UDC 543.3 ICP-MS DETERMINATION OF TRACE METALS IN SEAWATER SAMPLES TAKEN FROM THE AZERBAIJANI SECTOR OF THE CASPIAN SEA

M.M.Ahmadov, K.S.Safarova, F.Y.Humbatov, V.S.Balayev, B.A.Suleymanov

Institute of Radiation Problems of NAS Azerbaijan AZ 1143, Baku, F/Agayev str., 9; e-mail: <u>hfamil@mail.ru</u>

This study was conducted to investigate heavy metal contents (Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Pb, Cu, Ni, V and Zn) in the seawater of the Caspian Sea taken from 19 different sites of the Azerbaijani territory. Agilent 7700x Series ICP-MS was applied to analyze the seawater. It revealed that average concentrations of elements such as Al, Co, Fe, Cu, Cr, Mn and Pb in the surface water proved to comparatively higher than those at depths of 100 m. There was a little variation for nickel and vanadium. In contrast, concentrations of As and Ba in samples taken from 100 m, were higher than those in surface samples.

Keywords: trace metal, cations, polyatomic ions concentration, ICP-MS applied.

INTRODUCTION

The Caspian Sea is the largest lake on our planet. It is bigger than the Great American lakes and Lake Victoria in Africa by surface area. However, it is unique not only because of its size. As distinct from other lakes, the water of the Caspian is not fresh, but brackish. Each liter of Caspian water contains 10-13 grams of salt making this water unsuitable for drinking or irrigation. However, the comparison of the Caspian water to oceanic water shows that it contains three times less salt than that of the World Ocean. Physically, the Caspian Sea is one extremes. Its salinity varies greatly. of Morphologically, the Sea is divided into three parts, the northern shallow part (average depth 6 m), the middle section with an average depth of 190 m (maximum depth 788 m) and the southern Caspian with a maximum depth of 1025 m. The Caspian Sea occupies a deep depression on the boundary between Asia and Europe with a water level at present 27 m below sea level. It is approximately 1200 km long with a maximum breadth of 466 km, contains 79,000 km3 of water, and has a total coastline of more than 7000 km. The Caspian is fed by five major rivers or river groups: in the north the Volga (80% of total inflow) and the Ural (5%); in the

west the Terek, Sulak and Samur (4-5%) and the Kura (7-8%); and, in the south, the small mountain rivers from the Iranian Alborz range (4-5%). Azerbaijan has more than 800 km of coastline along the Caspian Sea and almost the entire country is a part of the Sea's catchment area. The littoral states of the Caspian Sea are the Republic of Azerbaijan, the Islamic Republic of Iran, the Republic of Kazakhstan, the Russian Federation and Turkmenistan [1-3].

Environmental problems of the Caspian Sea are multiple and various in their origin. On the one hand, they are caused by the commercial use of the sea; on the other hand, human activity impacts coastal areas, including inflows from rivers into the Caspian. As the Caspian is an inland water body, anthropogenic impacts on catchment area (about 3.5 million km2) accumulate here. Anthropogenic impact on the Caspian ecosystem occurs concurrently with various natural endogenous and exogenous processes. It is primarily sea level changes, periodical seismic activity, surges and retreats, mud volcanoes and neo-tectonics. Special features of the Caspian include constant alterations of its area, volume, and configuration of the coastline and water column structure.

Anthropogenic activity, as well as a natural impact, can have a chronic (long-term) or acute (short-term) effect. The sources of pollution are industrial, agricultural and accidental discharges and sewage. The main sources of pollution into the Caspian Sea have generally been considered to be offshore oil production and land-based sources, notably the Volga River. The presence of oil in the Caspian Sea region has been known since ancient times and the oil reserves were amongst the first to be exploited in the world. Baku was a centre for oil production at the turn of the 20th century and developed further development during the era of the Soviet Union. Exploration and exploitation, especially of offshore reserves, has blossomed since the break up of the Soviet Union. Public opinion polls in the region have highlighted concerns about the oil and gas industry as a source of pollution to the environment of the Caspian Sea. Oil production areas cover vast areas of the coastal zone, particularly along the south coast of the Apsheron peninsula in Azerbaijan. Population growth and industrial development in the Caspian region have generated an immense pollution problem. Ten million people live adjacent to the Caspian Sea and 60 million more live in the Volga River watershed. The World Bank estimates that one million cubic meters of untreated industrial wastewater is discharged into the Caspian annually. Soviet oil extraction left behind polluted soil and water, rusty equipment, and well fires that burned for years. Meanwhile, the five countries surrounding the sea are rushing to exploit still untapped oil deposits.

