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Method of low tantalum amounts determination in niobium and its compounds by ICP-OES technique

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ABSTRACT

A method of determination of low amounts of tantalum in niobium and niobium compounds without its prior separation by means of inductively coupled plasma optical emission spectrometry (ICP-OES) has been worked out. The method involves dissolution of the analyzed samples of niobium as well as its various compounds (oxides, fluorides, chlorides, niobates(V)) in fluoride environments, precipitation of sparingly soluble niobic(tantalic) acid ($Nb_2O_5(Ta_2O_5) \cdot xH_2O$), converting them into soluble complex compounds by means of oxalic acid with addition of hydrogen peroxide and finally analyzing directly obtained solutions by ICP-OES. This method permits determination of Ta in niobium at the level of 10^{-3}% with relatively good precision (≤8% RSD) and accuracy (recovery factor: 0.9–1.1). Relative differences in the results obtained by two independent methods (ICP-OES and ICP-MS) do not exceed 14%, and other elements present in niobium compounds (Ti, W, Zr, Hf, V, Mo, Fe, Cr) at the level of 10^{-2}% do not affect determination.

1. Introduction

Niobium and tantalum are elements of very similar chemical properties, which occur together in many minerals. Niobium and its alloys, intermetallic compounds and composites are widely used in many areas of modern technology. However, properties of niobium and its compounds strictly depend on their purity (including tantalum presence). The contents of the latter at the level >100 μg/g is especially undesirable in materials used in nuclear technology. Therefore, reliable and sensitive methods of tantalum determination in niobium and its compounds at low level are necessary to find tantalum contents as well as to evaluate the effectiveness of various methods of separation of tantalum from niobium.

A review of the methods of Ta determination in niobium and its compounds [1] indicates that inductively coupled plasma optical emission spectrometry (ICP-OES) is used mostly for the routine control of tantalum contents in niobium and its compounds. In this way Ta was determined in niobium metal [2,3], in high purity niobium metal [4–6], in niobium alloys [7–9], in ceramic materials containing niobium and uranium [10], in niobium carbide [11], and in high purity niobium(V) oxide [6]. In general, niobium samples were introduced into solution in fluoride media [2–11] and tantalum was determined directly in obtained solutions [2,5,7,8,10,11] or after preliminary separation of Ta from niobium matrix by means of ion exchange [3,4,6], extraction [6] or precipitation [9]. The lowest contents of Ta in the analyzed samples were rarely less than 50 μg/g (12 μg/g Ta in relation to Nb_2O_5 [6] and 22 μg/g in relation to Nb [4]), which occurred only in the case when preliminary separation of tantalum from niobium took place.

Different details of procedures and digestion conditions were used for various types of niobium compounds. However, there was no other universal method dedicated to determination of tantalum in the wide range of niobium materials, such as niobium metal, its oxide, fluoride, chloride as well as niobate(V). The aim of the present work is to establish a uniform procedure enabling determination of low amounts of Ta (<50 μg/g) in niobium as well as in various kinds of its compounds by means of the ICP-OES technique without its prior separation.

2. Experimental

2.1. Reagents

All chemicals used were exclusively of analytical reagent grade (p.a.), obtained from POCH S.A. (Poland), *i.e.* hydrofluoric acid (40%), nitric acid (65%), hydrochloric acid ULTRANAL (30%), hydrogen peroxide (30%), ammonia solution (25%) and oxalic acid dihydrate.

Calibration standards were prepared from commercially available ICP standard Ta solution (starting reagent 99.99% Ta;

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concentration 1000 μgTa/mL in 1% HNO_3 and 0.5% HF, ROTH) and niobium(V) oxide Specpure (Alfa Aesar).

Ultrapure water (class I according to ASTM, < 18 MΩ/cm) and argon of special purity (99.999%) were used.

2.1.1. *Types of niobium samples*

Metallic niobium 99.8% (Sigma Aldrich), niobium(V) fluoride 98.0% (Sigma Aldrich), niobium(V) chloride 99.80% (Acros Organics), sodium niobate technical grade, as well as various kinds of niobium(V) oxides of nominal purity: 99.99% (Acros Organics), and $>$ 99.9% (Schuchardt, Germany), $>$ 99.9% (Fluka AG, Switzerland), Specpure (POCH S.A., Poland), were used.

