

Application of Orange G as a Complexing Reagent in Spectrophotometric Determination of Osmium(IV)

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Formation of Os(IV)-Orange G (OG) complex was investigated. The following optimum conditions for the reaction were established: pH = 5.80 (0.20 mol L⁻¹ acetate buffer solution); heating time on a boiling water bath: 30 min; excess of reagent: 2-fold. Effective molar absorptivity ϵ at 540 nm was 1.1×10^4 L mol⁻¹ cm⁻¹. Stoichiometric ratio of the components of Os(IV)-OG compound was determined applying mole ratio and continuous variations methods. Linear range for Os(IV) determination was 0.01–7.70 $\mu\text{g mL}^{-1}$. Selectivity of spectrophotometric determination of Os(IV) using Orange G was investigated in the presence of platinum and other metal ions after elimination of the effect of interfering components using masking agents.

Badano kompleks Os(IV)-Oranz G. Ustalono optymalne warunki reakcji: pH = 5,80 (0,20 mol L⁻¹ roztwór buforu octanowego); czas ogrzewania na łaźni wodnej: 30 min; nadmiar odczynnika: 2-krotny. Efektywny współczynnik absorpcji molowej wynosił: $\epsilon = 1,1 \times 10^4$ L mol⁻¹ cm⁻¹ przy $\lambda = 540$ nm. Stechiometryczny stosunek składników kompleksu Os(IV)-OG wyznaczono przy użyciu stosunku molowego i metody ciągłych zmian. Liniowy zakres oznaczania Os(IV) mieścił się w granicach od 0,01 do 7,70 $\mu\text{g mL}^{-1}$. Selektywność spektrofotometrycznego oznaczania Os(IV) przy użyciu Oranżu G badano w obecności platyny i innych jonów metali, po usunięciu ich wpływu za pomocą czynników maskujących.

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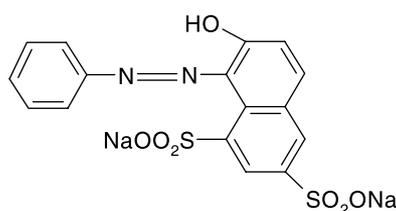
One of the most important tasks of analytical chemistry of platinum group elements (PGEs) is determination of osmium in various complex samples. In industrial and research laboratories, spectrophotometry still plays an important role in determination of PGEs, including osmium. Main advantages of spectrophotometric methods include rapidity of analysis and simplicity of determination of PGEs at low and high concentrations at affordable costs [1–3]. Recently, new application fields of spectrophotometry in the analysis of PGEs have appeared, *e.g.* numerous new materials based on PGE are produced by chemical, electronic, electrotechnical, and medical industry; usually these materials comprise few components and represent various contents of PGEs [1].

Osmium differs from other PGEs with respect to more pronounced redox properties and speciation in aqueous solutions. Therefore, a number of spectrophotometric reagents used for selective determination of osmium are much lower than for other noble metals. The measurement of osmium content in real samples is always complicated by the presence of one or a few PGEs, what considerably decreases selectivity of analysis. Also, many spectrophotometric methods utilizing organic reagents have a few drawbacks: complicated sample preparation, which requires previous separation of osmium (in the form of tetraoxide; or extraction of coloured osmium compounds with organic solvents); use of expensive, unavailable and rare reagents; unknown reaction mechanism between the metal and the reagent. It is significant that none of the known spectrophotometric techniques provide sufficiently accurate determination of osmium in the presence of all PGEs [1, 4–6]. It would be thus of interest to continue a search for new, simple, sensitive, selective and accessible spectrophotometric methods for determination of osmium.