Since the Caspian is an enclosed body of water, it has limited carrying capacity compared to larger and more open bodies of water. Waste products discharged into the Caspian are of biogeochemical nature and accumulated in the Sea for years. In the former Soviet Union, water and sediment quality measurements were taken on a regular basis and with good coverage. In Azerbaijan, the situation is reported to be changing, and monitoring surveys rose in number. Over this same period the flux of pollutants into the Caspian has changed, with a drastic reduction in industrial and agricultural activity in Turkmenistan, Kazakhstan, the Russian Federation and Azerbaijan [4-5].

The present study was conducted to investigate the heavy metal contents (Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Pb, Cu, Ni, Zn) of seawater the Caspian sea were collected from 19 different sites in Azerbaijan region as shown in Table1.

Station No	Easting	Northing	Station No	Easting	Northing
St.1	411348	4458000	St.11	520428	4438465
St.2	411348	4448000	St.12	519131	4434107
St.3	411341	4437826	St.13	524875	4440258
St.4	422743	4434115	St.14	529128	4433186
St.5	434459	4433772	St.15	538557	4431453
St.6	446087	4439695	St.16	546276	4426972
St.7	472503	4461630	St.17	411348	4408000
St.8	506110	4456694	St.18	411348	4378000
St.9	506927	4443481	St.19	393000	4350000
St.10	515426	4443978			

Table 1. The geographical coordinates of sampling stations

It is know the analysis of ultra trace elements in sea water samples is one of the most difficult analytical tasks in the field of environmental monitoring, as extremely low detection limits for elements buried in a highly saline matrix is required. The use of Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for direct sea water analysis is currently limited by spectral and non-spectral interferences caused by the sea water matrix. Although the technique of ICP-MS is very powerful by virtue of its sensitivity and selectivity, one of the operational limitations is that of the total amount of dissolved solids which may be introduced into the system. This limitation comes about in part because samples may deposit condensate on the sampler cones over a prolonged analytical run causing signal changes and hence degrading the signal stability. The effect of such higher levels of dissolved salts may also be a reason of polyatomic interferences on key target analytic elements. Table 2 indicates the extent of these interfering ion species.

One particular example of this effect is in the determination of trace elements in heavier environmental samples such as seawater and borehole waters. In particular, the effect of oxide / hydroxide adduct ions of alkali and alkaline earth elements, common constituents in such natural samples, can effectively mask manifestations of most transition elements [7-11]. Pre-concentration techniques such as solvent extraction, ion exchange and carrier precipitation have been extensively studied with reference to sea water analysis [12, 13].

Table 2. Major interferences of alkali and alkaline earth adduct ions on transition metal isotopes in
a typical seawater matrix.

		* *	seawater matrix.		
Isotope	Cl	Ca	Na	K	Ar
V ⁵¹	ClO				
Cr ⁵² Cr ⁵³	ClOH				ArC
Cr ⁵³	Cl ³⁷ O				
Mn ⁵⁵ Fe ⁵⁶			Na ₂ O	K ³⁹ O	ArNH
Fe ⁵⁶		CaO		КОН	ArO
Fe ⁵⁷		CaOH			ArOH
Ni ⁵⁸		Ca ⁴² O			
Co ⁵⁹		Ca ⁴³ O,			
		Ca ⁴² OH			
Ni ⁶⁰		Ca ⁴⁴ O,			
		Ca ⁴³ OH			
Cu ⁶³			NaAr		
Zn ⁶⁴		Ca ⁴⁵ O			
Cu ⁶⁵		Ca ⁴⁵ OH			
Zn ⁶⁷	ClO ₂				
As ⁷⁵	ArCl				

To avoid these problems the Agilent 7700x Series ICP-MS are applied to analyze seawater. The Agilent 7700x ICP-MS combines the simplicity of a single collision cell mode (helium mode) for polyatomic interference removal with the superior matrix tolerance of its unique High Matrix Introduction (HMI) system. In order to reduce salt deposition in the nebulizer the humidifier is used. The humidifier humidifies the carrier gas and reduces salt deposition in the nebulizer. Third generation Octopole Reaction System (ORS3) cell technology provides higher sensitivity and more effective interference removal than ever before in complex, high matrix samples, eliminating the need for reactive cell gases in routine analysis. Helium mode on the ORS3 is so effective that interference correction equations can also be eliminated. These two factors redefine ease of use in ICP-MS, removing two of the most common causes of errors in multielement analysis of complex samples.