2.2. *Apparatus*

A simultaneous inductively coupled plasma optical emission spectrometer (iCAP MFC 6300 Duo, Thermo Fisher Scientific) was used in the study employing a high-resolution Echelle optical system and charge injection device (CID) array detector, with an extended spectral range of 166–847 nm and dual (lateral and axial) plasma view.

Appropriate solutions were introduced into the ICP plasma using a HF-resistant sample introduction system including a Teflon nebulizer, a Teflon spray chamber and a quartz torch with a ceramic injector tube (fluoride solutions prepared by standard methods), or a glass system designed for saline solution (5% TDS) nebulization (non-fluoride solutions prepared by Procedures 1 and 2).

The following ICP-OES operating conditions (excitation power and carrier gas flow rate) were used: 1150 W power for fluoride solutions, 1250 W power for solutions containing complexing salts (higher power values resulted in increased background signal) and the aerosol carrier gas flow rate $>$ 0.5 L/min Table 1.

2.2.1. *Procedures*

1) Dissolution of niobium samples

a) 1 ± 0.0002 g (as Nb_2O_5) of sample (Nb, NbF_5, Nb_2O_5, $NaNbO_3$) was placed in a platinum dish, 10 mL conc. HNO_3 was introduced, followed by adding HF (15–25 mL) in portions (during heating at 100 °C for about 30 min) until complete dissolution. The obtained solution was transferred into a PE beaker.

b) 2.033 ± 0.0002 g $NbCl_5$ was placed in a glass beaker and dissolved in 10 mL conc. HCl.

2) Precipitation of $Nb_2O_5(Ta_2O_5)\cdot xH_2O$

To both solutions obtained in procedures 1(a) and 1(b) aqueous ammonia solution (1+1, v/v) was added up to pH 9. Precipitated $Nb_2O_5(Ta_2O_5)\cdot xH_2O$ was filtered and washed with water.

3) Obtaining soluble complexes of Nb(V) (Ta(V))

a) $Nb_2O_5(Ta_2O_5)\cdot xH_2O$ was dissolved in 60 mL 3% $H_2C_2O_4$

b) $Nb_2O_5(Ta_2O_5)\cdot xH_2O$ was dissolved in 60 mL 3% $H_2C_2O_4$ and 40 mL 30% H_2O_2.

Dissolution of niobium samples precipitation of niobic(tantalic) acid and its conversion into soluble complexes were carried out in 1 day. Evaporation of solutions in order to concentrate them was avoided. Clear solutions were stored before analysis at 4 °C. Stock solutions (1%) were stable for ca. 1 week. In tantalum determination by ICP-OES matrix, solutions of maximum concentration of 0.2% (m/v in relation to Nb_2O_5) were used.

3. Results and discussion

3.1. *Effect of niobium matrix in the solution on conditions of tantalum excitation*

During determination of tantalum in niobium samples without its prior separation significant effect of niobium-matrix presence on the conditions of tantalum excitation may be expected. This effect has been established (Fig. 1).

As can be seen the increase of concentration of niobium matrix in the excited solution (M) reduces relative tantalum signal intensity ($I_{M+0.2}$–I_M) during excitation, *e.g.* in a 0.4% Nb_2O_5 solution it is almost twice lower than that in a solution without matrix.

Therefore in further tests Ta emission signals were measured directly with ICP using matrix-matched standards and simultaneous background correction was used to overcome any background shifts.

Table 1
ICP-OES spectrometer parameters used for analysis.

