Multi-element calibration working standard solutions were prepared (in 1% HNO_3) by appropriate dilution of 10 mg /L multi-element stock standard solutions - Environmental Calibration Standard - Part #5183-4688 in 5% HNO_3.

Elemental concentrations in water samples were measured using an inductively coupled plasma mass spectrometer (ICP-MS). The Agilent 7700x Series ICP-MS applied to analyze the water samples.

The method is based on the direct introduction of samples into an inductively coupled plasma mass spectrometer (ICP-MS), without any chemical pre-treatment. An Agilent 7700x ICP-MS system was used to measure each sample in helium mode, using standard Agilent-recommended auto- tuning for robust tuning conditions (around 1.0 % CeO/Ce).

3. **Results and Discussion**

The samples were analyzed for Al, As, Ba, Cd, Cr, Cu, Fe, Mn, Pb, Cu, Ni, V, U, Th, Zn and major cations using an Agilent model 7700x inductively coupled plasma mass spectrometer. The blank and calibration solutions were measured in optimized conditions. The calibration curve was automatically plotted by the instrument. The linear correlation coefficient (R) in all calibration curves was higher than 0.9995. Instrument drift and matrix effects during measurement were corrected using Sc, Ge, Rh, In, Tb, and Bi, containing internal standards (which were prepared by appropriate dilution of stock ICP-MS Internal Standard (Mix Part# 5188-6525)). After appropriate dilution, the solution added on-line during analysis using a second channel of the peristaltic pump. For quality control purposes, duplicate samples, SRM samples were analyzed.

The 7700x ICP-MS Operating Conditions were used for He mode. CRMs were purchased from the NRCC (National Research Council of Canada). Absolute standards were analyzed to validate our procedure: SLRS-5 (river water) and SRM 1640.

Determined elemental concentrations of the water samples are presented in Table 1.

Table 1. Element concentrations in water samples

	Unit	**Nakhchivan Sederek-Aras**	**Nakhchivan Sederek-Arpachay**	**Nakhchivan Babek Aras**	**Nakhchivan Sederek-Shirarkh**	**Mean (Aras)**	**WHO**
Cu	ug/L	1.456	0.814	1.790	1.072	1.623	**2000**
Ni	ug/L	3.030	0.912	2.697	2.111	2.864	**70**
Mn	ug/L	1.539	13.510	1.258	1.544	1.399	**100**
Cr	ug/L	0.517	0.127	0.158	0.176	0.338	**50**
Pb	ug/L	0.056	0.035	0.075	0.040	0.066	**10**
Cd	ug/L	<0.02	<0.02	<0.02	<0.02	<0.02	**5**
Na	mg/L	93.32	15.99	97.29	227	95.305	**50**
Mg	mg/L	45.07	8.146	37.02	93.47	41.045	**30**
Ca	mg/L	73.1	41.54	44.46	67.6	58.780	**75**
K	mg/L	6.893	2.451	5.792	11.043	6.343	**100**
Al	ug/L	3.451	7.732	5.831	2.388	4.641	**200**
V	ug/L	14.031	2.045	7.968	12.457	11.000	
Fe	ug/L	6.232	25.77	8.66	4.197	7.446	**300**
Zn	ug/L	2.333	1.087	1.523	1.006	1.928	**5000**
As	ug/L	14.884	3.648	18.956	31.714	16.920	**10**
Se	ug/L	<0.8	<0.8	<0.8	<0.8	<0.8	**40**
Mo	ug/L	4.223	1.026	4.100	7.743	4.162	**10**
Ba	ug/L	57.963	31.144	38.205	50.240	48.084	**700**
Th	ug/L	0.030	0.020	0.013	0.014	0.022	
U	ug/L	3.499	0.984	3.074	3.880	3.287	**15**

In all sampling points, the concentration of the cadmium and selenium in river waters was less than 0.02 μg/L and 0.8 μg/L respectively. Measured concentrations of the dissolved metal elements (except for As, Mg, and Na) in investigated water samples from Aras River and

Shirarkh Canal are under the limit values presented in World Health Organization (WHO) guideline. Results of the analysis showed that all metal concentrations (dissolved) were below the WHO guideline limit value in Arpachay River water. Determined radionuclide (U and Th) concentrations in all investigated samples were also below suggested values of the WHO guideline.