MATERIALS AND METHODS

Water samples, collected from surface and bottom seawater layers with a 12-liter PVC Niskin sampler are equipped with a Tefloncoated spring, suspended from stainless steel hydro wire and allowed to flush for 5 min before closing in acid cleaned polythene containers, packed and transported in ice-box for subsequent analysis. Surface samples were collected from sites with depths not exceeding 5 meters, whereas bottom samples were collected from 9 sites with a depth of 100 meter.

All plastic-ware sample bottles, pipette tips, filtration unit and flasks were soaked in $10\% \text{ v/v} \text{ HNO}_3$ for 24 h and rinsed with ultra pure water before being used. Milli-Q ultra pure water (resistivity 18.2 M Ω cm, pH (5.5–6.5) was used throughout, and all laboratory operations. The acid concentration of the samples is adjusted to approximate at 1% (v/v) nitric acid solution by adding an appropriate volume of nitric acid in the laboratory.

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). Agilent's new High Matrix Introduction (HMI) accessory for the 7700x and 7700s ICP-MS systems permits the direct analysis of samples containing percent level dissolved solids including neat seawater without prior dilution. The HMI accessory uses a combination of highly optimized

robust plasma conditions and a technique termed "aerosol dilution". The net effect is a reduction in the amount of sample and solvent reaching the plasma and MS interface of up to 30x compared to a standard system. This reduction leads to higher plasma temperature, reduced suppression, improved stability and significantly lowers oxides.

The samples were analyzed for Ba, Al, As, Cr, Fe, Mn, Ni, Cu, Cd and Pb using an Agilent model 7700x inductively coupled plasma-mass spectrometry. Multi-element calibration working standards solutions were prepared by appropriate dilution of 10 mg /L multi-element stock standard solutions - Part # 8500-6940 in 5% HNO₃ in /1%HNO₃ correspondingly. The blank and calibration solutions were measured under optimized conditions. The calibration curve was automatically plotted by the instrument. Linear correlation coefficients (r) in all calibration curves were better than 0.9995. Instrument drift and matrix effects during measurement were corrected by using the internal standards include Sc, Ge, Rh, In, Tb and Bi were prepared by appropriate dilution from stock ICP-MS Internal Standard Mix Part# 5188-6525 and added online at the time of analysis using a second channel of the peristaltic pump. For quality control purposes, duplicate samples, matrixspike sample were analyzed. 7700x ICP-MS Operating Condition used for He mode

RF Power: 1600 W S/C Temp: 2 deg C Plasma Gas flow: 15 L/min Discriminator: 4.5 mV Carrier Gas flow: 0.6 L/min Pulse HV: 945 V Nebulizer Pump: 0.1rps Analog HV: 1682 V Smpl Depth: 8 mm He Gas flow: 5 mL/min Dilution Gas flow: 0.4 L/min.

RESULTS AND DISCUSSION

The values and ranges of element concentrations in the water samples taken from the surface-5 m depth are presented in Table 3. The concentrations of the analyzed elements in Caspian sea surface water were as following: Al: ranged between 2.756—11.155 ug/L, average concentration-5.817 ug/L which their maximum has been measured in station 6, As: ranged between 1.315-1.658 ug/L, average concentration-1.524 ug/L its maximum is present in station 7, Ba: ranged between 10.276-20.927 ug/L, average concentration-15.114 ug/L, with the maximum is shown is station 6, Co: ranged between 0.022-0.047 ug/L, average concentration-0.037 ug/L, maximum is present in station 3, Fe: ranged between 3.742-20.029 ug/L, average concentration-8.366 ug/L which their maximum has been measured in station 11, Mn: ranged between 0.236-1.714 ug/L, average concentration-0.810 ug/L, its maximum is present in station 5. Ni: ranged between 0.788-1.003 ug/L, average concentration-0.893ug/L, which their maximum has been measured in station 3, Cu: rangede between 0.685-4.121 ug/L, average concentration-1.01 ug/L, its maximum is present in station 6, Cr: ranged between 0.146-0.298 ug/L, average concentration-0.236 ug/L, its maximum is present in station 3, Cd: ranged between <0.01-0.028 ug/L, average concentration-0.012, its maximum is present in station 7, Pb: ranged between 0.037-2.287 ug/L, average concentration-0.246 ug/L, with the maximum is shown is station 11, V: ranged between 0.853–0.997 ug/L, average concentration-0.923 ug/L and its maximum is present in station 3 and Zn: ranged between 0.507-4.710 ug/L, average concentration-2.001 ug/L with their maximum having been measured in station 7 respectively (Table 3).