Operating parameter	Parameter value	
Type of niobium matrix	HF	$H_2C_2O_4$ $H_2C_2O_4$+H_2O_2
Excitation frequency (MHz)	27.12	
RF forward power (W)	1150	1250
Plasma view	Axial	
Detector	CID-86 chip	
Spectral range (nm)	166–847	
Spectral resolution	7 pm at 200 nm	
Torch	Duo EMT torch kit—HF	Duo EMT torch kit
Center tube	Center tube ceramic	
(HF resistant) 2 mm ID	Center tube 1.5 mm ID	
Spray chamber	Duo spray chamber (HF resistant)	Cyclonic spray chamber
Nebulizer	MiraMist nebulizer (HF resistant)	Seaspray nebulizer
Argon flow rate		
—Aerosol carrier gas flow (L/min)	0.45	0.40
Auxiliary gas flow (L/min)	0.50	1.00
Plasma gas flow (L/min)	12.00	12.00
—Coolant gas flow(L/min)	14.00	14.00
Analysis sample pumping rate (mL/min)	1.6	
Sample flush time (s)	30	40
Stabilization time (s)	5	
Integration time of emission lines:		
—Low (166–230 nm) (s)	30	
—High (230–847 nm) (s)	10	

All tantalum subarrays were checked for any necessary background corrections and spectral interferences. Niobium-matrix standard solutions prepared by suitable dilution of tantalum stock solution (1000 μgTa/mL, ROTH) and 1% niobium(V) solution (in relation to Nb_2O_5) were applied.

3.2. Selection of kind of niobium solutions for analysis and corresponding nebulization and exciting systems

Niobium and tantalum occur in soluble form in aqueous solutions of pH 0–14 exclusively as complexes, mainly fluoride, oxalate, tartrate and some others. So it is important to choose the proper kind of niobium complex solution, because it imposes further analytical limitations.

In the case of fluoride solutions the use of glass and quartz components in ICP-OES apparatus (spray chamber, nebulization system) should be avoided, but known procedure of binding HF excess by means of boric acid used in standard sample microwave digestion cannot be applied because it results in degradation of niobium and tantalum fluoride complexes. On the other hand, the use of ICP-OES components made of HF-resistant materials (*e.g.* Teflon, ceramic) is generally connected with worsening exciting conditions of generally very weakly excitable tantalum.

In the case of non-fluoride solutions, relatively high concentrations of complexing agents are necessary, which may substantially affect both the conditions of tantalum excitation and the operation of the apparatus itself.

Fig. 2 presents comparison of analytical signal profiles (Cts/s) obtained for tantalum in the concentration range of 0–1 μgTa/mL in aqueous solutions with no matrix (A), in niobium(V) solutions in the form of fluoride complexes (B) and oxalate complexes (C). It can be clearly seen that tantalum analytical signal for oxalate solutions introduced into plasma by means of a standard quartz sample introduction system is comparable to that of aqueous solutions containing no matrix. However, analytical signal of tantalum in fluoride complex solutions nebulized into plasma by means of an HF-resistant Teflon sample introduction system is several times weaker (B).

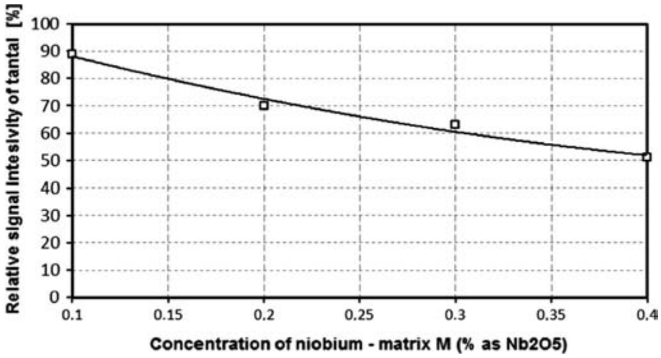

Fig. 1. The effect of niobium matrix concentration (M, % as Nb_2O_5) on relative tantalum signal intensity ($I_{M+0.2}-I_M$) at $\lambda=240.0$ nm. $I_M+_{0.2}$—signal intensity for matrix of a given concentration and the addition of 0.2 μgTa/mL. I_M–signal intensity for matrix of the same concentration without addition of Ta.