Pollution by heavy metals is considered to be a serious problem due to their toxicity and their ability to accumulate in the biota. For heavy metal contamination assessment in water resources, several methods were developed. The heavy metal pollution index (HPI) represents the total water quality with respect to heavy metals and based on the weighted arithmetic quality mean method. The pollution indexes are used to estimate the pollution of the water. Generally, heavy metal pollution index (HPI), heavy metal evaluation index (HEI) and degree of contamination (Cd) are used to evaluate water for drinking as well as irrigation purposes [13]. HPI is a method that rates the aggregate influence of individual heavy metal on the overall quality of water. The Heavy Metal Pollution Index (HPI) and the sub-index of each parameter (Qi) are calculated using the following correlations (Eq. 1) and (Eq.2) [18].

$$HPI=\frac{\sum_{i=1}^{n} WiQi}{\sum_{i=1}^{n} Wi} \quad (1)$$

$$Q_i = \sum_{i=1}^{n} \frac{|Mi-Ii|}{Si-Ii} * 100 \quad (2)$$

where Q_i is the sub-index of the ***i***th parameter; W_i is the unit weight of the ***i***th parameter; ***n*** is the number of parameters; M_i is the monitored value of the heavy metal of ***i***th parameter; I_i is the ideal value of ***i***th parameter; S_i is the standard value of the ***i***th parameter. The computational method for calculating HPIs are given in Table 2 - Table 4 for waters taken from the Aras River and Shirarkh Canal.

Table 2. HPI calculations for the Arpachay River water

Heavy metals	**S_i**	**I_i**	**W_i**	**M_i**	**Q_i**	**$W_i * Q_i$**
As	50	10	0.02	3.648	15.8800	0.3176
Cr	50	10	0.02	0.517	23.7075	0.4742
Mn	100	50	0.01	13.51	72.9800	0.7298
Fe	1000	100	0.001	25.77	8.2478	0.0082
Ni	70	5	0.0143	0.912	6.2892	0.0899
Cu	1000	50	0.001	0.814	5.1775	0.0052
Se	40	10	0.025	0.8	30.6667	0.7667
Al	200	10	0.005	7.732	1.1937	0.0060
Pb	50	10	0.02	0.035	24.9125	0.4983
U	50	15	0.02	0.984	40.0452	0.8009
Zn	3000	1000	0.00033	1.087	49.9457	0.0165
Ba	700	100	0.00143	31.144	11.4760	0.0164
ΣW_i=0.138						ΣW_iQ_i=3.7296
Heavy metal Pollution Index (HPI) = 3.7296/0.138 = 27.03						

Table 3. HPI calculations for the Aras River waters

Heavy metals	Si	Ii	Wi	Mi	Qi	Wi∗Qi
As	50	10	0.02	16.92	17.3000	0.346
Cr	50	10	0.02	0.338	24.1550	0.4831
Mn	100	50	0.01	1.339	97.3220	0.9732
Fe	1000	100	0.001	7.446	10.2838	0.0103
Ni	70	5	0.0143	2.864	3.2862	0.0470
Cu	2000	50	0.001	1.623	2.4809	0.0025
Se	40	10	0.025	0.8	30.6667	0.7667
Al	200	10	0.005	4.641	2.8205	0.0141
Pb	50	10	0.02	0.066	24.8350	0.4967
U	50	15	0.02	3.287	33.4657	0.6693
Zn	3000	1000	0.00033	1.928	49.9036	0.0165
Ba	700	100	0.00143	48.084	8.6527	0.0124
			ΣW_i=0.138			ΣW_iQ_i=3.838
Heavy metal Pollution Index (HPI) = 3.838/0.138 = 27.81						

Similarly the HPI values for Shirarkh Canal water sample were calculated and the results were given in Table 4.