Table 3. Concentrations of analyzed elements in surface water (5 m deep)

	Al	V	Cr	Mn	Fe	Со	Ni	Cu	Zn	As	Cd	Ba	Pb
	ug/L	ug/L	ug/L										
St1	8.222	0.959	0.240	1.524	12.75	0.045	0.911	1.010	1.139	1.601	< 0.02	19.24	0.139
St2	6.078	0.896	0.235	1.400	8.206	0.039	0.917	0.872	1.281	1.654	< 0.02	20.20	0.091
St3	7.648	0.997	0.298	1.484	11.71	0.047	1.003	1.040	1.529	1.619	< 0.02	20.07	0.171
St4	8.278	0.896	0.265	1.583	8.969	0.039	0.914	1.082	2.283	1.652	< 0.02	20.57	0.100
St5	9.131	0.970	0.213	1.714	11.50	0.046	0.909	0.840	1.909	1.615	< 0.02	20.63	0.037
St6	11.16	0.926	0.220	1.414	11.63	0.042	0.945	4.121	3.856	1.650	< 0.02	20.93	0.383
St7	7.852	0.953	0.175	1.271	12.36	0.042	0.864	0.770	4.710	1.658	0.028	16.78	0.349
St8	6.203	0.949	0.248	0.903	5.308	0.037	0.909	0.734	1.997	1.539	< 0.02	12.65	0.122
St9	4.814	0.867	0.147	0.433	5.063	0.037	0.908	0.731	2.974	1.541	< 0.02	12.02	0.085
St10	4.073	0.938	0.272	0.428	12.58	0.039	0.937	0.823	1.699	1.480	< 0.02	13.50	0.189
St11	4.852	0.959	0.259	0.483	20.03	0.035	0.878	0.756	2.550	1.435	< 0.02	12.47	0.257
St12	3.531	0.880	0.146	0.333	4.163	0.037	0.947	0.834	2.174	1.560	< 0.02	12.10	0.133
St13	5.045	0.995	0.220	0.257	5.847	0.029	0.915	1.147	0.790	1.599	< 0.02	11.31	0.095
St14	3.203	0.871	0.246	0.323	3.742	0.022	0.806	0.756	0.954	1.326	< 0.02	10.28	0.070
St15	3.995	0.950	0.271	0.287	5.429	0.029	0.822	0.706	0.885	1.315	< 0.02	10.98	0.074
St16	2.756	0.876	0.283	0.236	5.687	0.035	0.830	0.685	0.507	1.369	< 0.02	10.83	0.084
St17	5.372	0.853	0.245	0.421	4.165	0.036	0.859	0.713	0.677	1.432	< 0.02	13.71	0.075
St18	4.748	0.895	0.247	0.442	5.899	0.037	0.898	0.870	3.637	1.418	< 0.02	14.85	0.118
St19	3.574	0.900	0.258	0.446	3.905	0.036	0.788	0.700	2.477	1.498	< 0.02	14.06	0.080
MIN	2.756	0.853	0.146	0.236	3.742	0.022	0.788	0.685	0.507	1.315	< 0.02	10.28	0.037
MAX	11.16	0.997	0.298	1.714	20.03	0.047	1.003	4.121	4.710	1.658	0.028	20.93	0.383
MEAN	5.045	0.926	0.246	0.446	5.899	0.037	0.909	0.823	1.909	1.541	< 0.02	13.71	0.100
STDEV	2.306	0.044	0.042	0.553	4.386	0.006	0.054	0.766	1.165	0.114		3.91	0.094

Concentrations of analyzed elements in Caspian seawater column, 100 m deep, were as follows: Al: ranged between 2.774—7.394 ug/L, average concentration-4.104 ug/L , As: ranged

between 1.645—2.719 ug/L, average concentration-2.445 ug/L, Ba: ranged between 20.931–41.437 ug/L, average concentration-30.345 ug/L , Co: ranged between 0.016—0.030