Unfortunately, during the analysis of numerous series of samples prepared in the form of oxalate complexes, an unfavorable intensity drift of the tantalum analytical line was observed as the effect of salt deposition on the ICP torch center tube and plasma quenching. Therefore, H_2O_2 was added in controlled amounts, which oxidized CO formed during oxalate decomposition and improved the solubility of niobium(V) and tantalum (V) species through the formation of their *peroxo*-complexes. Fig. 3 presents signal intensity changes of the tantalum analytical line ($\lambda_{Ta}=240$ nm) obtained during repeated excitation of standard niobium solutions (0.2% in relation to Nb_2O_5) containing 1 μgTa/mL prepared with and without H_2O_2 addition. As can be seen, tantalum analytical signal is stable only in the case of solutions containing H_2O_2.

For the established optimal procedure of niobium samples preparation (Procedures 1a, 1b, 2, 3b), the sensitivity of its several analytical lines was checked by determining the slope (b) of the regression line and its significance using Student-t test. The calculated value of $t_{b(\alpha,f)}$ being no less than the Student-t distribution critical value for $\alpha=0.05$ level of significance and $f=n-2$

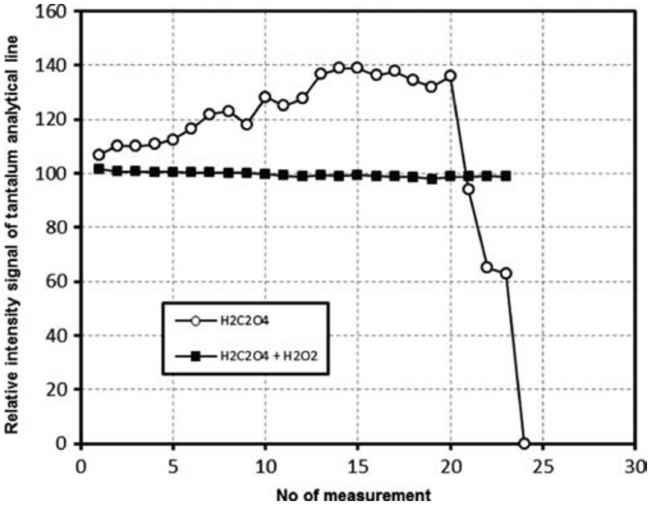

Fig. 3. Variations of relative* (in relation to a solution containing 1 μgTa/mL with no niobium matrix) signal of tantalum analytical line during repeated excitation of standard niobium solutions containing 1 μgTa/mL prepared with and without addition of H_2O_2.

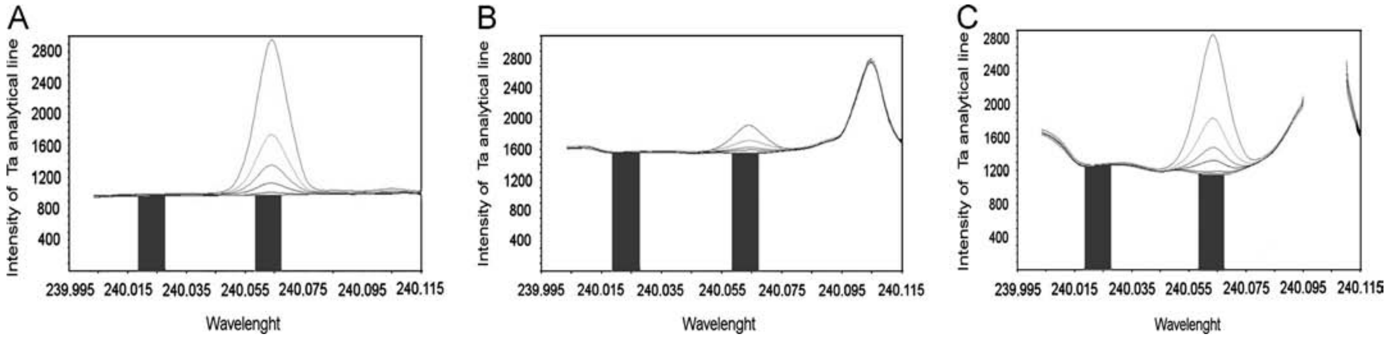

Fig. 2. Profiles of blank and standards for analytical line λ_{Ta} 240.0 nm. A—standards containing no matrix, B—standards with matrix (fluoride complexes of Nb(V)), and C—standards with matrix (oxalate complexes of Nb(V)).

Table 2
Sensitivity and detection limits of selected tantalum emission lines.