Table 4. HPI calculations for the Shirarkh Canal water

Heavy metals	S_i	I_i	W_i	M_i	Q_i	W_i*Q_i
As	50	10	0.02	31.714	54.285	1.0857
Cr	50	10	0.02	0.176	24.560	0.4912
Mn	100	50	0.01	1.544	96.912	0.9691
Fe	1000	100	0.001	4.197	10.645	0.0106
Ni	70	5	0.0143	2.111	4.445	0.0636
Cu	2000	50	0.001	1.072	2.509	0.0025
Se	40	10	0.025	0.8	30.667	0.7667
Al	200	10	0.005	2.388	4.006	0.0200
Pb	50	10	0.02	0.04	24.900	0.4980
U	50	15	0.02	3.88	31.771	0.6354
Zn	3000	1000	0.00033	1.006	49.950	0.0165
Ba	700	100	0.00143	50.24	8.293	0.0119
			ΣW_i=0.138			ΣW_iQ_i=4.571
Heavy metal Pollution Index (HPI) = 4.571/0.138 = 33.12.						

Generally, the critical heavy metal pollution index HPI value is taken to be 100 [14]. Low heavy metal pollution (HPI < 100), heavy metal pollution on the threshold risk (HPI = 100) and high heavy metal pollution (critical pollution index) (HPI > 100). If the samples have HPI values greater than 100, water is not potable. Heavy metal pollution index can be classified into three categories [15] like low (<19), medium (19–38) and high (>38). Calculated HPI values in our study classified the water samples as mediumly polluted by heavy metals.

As concentration (Table 1) ranged within 3.648–31.714 μg/L exceeding limit value presented in WHO guideline of 10 μg/L in water samples taken from the Aras River and Shirarkh Canal. Drinking water quality is critical for human health and quality of life. As occurs in an environment in four oxidation states: arsenate (+5), arsenide (+3), elemental arsenic (0), and arsine (−3). Arsenide is the most abundant As species, while arsenate is stable in oxygenated aquatic environments. In natural water, As is found mainly in inorganic forms, that are considered much more toxic than the organic one. Due to its toxicity, As is ranked on the first position in the list of priority substances compiled by the United States Department of Health and Human Services, Public Health Service [16, 17], and a has low regulatory limit (10 μg/L) in drinking water [18]. Arsenic presence in the groundwater is the result of the weathering of rocks and sediments. Drinking of arsenic-contaminated water causes poisoning to the blood, central nervous system, lung, and skin cancer, breathing problems, vomiting and nausea. Its presence in Third World countries is becoming hazardous. The countries that are suffering from the problems of arsenic are India, Bangladesh, Taiwan, China, Brazil, Chile, South Korea, Thailand, and Indonesia. Arsenic is a geogenic problem worldwide but anthropogenic sources, such as the processing of metals and manufacture of pesticides and their byproducts, are contributing equally to the levels of arsenic in the environment. High As contents may cause adverse health effects on humans: dermal and cardiovascular diseases, skin and bladder cancer, diabetes mellitus, and so forth [19, 20].

The natural and anthropogenic occurrence of As in drinking water has been accepted as a major public health issue. The risk of cancer is estimated quantitatively, while the noncancer risk is estimated by using safety factors. The noncancer risks hazard quotient (HQ) for As in water samples were determined. The carcinogenic risk (CR) was calculated using the reference dose (RfD) and cancer slope factor (CSF) [21]. To calculate HQ, for each water sample, measured concentration values of As were used.

$$HG = ADD/RfD \tag{3}$$

where ADD (mg /kg/day) represents the average daily dose by ingestion and RfD is the reference dose for ingestion RfD = 0.300 μg/ kg/ day.