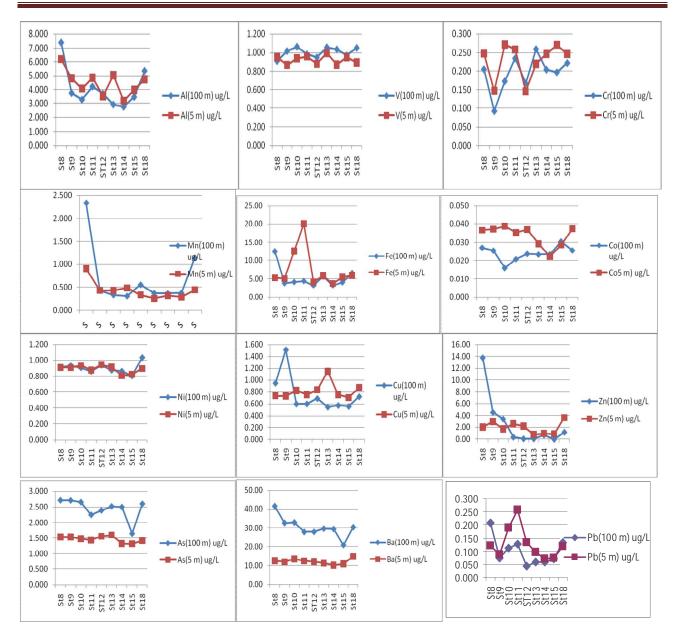
ug/L, average concentration-0.024 ug/L, Fe: ranged between 3.158—12.498 ug/L, average concentration-5.275 ug/L, Mn: ranged between 0.310—2.339 ug/L, average concentration-0.692 ug/L, Ni: ranged between 0.802–1.035 ug/L, average concentration- 0.903 ug/L, Cu: ranged between 0.548–1.514 ug/L, average concentration- 0.751 ug/L, Cr: ranged between 0.093–0.260 ug/L, average concentration-0.195 ug/L, Cd: ranged between 0.011–0.033 ug/L, average concentration-0.017, Pb: ranged between 0.045–0.206 ug/L, average concentration-0.074 ug/L, V: ranged between 0.911— 1.066 ug/L, average concentration-1.006 ug/L and Zn: ranged between 0.078—13.770 ug/L, average concentration-3.019 ug/L respectively (Table 4). Maximum concentrations in water column depth 100 m for Al, Mn, Fe, Zn, As, Ba and Pb has been measured in station 8, for Cr and Cd is shown in station 13, for Cu in station 9, for V in station 11 and for Ni in station 18 respectively.

	Al	v	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Cd	Ba	Pb
	ug/L	ug/L	ug/L	ug/L	ug/L								
St8	7.394	0.911	0.206	2.339	12.50	0.027	0.914	0.950	13.770	2.719	< 0.02	41.44	0.206
St9	3.765	1.019	0.093	0.429	3.806	0.026	0.935	1.514	4.566	2.715	< 0.02	32.60	0.074
St10	3.291	1.066	0.173	0.334	4.152	0.016	0.906	0.600	3.407	2.659	< 0.02	32.88	0.110
St11	4.228	0.984	0.235	0.310	4.405	0.021	0.858	0.601	0.362	2.250	0.026	27.87	0.128
St12	3.709	0.950	0.167	0.555	3.158	0.024	0.939	0.692	0.079	2.391	< 0.02	27.93	0.045
St13	2.938	1.061	0.260	0.371	5.653	0.023	0.874	0.548	0.078	2.521	0.033	29.64	0.059
St14	2.774	1.038	0.204	0.366	3.163	0.023	0.863	0.575	0.705	2.502	< 0.02	29.44	0.060
St15	3.482	0.973	0.197	0.376	4.062	0.030	0.802	0.558	< 0.03	1.645	< 0.02	20.93	0.071
St18	5.358	1.054	0.222	1.144	6.582	0.026	1.035	0.722	1.181	2.605	< 0.02	30.38	0.133
MIN	2.774	0.911	0.093	0.310	3.158	0.016	0.802	0.548	0.078	1.645	< 0.02	20.93	0.045
MAX	7.394	1.066	0.260	2.339	12.50	0.030	1.035	1.514	13.770	2.719	0.033	41.44	0.206
MEAN	4.104	1.006	0.195	0.692	5.275	0.024	0.903	0.751	3.019	2.445	0.030	30.34	0.098
STDEV	1.452	0.055	0.048	0.670	2.929	0.004	0.066	0.312	4.648	0.337		5.44	0.051

