

MATHEMATICAL ELIMINATION OF SPECTRAL INTERFERENCES IN NATURAL WATERS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

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Abstract- The main characteristics of an inductively coupled plasma mass spectrometer are investigated. It has been shown that the device has several significant limitations due to the complex emission spectra of many elements and the associated numerous spectral interferences. It was revealed that it is possible to correct the spectral interference using the mathematical method, as well as eliminate them by changing the setting in the mass spectrometer with inductively coupled plasma. Optimal conditions are considered that provide a significant reduction or elimination of spectral interference using the device tuning.

Keywords: Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Spectral Interference, Isobar Overlays, Analyte.

1. INTRODUCTION

In recent years, mass spectrometry with inductively coupled plasma (ICP-MS) has taken a leading position among instrumental methods of elemental analysis in various fields of chemistry, geology, biology and medicine. The ICP-MS method is based on the use argon inductively coupled plasma as a source of ions and a mass spectrometer for the separation and subsequent detection of these ions.

Inductively coupled argon plasma (ICP) has been used since the mid 60th of the last century as a highly efficient source for atomic emission spectroscopy [1-3]. This source is still successfully used for multi-element atomic emission analysis (ICP-AES), although it has a number of significant limitations due to the complex emission spectra of many elements and the associated numerous spectral interferences.

Spectral interference refers to a change in the intensity of the signals of the ions being determined by superimposing on them the signals of other ions having the same value of m/z . Spectral interference is divided into:

- Isobaric overlays caused by overlapping signals of isotope of different elements ions with the same nominal mass, for example, ^{87}Rb on ^{87}Sr ;
- Superposition of doubly charged ions, for example $^{138}\text{Ba}^{2+}$ on $^{69}\text{Ga}^{+}$;
- Polyatomic overlays caused by the formation in the plasma of ions containing more than one atom of an element.

Isobaric interference and the imposition of doubly charged ions rarely pose a problem in the quantitative analysis of stable elements in routine objects. In most cases, such overlays can be avoided by choosing alternative isotopes for measurements. Isobaric interference causes difficulties in geological analysis when determining the ratios of $^{87}\text{Rb}/^{87}\text{Sr}$ and some others.

Polyatomic interference, which is the most problematic, arises from ion-molecular reactions in a plasma discharge. Obviously, depending on the composition and content of elements in the sample, the composition and ratio of the prevalence of polyatomic ions changes. For example, when analyzing aqueous solutions in plasma, the most common ions are those containing an oxygen atom - oxides (MeO^{+} , ArO^{+}), argon - argides (Ar^{2+} , MeAr^{+}) and their variations with the inclusion of other atoms, for example, hydrogen - hydroxides (HnO^{+} , ArOH^{+} , MeOH^{+}), hydrides (ArH^{+} , MeH^{+}), etc. When analyzing samples with a high carbon content, a problem arises with the determination of chromium from the most common ^{52}Cr isotope due to the superposition of the $^{40}\text{Ar}^{12}\text{C}^{+}$ signal. A similar situation arises in the determination of arsenic and vanadium against a background of high chloride content - superposition of $^{35}\text{Cl}^{16}\text{O}^{+}$ on $^{51}\text{V}^{+}$, $^{40}\text{Ar}^{35}\text{Cl}^{+}$, and $^{40}\text{Ca}^{35}\text{Cl}^{+}$ on $^{75}\text{As}^{+}$, etc. Some of the most common interferences are listed in Table 1. The main methods for controlling spectral interference are described below.

The aim of this work is to calculate the equation for correcting spectral interference by a mathematical method and eliminate them by increasing the resolution in a mass spectrometer with inductively coupled plasma.

2. EXPERIMENTAL PART

In this work we used the following high purity substances: deionized water obtained on a Mill-Q - Integral (Millipore) installation; concentrated hydrogen peroxide of the "emprove" quality (Merck); acids, nitric (obtained on a DuoPur device (Milestone)), hydrofluoric, and perchloric of the "suprapure" quality (Merck); and also, multielement and single element certified solutions.