Average daily doses (ADD) for deterministic risk assessment were calculated according to the following equation

$$ADD = CIR*ED*EF/BW*AT \tag{4}$$

where C is the available concentration of As in water (μg/L), IR is the ingestion rate (2 L/day), EF is the exposure frequency (365 days/year), ED is the exposure duration (30 years), BW is the body weight (70 kg), and AT is the averaging time (30 years) [21–23]. The CR was determined using the average daily dose (ADD) and cancer slope factor (CSF). CSF is a measure of chemical potency and is particular to different pollutants. The carcinogenic risk increases linearly as the chemical dose rises. CSF is based on real studies that reflect health effects from carcinogenic pollutants at specific levels [23]. The acceptable or tolerable range is between 10^{-6} and 10^{-4} according to the US EPA [21]. CRs were calculated based on the concentration of As in each water sample investigated in this study [21]. US EPA database was used for CSF units [24].

$$CR = ADD*CSF \quad (5)$$

To evaluate the total potential non-carcinogenic risk, hazard quotients (HQ) were calculated. HQ > 1 suggests a potential risk for humans or the necessity for a supplementary study. Table 6 presents ADD, HQ, and CR values for the ingestion pathways of As in river and canal waters for each investigated sampling point.

Table 6: Hazard quotient for As content in water in each sampling point

Sampling point	ADD	HQ	CR
Arpachay River	1.042×10^{-4}	0.34733	1.563×10^{-4}
Aras River – Sederek	4.252×10^{-4}	1.4173	6.378×10^{-4}
Aras River – Babek	5.416×10^{-4}	1.8053	8.124×10^{-4}
Shirarkh Canal	9.061×10^{-4}	3.020	13.59×10^{-4}

The carcinogenic risk of As through ingestion exceeded the tolerable risk of $1x10^{-4}$ indicating that the usage of these river waters as a drinking water for a long period could raise the probability of cancer. Calculated HQ values are bigger than 1 in Aras River and Shirarkh Canal waters which means using these waters for drinking purposes raises the potential risk for human health. However, the present approach to evaluate the risks to human health has many uncertainties, such as variations in As concentrations, availability in time and location and variations in exposure conditions. Therefore, further investigations are required to evaluate As concentration and presence in more points from the area and to corroborate the evaluated risks with the epidemiological data.

4. Conclusion

Dissolved As concentrations in water samples were in the range of 3.648–31.714 μg/L, thus exceeding limit value presented by WHO guideline (10 μg/L) in analyzed water samples taken from the Aras River and Shirarkh Canal. Only in Arpachay River water As concentration was below the guideline limit value.

Calculated HPI values in this research classified investigated water samples as mediumly polluted with heavy metals. The carcinogenic risk of As through ingestion exceeded the tolerable risk of $1x10^{-4}$ (Table 6) indicating that the usage of these river waters for drinking purposes over a long period could raise the probability of cancer. Calculated HQ values are bigger than 1 in Aras River and Shirarkh Canal waters which means using these waters for drinking purposes raises the potential risk for human health. Therefore, further investigations are required to evaluate the As concentration and presence in more points from the area and to corroborate the evaluated risks with the epidemiological data.

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ОЦЕНКА ЗАГРЯЗНЕНИЯ ТЯЖЕЛЫМИ МЕТАЛЛАМИ И РАДИОНУКЛИДАМИ В ОБРАЗЦАХ ВОДЬ, ВЗЯТОГО ИЗ НАХЧЫВАНСКОЙ ОБЛАСТИ АЗЕРБАЙДЖАНА