Tantalum emission line [nm]	Spectral niobium interferences	Matrix concentration [% as Nb_2O_5] [nm]	Statistical parameters of linear regression: $y=a+bx$					s_y [Cts/s]	IDL [µgTa/mL]	MDL in relation to Nb_2O_5 [µgTa/mL]
			v_m [%]	r^2	b [(Cts/s/ug/mL)Ta]	t_b	a [µgTa/g]			
226.230	226.152 226.213 226.322	0.2	8.5	0.9940	1582	$t_b > t_{crit.}$	4	52	0.1	50
263.558	263.251 263.471 263.798	0.2	0.8	0.9999	1263	$t_b > t_{crit}$	0.1	4.1	0.01	5
240.063	239.849 240.534	0.2	1.1	0.9999	1178	$t_b > t_{crit.}$	−0.06	5.2	0.02	10
228.916	228.635 228.887 229.568	0.2	2.6	0.9994	976	$t_b > t_{crit.}$	−0.3	9.8	0.03	20
268.517	268.247 268.371 268.639	0.2	3.8	0.9980	819	$t_b > t_{crit.}$	0.7	12	0.05	30
233.198	232.622 233.228 233.481	0.2	2.2	0.9996	632	$t_b > t_{crit.}$	−0.2	5.3	0.03	20

v_m—the calibration coefficient of variation (precision of calibration); r^2—square of correlation coefficient of the regression line;
b—slope of the regression line; t_b—statistics of Student's t-distribution of b; a—intercept, s_y—residual standard deviation, **IDL**—instrumental detection limit, and **MDL**—method detection limit

Table 3
Accuracy and precision of selected best sensitive tantalum emission lines.

Matrix concentration [% as Nb_2O_5]	Ta added [µg/g] in relation to Nb_2O_5	Ta determined in relation to Nb_2O_5 [µg/g]	
		Ta 240.063 nm	Ta 263.558 nm
0.2	**5**	5.3 ± 0.5 $n=6$	7.5 ± 2.6 $n=6$
	RSD, %	9.5	33.2
	Recovery, *R*%	**106**	**151**
	***F*-test**	$F_{eksp}=24.7 > F_{crit}(95\%, 5, 5)=7.15$	
0.2	**10**	9.2 ± 0.7 $n=6$	9.1 ± 1.9 $n=6$
	RSD, %	7.5	19.8
	Recovery *R*%	**92.4**	**90.6**
	***F*-test**	$F_{eksp}=6.61 < F_{crit}(95\%, 5, 5)=7.15$	
0.2	**15**	14.5 ± 0.6 $n=6$	14.7 ± 1.7 $n=6$
	RSD, %	4.3	10.9
	Recovery *R*%	**96.9**	**98.0**
	***F*-test**	$F_{eksp}=6.72 < F_{crit}(95\%, 5, 5)=7.15$	

Table 4
Testing of the recovery of tantalum for Nb_2O_5 samples.

Matrix concentration [% as Nb_2O_5]	Ta added in relation to Nb_2O_5 [µg/g]	Ta determined [µg/g] in relation to Nb_2O_5
		Ta 240.0 nm
0.2	**50**	53.8 ± 4.6 $n=6$
	RSD, %	8.1
	Recovery %	**108**
	(Dixon–Dean test-*Q*)	$Q_{exp}= 0.070 < Q_{crit}(95\%, 6)=0.625$
0.2	**250**	232 ± 6 $n=6$
	RSD, %	2.6
	Recovery %	**93.1**
	(Dixon–Dean test-*Q*)	$Q_{exp}=0.610 < Q_{crit}(95\%, 6)= 0.625$
0.2	**500**	477 ± 7 $n=6$
	RSD, %	1.4
	Recovery %	**95.3**
	(Dixon–Dean test-*Q*)	$Q_{exp}=0.610 < Q_{crit}(95\%, 6)$: 0.625

degrees of freedom, means that the $t_{b(\alpha,f)}$ is significant and the method is sensitive.