Multielement standards were as follows: Tuning Solution (Agilent Technologies) containing 1 µg/L Li, Mg, Co, Y, Ce, and Tl; Multielement Calibration Standard 1 (Agilent Technologies) containing 10 mg/L Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and Th; and ICP-MS 68B A (High-Purity Standards) containing 100 mg/L Li, Be, Mg, Al, P, K, Ca, Sc, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Y, Cd, In, Ba, La, Ce, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb, Re, Pb, Bi, Th, and U. Single element standards were as follows: In solutions (Agilent Technologies) with the concentration 10 mg/L Ba, La, Ce, Pr, Nd, Tb, and Lu solutions with the concentration 1000 mg/L (High Purity Standards). Also, we used a Araz river standard sample of natural waters with the following certified concentration of elements from La to Lu. Solutions of lower concentrations were prepared by a consecutive weight method using dilutions with a 5% HNO₃ solution. The maximum dilution in one cycle was 100 fold.

Samples of suspended and dissolved fractions of river waters (Table 1) (differentiation criterion is particle size of 0.45 µm) selected at the territory of the Araz river were test materials.

The first step in the preparation of water samples was the separation of the suspended fraction from the dissolved one. This was done by filtering a sample through a cellulose acetate membrane filter 47 mm in diameter with a pore size of 0.5 µm (Sartorius). The first portion of the filtrate up to 40 mL in volume was rejected to wash the filter and filtering system. The other portion of a required volume was preserved by adding concentrated nitric acid to a 4% (v/v) together with the introduction of an internal in standard by the weight method to the concentration 0.5 µg/L. Thus, we prepared the dissolved fraction to the analysis. Filtering and conservation were carried out no more than three hours after the sampling in the place of sampling or in a laboratory.

The ash was decomposed to damp salts in two steps. At the first step, a mixture of concentrated fluorid, nitric, and perchloric acids, 200, 600, and 200 µL in volume, respectively, was used. At the second step, we used a mixture of concentrated nitric acid and hydrogen peroxide, 200 and 20 µL in volume, respectively. At the beginning of each step, the components of one or another mixture were added to ashes (the first step) or damp salts (the second step) under weak short-time heating. Then the mixture obtained was allowed to stand under normal conditions for 18-24 h and slowly evaporated to damp salts. The preparation of the suspended part was finished with the dissolution of damp salts in 5% HNO₃ with transferring to a test tube by the weight method and the

addition of an internal in standard to the concentration 1 µg/L by the weight method. The final volume was 10 mL.

We used an Agilent Technologies ICP-MS-7700 mass spectrometer with inductively coupled plasma in the mode of the introduction of low mineralized samples without collision gas helium [4]. The operation characteristics of the device were as follows: applied power 1440 W; distance from the inductor coil to the first cone 8.0 mm.

For the primary and secondary selection of the substance plasma, we used nickel cones with orifice diameters of 1 and 0.4 mm, respectively, in the interface zone. The adjustment of the mass spectrometer was performed using a tuning solution with the maximum sensitivity for Ce containing an acceptable level of oxides. Element concentrations were calculated by the method of an external calibration graph using a Multielement Calibration Standard 1 or an ICP-MS-68BA standard.

Table 1 shows isobaric and polyatomic influences observed when determining the composition of water. As can be seen from Table 1, despite the presence of several isotopes in almost every isobar, practically none of them is free from interference.