Table 4. Concentrations of analyzed elements in Caspian seawater column, 100 m deep

Average concentrations of elements as Al, Co, Fe, Cu, Cr, Mn and Pb in surface water were comparatively higher than those in deep water at 100 m. They slightly varied in nickel concentrations, with average concentration of - 0.893 ug/L in surface water and 0.903 ug/L at 100m. Slightly higher vanadium average concentrations were found in the samples taken at 100m with – 1.006 ug/L and - 0.923 ug/L in

surface samples. In contrast, the concentrations of As, Ba in samples taken from 100m, were significantly higher than those in surface samples. In the surface water from the site of 11, concentrations of Pb and Fe were drastically higher than those in other sites. A similar behavior was observed for concentrations of Mn and Zn in site 8 in the sample taken from 100 m deep, as shown in Fiq.1.



Fiqure 1. Spatial distribution of average concentration of heavy metals in seawater taken from sites at depths of 5 m and 100 m respectively.

The research study is the maiden attempt to have covered Baku Gulf surveyed areas of the Caspian Sea. Traces of metal levels identified in the course of the cruise are the first reported values of heavy metals in the seawater. It is now well know that trace metals are present in seawater and sediment in various chemical forms. Identification and speciation of heavy metal traces in the seawater, as well as sediments discovered are of great significance for their interactions with suspended matters, sediments and their uptake by aquatic organisms to be an area of great interest in today's aquatic metal chemistry. Data for distribution of trace metals between the water column, sediment and suspended particulate material are important and necessary for further studies.

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XƏZƏR DƏNİZİNİN AZƏRBAYCAN SEKTORUNDAN GÖTÜRÜLMÜŞ SU NÜMUNƏLƏRİNDƏ METAL İZLƏRİNİN ICP-MS-LƏ TƏYİNİ

M.M. Əhmədov, K.S.Səfərova, F.Y.Hümbətov, V.S.Balayev, B.A.Süleymanov

Təqdim olunan işdə Xəzər dənizinin Azərbaycan sektorunun 19 müxtəlif nöqtəsindən götürülmüş su nümunələrinin tərkibində ağır metalların (Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Pb, Cu, Ni, Zn) miqdarı tədqiq edilmişdir. Dəniz suyu, Agilent firmasının 7700x seriyalı İCP-MS ilə analiz edilmişdir. Göstərilmişdir ki, Al, Co, Fe, Cu, Cr, Mn və Pb elementlərinin suyun səthindəki orta konsentrasiyaları 100 m dərinlikdəki orta koncentrasiyalarından nisbətən yüksəkdir. Nikel və vanadium konsentrasiyalarında isə kiçik dəyişmələr müşahidə edilir. Bunların əksinə As və Ba-un 100 m dərinlikdəki orta konsentrasiyaları səthdəki konsentrasiyalarından kifayət qədər böyükdür. **Açar sözlər:** ağır metallar, kationlar, çoxatomlu ionlar, qatılıq.

ИСП-МС ОПРЕДЕЛЕНИЕ СЛЕДОВ МЕТАЛЛОВ В ОБРАЗЦАХ МОРСКОЙ ВОДЫ, ВЗЯТЫХ ИЗ АЗЕРБАЙДЖАНСКОГО СЕКТОРА КАСПИЙСКОГО МОРЯ

М.М.Ахмедов, К.С.Сафарова, Ф.Ю.Гумбатов, В.С.Балаев, Б.А.Сулейманов

Настоящее исследование было проведено с целью изучения содержания тяжелых металлов (Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Pb, Cu, Ni, Zn, V) в образцах морской воды, взятых с 19 разных точек Азербайджанского сектора Каспийского моря. Для анализа морской воды был применен масс-спектрометр с индуктивно-связанной плазмой (ICP-MS) фирмы Agilent серии 7700х. Было показано, что средние концентрации элементов Al, Co, Fe, Cu, Cr, Mn и Pb в поверхностных водах сравнительно выше, чем в образцах, взятых с глубины 100 метров, а концентрации Ni и V при этом отличаются незначительно. В отличие от вышеуказанных элементов, концентрации As и Ba в образцах, взятых с глубины 100 м, были выше, чем в образцах, взятых с поверхности.

Ключевые слова: тяжелые металлы, катионы, многоатомные ионы, концентрация.

Поступила в редакцию 21.01.2014.