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Резюме: Это исследование было проведено для определения основных катионов (Ca, K, Mg и Na), тяжелых металлов (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, V и Zn) и радионуклидов (U, Th) в проб водь, взятых из четырех разных точек отбора проб в Нахчыванской Автономной Республике. Элементные концентрации определяли с помощью масс-спектрометрической системы с индуктивно-связанной плазмой. Измеренные концентрации растворенных металлических элементов (за исключением As, Mg и Na) в исследованных пробах воды из реки Арас и канала Ширарх были ниже предельных значений, представленных в руководящих принципах Всемирной организации здравоохранения (ВОЗ). Результаты анализа показали, что все измеренные концентрации металлов (растворенных) в исследуемых пробах воды реки Арпачай также были ниже предельного значения, установленного ВОЗ. Определенные концентрации радионуклидов (U и Th) во всех исследованных образцах также были ниже рекомендуемых значений руководящих принципов ВОЗ. Индекс загрязнения тяжелыми металлами (HPI), риск неракового заболевания (HQ) и риск рака (CR) были рассчитаны для исследованных проб воды. Рассчитанные значения HPI в этом исследовании классифицировали исследованные пробы воды как средне загрязненные тяжелыми металлами. Канцерогенный риск As при проглатывании превысил допустимый риск 1x10-4, что указывает на то, что использование этих речных вод в качестве питьевой воды в течение длительного периода может повысить вероятность возникновения рака. Рассчитанные значения HQ превышают 1 в водах реки Арас и канала Ширарх, которое означает, что использование этих вод в питьевых целях повышает потенциальный риск для здоровья человека.

Ключевые слова: Азербайджан, река Арас, тяжелые металлы, радионуклиды, загрязнение, риск рака

AZƏRBAYCANIN NAXÇIVAN BÖLGƏSİNDƏN GÖTÜRÜLƏN SU NÜMUNƏLƏRİNDƏ AĞIR METALLAR VƏ RADİONUKLİDLƏRLƏ ÇİRKLƏNMƏNİN QİYMƏTLƏNDİRİLMƏSİ

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Xülasə: Bu tədqiqat işi Naxçıvan Muxtar Respublikası ərazisindən götürülən su nümunələrində əsas kationların (Ca, K, Mg və Na), ağır metalların (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, V və Zn) və radionuklidlərin (U və Th) miqdarını müəyyənləşdirmək məqsədilə həyata keçirilmişdir. Element konsentrasiyaları induktiv əlaqəli plazma kütlə spektrometr (ICP-MS) cihazı vasitəsilə müəyyənləşdirilmişdir. Araz Çayı bə Şirarx Kanalından götürülərək tədqiq olunan su nümunələrində ölçülən element (As,Mg və Na istisna olmaqla) konsentrasiyaları Dünya Sağlamlıq Təşkilatının (DST) təlimatında təqdim olunan limit qiymətlərindən aşağıdır. Analizin nəticələri onu göstərir ki, Arpaçay Çayının suyunda da həllolmuş bütün metalların konsentrasiyaları DST-nin təlimatında verilən limit qiymətlərindən aşağıdır. Həmçinin, analiz olunan bütün nümunələrdə radionuklidlərin (U və Th) konsentrasiyası üçün də DST tərəfindən təqdim olunan həddən kiçik qiymətlər müşahidə olunur. Tədqiq olunan su nümunələri üçün ağır metalla çirklənmə indeksi (AÇİ), xərçəngə tutulmamaq riski (XTR) və xərçəng riski (XR) hesablanmışdır. Bu tədqiqatda hesablanan AÇİ qiymətləri, analiz olunan su nümunələrini ağır metallarla orta dərəcədə çirklənmiş olaraq təsnif etmişdir. As-in xərçəng riski, qidalanma ilə qəbul üçün icazə verilən risk qiymətini ($1x10^{-4}$) aşmışdır ki, bu da suyun uzun müddət ərzində içilməsinin xərçəng ehtimalını artıra biləcəyini göstərir. Araz Çayı və Şirarx Kanalı sularında XTR > 1 olması bu suların içmə məqsədilə istifadəsinin insan sağlamlığına potensial risk yaratması deməkdir.

Açar sözlər: Azərbaycan, Araz Çayı, ağır metallar, radionuklidlər, çirklənmə, xərçəng riski