Table 1. Expected isobaric and polyatomic influences

Elements	Mass a.m.u..	Interference
Ni	60	²⁴ Mg ³⁶ Ar ⁺ , ⁴⁴ Ca ¹⁶ O ⁺ , ⁴³ Ca ¹⁶ OH ⁺ , ²⁵
Cu	63	²³ Na ⁴⁰ Ar ⁺ , ²⁵ Mg ³⁸ Ar ⁺ , ²⁷ Al ³⁶ Ar ⁺ , ⁴⁷ Ti ¹⁶ O ⁺
Zn	66	²⁶ Mg ⁴⁰ Ar ⁺ , ²⁸ Si ³⁸ Ar ⁺ , ³⁰ Si ³⁶ Ar ⁺ , ⁵⁰ Ti ¹⁶ O ⁺ , ⁵⁰ Cr ¹⁶
Rb	85	⁴⁵ Sc ⁴⁰ Ar ⁺ , ⁴⁷ Ti ³⁸ Ar ⁺ , ⁴⁹ Ti ³⁶ Ar ⁺ , ⁶⁹ Ga ¹⁶ O ⁺
Sr	88	⁴⁸ Ti ⁴⁰ Ar ⁺ , ⁴⁸ Ca ⁴⁰ Ar ⁺ , ⁵⁰ Ti ³⁸ Ar ⁺ , ⁵⁰ V ³⁸ Ar ⁺
Zr	90	⁵⁰ Ti ⁴⁰ Ar ⁺ , ⁵⁰ V ⁴⁰ Ar ⁺ , ⁵⁰ Cr ⁴⁰ Ar ⁺ , ⁵² Cr ³⁸ Ar ⁺ , ⁵⁴ Fe
Cs	133	⁹³ Nb ⁴⁰ Ar ⁺ , ⁹⁵ Mo ³⁸ Ar ⁺ , ⁹⁷ Mo ³⁶ Ar ⁺
La	139	⁹⁹ Ru ⁴⁰ Ar ⁺ , ¹⁰¹ Ru ³⁸ Ar ⁺ , ¹⁰³ Rh ³⁶ Ar ⁺
Ce	140	¹⁰⁰ Mo ⁴⁰ Ar ⁺ , ¹⁰⁰ Ru ⁴⁰ Ar ⁺ , ¹⁰⁰ Rd ³⁸ Ar ⁺ , ¹⁰⁰ Ru ³⁸ Ar ⁺
W	182	¹⁴² Nd ⁴⁰ Ar ⁺ , ¹⁴² Ce ⁴⁰ Ar ⁺ , ¹⁴⁴ Nd ³⁸ Ar ⁺ , ¹⁴⁴ Sm ³⁸ Ar ⁺
Pb	208	¹⁶⁸ Er ⁴⁰ Ar ⁺ , ¹⁶⁸ Yb ⁴⁰ Ar ⁺
Th	232	¹⁹² Os ⁴⁰ Ar ⁺ , ¹⁹² Pt ⁴⁰ Ar ⁺ , ¹⁹⁴ Pt ³⁸ Ar ⁺
U	238	¹⁹⁸ Hg ⁴⁰ Ar ⁺ , ²⁰⁰ Hg ³⁸ Ar ⁺ , ²⁰² Hg ³⁶ Ar ⁺ , ²⁰³ Tl ³⁵ Cl ⁺

The copper content in samples of well waters was within the norm. In the samples of arable soils the copper content exceeded the maximum permissible concentration (MPC) 4 times (Table 2). Manganese falls into rivers mainly in the leaching of minerals and ores containing manganese. The increased content of molybdenum was also observed in the bottom sediments of the river in the area. Bottom sediments are capable of depositing metals in the form of various compounds. Heavy metals in bottom sediments are in the process of constant migration, at which their continuous exchange through the geochemical barrier system "water environment - bottom sediments" [5]. The content of manganese in some water samples of the Araz River and in the waters of the well exceeded the MPC by 3-5 times

Table 2. The content of heavy metals in the waters of the Araz River and wells

Compound	Mass	Conc	Units	Count
Al	27	13.991	mg/l	146
Cr	52	0.394	mg/l	116
Mn	55	1700.185	mg/l	113 044
Fe	56	149.973	mg/l	27 979
Co	59	1.162	mg/l	313
Ni	60	8.906	mg/l	709
Zn	66	54.816	mg/l	2 897
As	75	6.852	mg/l	56
Mo	95	124989.673	mg/l	40 306
Cd	111	0.332	mg/l	776
Sn	118	341.687	mg/l	443
Pb	208	<0.000	mg/l	6 355
Th	232	134.064	mg/l	160
U	238	4.291	mg/l	200 516

The peculiarities of ICP are that very peculiar processes take place in it, during which a compound is formed which can never be achieved under ordinary conditions, i.e., ions that cannot be combined under ordinary conditions and which will not be stable in the atmosphere can be combined. In plasma, they are stable, and this is one of the problems.