The highest sensitivity and good precision of calibration[1] ($<3\%$) may be observed for two emission lines: 263.558 nm and 240.063 nm. Taking into account relatively low instrumental detection limits IDL obtained for both of them (the ratio of the triple residual standard deviation[2] of the calibration curve to the slope value), they seem to be relatively the most useful for determination of tantalum traces in niobium samples. Under optimum conditions they should enable tantalum determination at the level of ≤1×10^{-3}% (in relation to Nb_2O_5), without the need to separate these elements. The results are presented in Table 2.

The accuracy and precision of tantalum determinations for the best sensitive emission lines presented in Table 2: 240.063 nm and 263.558 nm were verified by testing the recovery of the tantalum analyte using spiked high purity niobium(V) oxide (Alfa Aesar). Tantalum in an amount close to MDL was added to the niobium sample after its dissolution. As it can be seen, tantalum recovery and precision results were satisfactory only for 240.063 nm emission line, ranging from 0.9–1.1 recovery factor and ≤10% RSD, respectively (Table 3). This line was selected as the optimal for further studies.

The accuracy of the developed analytical method comprising preliminary solubilization of niobium samples (Procedures 1a, 1b), precipitation of niobic acid (Procedure 2) and dissolution in a mixture of oxalic acid and hydrogen peroxide (Procedure 3b) was

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[1] The calibration coefficient of variation (precision of calibration) is equal to the ratio of the residual standard deviation of the calibration curve[2] to the product of slope value and average standards concentration.

[2] The residual standard deviation (the standard error of fit) is equal to the square root of the sum of the squared differences divided by the degrees of freedom.

Table 5
Results of tantalum determination in $NbCl_5$ by means of the elaborated procedure.

Determination no.	Determined Ta content [%] in relation to $NbCl_5$
1	0.0022
2	0.0025
3	0.0028
4	0.0024
5	0.0022
Mean content [%]	**0.0024 ± 0.0003**
Reference content [%]	**0.0025**
Comparison of the reference and the mean values (*t*-Student test)	$t_{exp}=0.718 < t_{crit}(95\%, 4)=2.776$

Table 6
Testing of tantalum recovery for other kind of niobium samples.

Tested sample	Recovery of Ta (%) Ta 240.0 nm	
	Added Ta [μg/g in relation to Nb_2O_5]	Recovery of Ta [%]
$Nb_2O_5 > 99.9\%$, Schuchardt	100	99.0
Nb, 99.8%, Sigma Aldrich	100	95.4
$NbCl_5$, 98.9%, Acros Organics	100	95.1
NbF_5, 98%, Sigma Aldrich	100	102.4
$NaNbO_3$, technical gr.	100	94.5
Mean recovery of Ta (%)	**97.3 ± 4.2, RSD = 3.4%**	

Table 7
List of the results of tantalum determination in available niobium preparations, obtained by ICP-MS and ICP-OES.

Niobium preparation	ICP-MS ^{181}Ta	ICP-OES Ta 240.0 nm	Relative error [%]
	Tantalum contents [μg/g]		
Nb_2O_5, Schuchardt, $> 99.9\%$	10.5	11.5	**10**
Nb, Sigma Aldrich, 99.8**%**	140	160	**14**
Nb_2O_5, Fluka > 99.9**%**	590	670	**14**
$NaNbO_3$, technical gr.	655	722	**10**
Nb_2O_5, POCH, specpure	2160	2090	**3**

Table 8
Effect of some elements on tantalum determination by means of ICP-OES and ICP-MS

Added interfering elements (Ti, W, Zr, Hf, Mo, V, Fe, Cr) in relation to Nb_2O_5 [μg/g]	Added Ta in relation to Nb_2O_5 [μg/g]	ICP-MS ^{181}Ta	ICP-OES Ta 240.0 nm
		Recovery of Ta (%)	
100	100	93.8	94.5

verified by testing the recovery of the tantalum analyte (at several constant levels in the working range of tantalum content) using spiked (tantalum was added to the niobium sample before its dissolution) niobic samples of various purities, obtained from various manufacturers, as well as by comparing the results obtained with those produced by another independent method (ICP-MS).

The precision of this method was measured by relative standard deviation (% RSD) obtained through repeated analysis of the same samples.