Long before measurements, the sample is dissolved in 2% nitric (or hydrochloric, depending on the chemical composition of the sample) acid, where the substance is separated into atoms of the elements of the substance, then the solution is pumped to the atomizer, in which it is converted into aerosol using argon. The aerosol enters the argon plasma, where under the influence of high temperature the inductive plasma is ionized. Thus, the processes of dissociation and ionization of elements almost in ICP-MS are completely separated from each other, and at the exit from the plasma the ions have some energy spread ($\Delta W < 10$ eV), not exceeding the ionization energies of atoms of various elements [4].

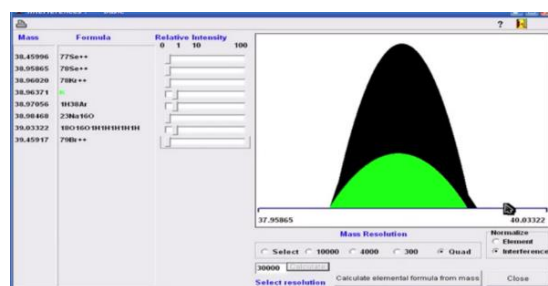
As an example, we can show the most common iron isotope Fe^{56} (green, and on the left are elements that can be superimposed on 56 mass with same mass) (Figure 1a).

Where "++" is a highly charged ions, which are easy to fight, as they are embedded in the device settings and using the settings you can reduce their number. In Figure 1, we can see 2 interference: 1) ArO , which is difficult to get rid of and 2) CaO if a lot of Ca is present in the solution. Others, e.g., NeO , are not strong interference.

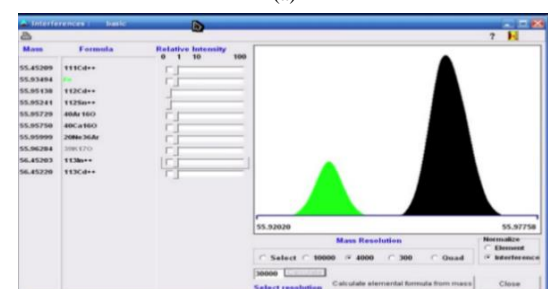
At a low resolution, it is very difficult to separate Fe from ArO , then by adjusting the ion optics of the mass spectrometer (carried out using the Autotune program from the ChemStation package) we increase the resolution of the device and, on average, the resolution is a separate signal for Fe and a separate signal for ArO (Figure 2b).

Such a procedure can also be done for element K with the most common 39 isotope. In Figure 2a, we see ArH with a large amount. If there is a small amount of K in the solution, then it will be very difficult to separate it from ArH even if the instrument is set to medium resolution and only at high resolution can a narrow peak be obtained (Figure 2b).

Even at absolutely identical settings of the mass spectrometer, a certain drift of the relative intensities of oxide and hydroxide ions was possible with time. Therefore, an important question is about the use of the found values of $I(E^{16}O)/I(E)$ and $I(E^{16}O^+H)/I(E)$ for the elimination of spectral interferences in routine analyses. The correction coefficients between the experimental and working values were the ratios $I(^{156})/I(^{140})$ and $I(^{157})/I(^{140})$; these were determined in the direct instrumental analysis by a tuning solution. Thus, if the ratios $I(Ce^{16}O)/I(Ce)$ or $I(Ce^{16}O^+H)/I(Ce)$ with time changed by several times, the ratios for the other elements $I(E^{16}O)/I(E)$ or $I(E^{16}O^+H)/I(E)$, correspondingly, also changed. The deviations from these monotonous dependences were not taken into account because of their insignificance.

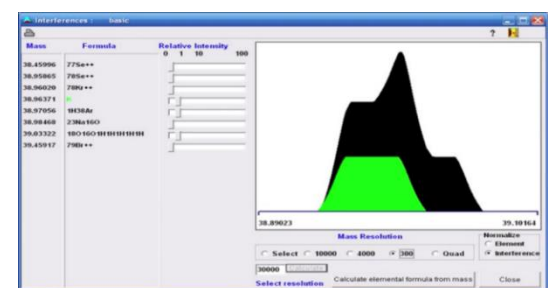


(a)

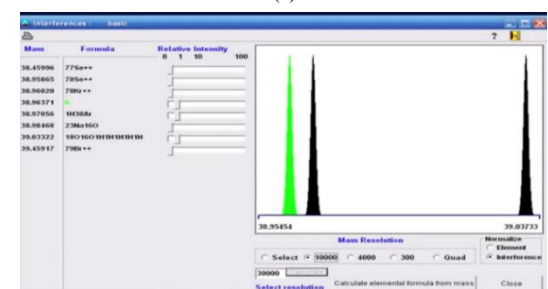


(b)

Figure 1. Spectral interference for element Fe^{56} , (a) at standard setting; (b) with a stable setting



(a)



(b)

Figure 2. Spectral interference for element K^{39} , (a) at standard setting; (b) with a stable setting

The choice of Ce for the determination of correction coefficients is explained by two reasons: (1) a rather high concentration of this element in the majority of analytical solutions, which enhances the memory effect of auxiliary solutions to a minimum extent and (2) a high natural relative abundance of ^{140}Ce and the high levels of formation of Ce oxide and hydroxide interferences, which in sum increased the accuracy of the determination of the absolute intensities of interferences in instrumental analysis. The described method of the transformation of the experimental values of relative intensities of interferences into the working ones is similar to that proposed in the work [6]. Thus, we can write the following equations of external corrections:

$$\begin{aligned} I(\text{Th}^{232})_K &= \\ &= I^{(232)} - 4.93 \times 10^{-4} \times \\ &\times (I^{(156)}/I^{(140)})_T I(\text{Ba}^{135}) \end{aligned} \quad (1)$$

$$\begin{aligned} I(\text{U}^{238})_K &= \\ &= I^{(238)} - 1.88 \times 10^{-7} (I^{(156)}/I^{(140)})_T I(\text{Ba}^{135}) - \\ &- 0.0152 (I^{(157)}/I^{(140)})_T I(\text{Ba}^{135}) \end{aligned} \quad (2)$$

$$\begin{aligned} I(\text{Mo}^{95})_K &= \\ &= I^{(95)} - (I^{(156)}/I^{(140)})_T (0.0523 I(\text{Ba}^{135}) + \\ &+ 7.97 \times 10^{-7} I(\text{Ce}^{140}) \times \\ &\times (I^{(157)}/I^{(140)})_T (1.13 I(\text{Ba}^{135}) + \\ &+ 0.00209 I(\text{Ce}^{140})) \end{aligned} \quad (3)$$

$$\begin{aligned} I(\text{Mn}^{55})_K &= \\ &= I^{(55)} - (I^{(156)}/I^{(140)})_T (0.00162 I(\text{La}^{139}) + \\ &+ 3.81 \times 10^{-4} I(\text{Ce}^{140}) + 1.15 I(\text{Pr}^{141}) - \\ &- (I^{(157)}/I^{(140)})_T (0.0211 I(\text{Ba}^{135}) + \\ &+ 5.37 \times 10^{-4} I(\text{La}^{139}) + 0.997 I(\text{Ce}^{140})) \end{aligned} \quad (4)$$

$$\begin{aligned} I(\text{Lu}^{175})_K &= \\ &= (I^{(175)} I^{(156)}/I^{(140)})_T 0.0015 (I(\text{Gd}^{157})_K + \\ &+ 0.639 I(\text{Tb}^{159})_K 6.67 \times 10^{-7} I(\text{Dy}^{163})_K) - \\ &- (I^{(157)}/I^{(140)})_T (1.01 I(\text{Gd}^{157})_K + 6.62 \times 10^{-4} I(\text{Dy}^{163})_K) \end{aligned} \quad (5)$$

where, K is index of external correction and T is index of tuning solution. The numerical coefficients in Equations (1)-(5) multiplied by one and the same ratio $((I^{(156)}/I^{(140)})_T$ or $(I^{(157)}/I^{(140)})_T$) and by one or another intensity were determined similarly.