The accuracy of tantalum determination by ICP-OES was proved at addition level 50, 250 and 500 μg/g Ta in relation to Nb_2O_5, using Acros Organic niobium(V) oxide (99.99%) for niobium matrix (0.2 % in relation to Nb_2O_5). The results are given in Table 4.

As can be seen, tantalum recovery in each case is satisfactory, ranging from 0.9 to 1.1. Similarly, the repeatability of the developed method, determined on the basis of deviations of measurement results obtained by the same analyst, for the same sample within a short time period, proved to be of acceptable precision under repeatability conditions (RSD≤8%).

The procedure developed was also verified during the determination of tantalum in niobium(V) chloride (Acros Organics, 99.8%), where tantalum content was guaranteed (but not certificated) to be 0.0025%. The mean value of analytical results for the tantalum emission line did not differ significantly (Student's *t*-test) from the guaranteed value (Table 5).

Recovery of tantalum at constant level of 100 μg/g (in relation to Nb_2O_5) for other niobic samples of lower purity is presented in Table 6.

As can be seen, the values of tantalum recovery are close to 100%, which proves that tantalum undergoes the same chemical transformations as niobium and is not lost during dissolution of niobium samples, precipitation of $Nb_2O_5 \cdot xH_2O$, and conversion thereof into soluble complexes by means of an $H_2C_2O_4$+H_2O_2 mixture.

Finally, the developed method of tantalum determination was verified by another independent method—inductively coupled plasma mass spectrometry (ICP-MS), which was considered a reference method. Niobium samples and tantalum standards were prepared according to Procedures 1a, 1b, 2, 3b, in the form of a 0.2% matrix solution (in relation to Nb_2O_5) in two series: spiked and unspiked with a known amount of tantalum (100 μg/g Ta in relation to Nb_2O_5). The determination was first carried out by means of ICP-OES, and then, upon 1000-fold dilution, by means of ICP-MS. The results of tantalum determination in various kinds of commercially available niobium samples are presented in Table 7. Relative differences between the results obtained by both independent methods for various materials do not exceed 14%.

3.3. *Effect of accompanying elements on tantalum determination*

Resistance of the developed method to the effect of some of the most often occurring elements in niobium preparations was evaluated during simultaneous determination of tantalum by means of two independent methods (ICP-OES and ICP-MS) in model mixtures of known content of these elements (100 μg/g of Ti, W, Zr, Hf, Mo, V, Fe—in relation to Nb_2O_5) and constant, comparable content of tantalum Ta (100 μg/g in relation to Nb_2O_5). The results are presented in Table 8. For both methods the mean values of tantalum recovery in these model solutions fit in the acceptable range of 0.9–1.1, which indicates the lack of potential coincidence with the elements under consideration.

4. Conclusions

A uniform analytical procedure of tantalum determination in various kinds of niobium preparations such as metallic niobium, niobium(V) oxide, niobium(V) chloride, niobium(V) fluoride and sodium niobate(V) without its prior separation has been proposed. It involves dissolution of the sample according to standard methods (in a mixture of HNO_3 and HF, or conc. HCl), precipitation of sparingly soluble niobic(tantalic) acids ($Nb_2O_5(Ta_2O_5) \cdot xH_2O$) from the obtained solution (by NH_3 aq. at pH 9) and converting the sample into soluble complexes (by means of an $H_2C_2O_4$+H_2O_2 mixture). The application of oxalate niobium complexes solutions and quartz components in ICP-OES apparatus gives better tantalum excitation and higher sensitivity of the method, necessary to determine low amounts of Ta. The obtained identical sample and standard solutions are directly analyzed by ICP-OES. The method permits determination of tantalum in niobium and its compounds at the level of 10^{-3}%, with relatively good precision (≤8% RSD) and accuracy (recovery of tantalum: 0.9–1.1). Relative differences in the results by the two independent methods (ICP-OES and ICP-MS) do not exceed 14%, and the presence (at comparable concentrations) of other elements occurring in niobium preparations (Ti, W, Zr, Hf, Mo, V, Fe, Cr) does not affect the determination (tantalum recovery at level 94% confirmed by the two methods mentioned).

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