Fundamental studies of the nature of the formation of polyatomic ions in the ICP contribute to the continuous improvement of the instrumental support of the method. New developments are aimed at creating conditions under which the formation of polyatomic ions cannot proceed or the highly efficient removal of interfering ions from the ion beam occurs.

The addition of molecular or inert gases, such as nitrogen, hydrogen, oxygen, methane, propane, xenon, and krypton to plasma-forming argon, has shown in practice the possibility of reducing a number of polyatomic interference and improving the detection limits of elements. Among the main reasons explaining the decrease in the intensity of some polyatomic ions from the group CCl^+ , ArC^+ , ArO^+ , Ar^{2+} , Cl^{2+} , N^{2+} , ArCl^+ and ArN^+ upon the introduction of oxygen, nitrogen or propane into argon, a change in the thermochemical properties of plasma is indicated. For example, the

addition of nitrogen significantly increased the analytical signal of elements with low ionization potentials and effective oxide formation [7].

The "cold plasma" mode provides a significant decrease in the temperature of the plasma source to 2500-3500 K by reducing the power of the high-frequency generator (600-800 W) and increasing the speed of the spraying gas flow (more than 1.1 l/min). The use of such conditions suppresses the formation of argon-containing and some other ions with high ionization potentials, for example, ArH^+ , ArN^+ , ArO^+ and CO^+ , and allows the determination of 39K, 44Ca, 56Fe at a level of several ng/L (ppt) in conventional quadrupole mass spectrometers. low resolution, which is completely impossible in normal plasma conditions (6000-8000 K: 1100-1400 W: 0.8-1.0 l/min).

The speed and convenience of carrying out multielement analysis, the modern analytical level of sensitivity, accuracy and reproducibility have made the ICP-MS method in world practice an irreplaceable technique for determining trace levels of elements in scientific and industrial facilities. In this regard, the problem of spectral interference of polyatomic ions, which manifests itself in the analysis of most samples, is becoming increasingly important, since ignoring spectral overlaps or misinterpreting the analysis results can lead to a large disregarded systematic error in determinations.

The provided available information on the considered group of spectral interferences can allow analysts not only to successfully correct practical results and choose the optimal conditions for the analysis, but also to predict the manifestation of a particular group of spectral overlays for a particular research object.

3. CONCLUSIONS

Thus, over the years of development of ICP-MS, significant progress has been made in eliminating spectral overlays. In more common quadrupole mass spectrometers, reaction-collision cells are widely used, which in many cases are superior to high-resolution mass spectrometers in the efficiency of ion separation [5, 6]. The use of modern technologies for eliminating spectral interference leads to the fact that the limits of determination, even for elements that are problematic for ICP-MS (Fe, Se, Ca, K, Na, etc.), are mainly limited by the purity of the reagents.

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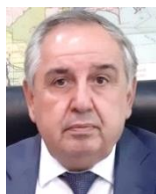
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BIOGRAPHIES



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Zulfugar Kamil Nurubeyli was born in Azerbaijan on May 6, 1976. He graduated as Master degree in Honors from Azerbaijan State Oil Academy, Baku, Azerbaijan in 2001. Since 2006 he received Candidate of Physics-Mathematical Sciences degree (Ph.D.).

He engaged in development and further improvement of analytical abilities of Mass-spectrometer. He has over 30 publications in Azerbaijan and abroad, also he is author and coauthor of several inventions and participated in several international conferences. He is the member of Institute of Engineering and Technology (IET), UK, and also member of Russian Mass-Spectrometry Society.



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