Although azo reagents are widely used in analysis, spectrophotometry of osmium in the presence of these compounds has not been practically investigated. However, we have studied an interaction between osmium (IV) and diazo dye Congo Red (CR) and monoazo dyes Tropaeolin O (TpO) and Tropaeolin OOO-I (TpOOOI) [7–9]. It has been found that these reagents form coloured compounds with osmium in aqueous media. Moreover, sensitive spectrophotometric methods for determination of osmium in various samples in the presence of these azo reagents have been elaborated (for CR: pH = 3.5, C(NaCl) = 0.01 mol L⁻¹, Os:CR = 1:1, $\epsilon_{490} = 1.2 \times 10^4$ L mol⁻¹ cm⁻¹, limit of detection (LOD) = 0.05 μ g mL⁻¹; for TpO: pH = 5.2 C(CH₃COONa + CH₃COOH) = 0.2 mol L⁻¹, Os:TpO = 3:2, $\epsilon_{540} = 1.3 \times 10^3$ L mol⁻¹ cm⁻¹, LOD = 0.29 μ g mL⁻¹; for TpOOOI: pH = 8.0 C(Na₂B₄O₇) = 0.01 mol L⁻¹, Os:TpOOOI = 1:4, $\epsilon_{364} = 9.4 \times 10^3$ L mol⁻¹ cm⁻¹, LOD = 0.13 μ g mL⁻¹). This work continues former investigations and shows the results of a study on reaction between Os(IV) and acidic monoazo dye Orange G.

Chemical structure of Orange G (C.I. 16230; Acid Orange 10; 7-hydroxy-8-(phenylazo)-1,3-naphthalendisulfoacid sodium salt) is given in Scheme 1. OG is used

as a marker in microbiological analysis, pH-indicator [10], and food colorant. OG absorption maximum in aqueous solutions appears at 478 nm ($\epsilon_{\lambda} = 1.95 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$) [11, 12]. Orange G is known as a spectrophotometric reagent in drug analyses, particularly of sedative medicines, and is used for proteins determination in different samples, particularly in milk, plasma, and serum [13–17].



In this paper we present the results of spectrophotometric study on the interaction between Os(IV) ions and Orange G. Based on the obtained findings, a new direct spectrophotometric method for determination of Os(IV) has been developed and tested using synthetic and real samples.

EXPERIMENTAL

Reagents and apparatus

All aqueous solutions were prepared using distilled water.

Stock solution of Os(IV) (H_2OsCl_6) was prepared by dissolving the exact mass of OsO_4 (analytical grade; $\omega(\text{OsO}_4) = 99.9\%$) from a hermetically sealed glass ampoule (Corp. Aurat, Russia) in concentrated HCl, following the modified method [18]. The obtained osmium solution was stored for 1 month; according to reference [19], in concentrated HCl OsO_4 reduces to OsCl_6^{2-} rapidly under heating and slowly at the room temperature.

Due to possible osmium loss caused by OsO_4 volatility, standardization of the obtained stock solutions was carried out titrimetrically using KI as a reagent; the generated I_2 was titrated with $\text{Na}_2\text{S}_2\text{O}_3$. Identification of Os(IV) was carried out spectrophotometrically by comparing the recorded electronic absorption spectra with the literature data [20, 21]. It followed from the obtained spectra that in standard stock solutions Os(IV) exists in the form of OsCl_6^{2-} . Hence, the initial OsO_4 was quantitatively transformed to OsCl_6^{2-} .

Standard working solutions of Os(IV) were prepared by dissolving an aliquot of Os(IV) stock solution in *ca* $0.5\text{--}1 \text{ mol L}^{-1}$ HCl aqueous solution up to $\text{pH} < 1$. According to reference [1], OsCl_6^{2-} does not undergo hydrolysis under these conditions ($C_{\text{HCl}} > 0.5 \text{ mol L}^{-1}$, room temperature), and according to reference [22], nearly 93% of Os(IV) exist in the form of OsCl_6^{2-} in $0.1\text{--}3 \text{ mol L}^{-1}$ HCl solution.

Aqueous solutions of Orange G were prepared from commercially available OG compound (Shostkinsky Chemical Plant, Ukraine). UV–VIS measurements were performed with a UV–VIS scanning spectrophotometer (CARY.WIN – UV–VIS–50, Varian, USA) and a KFK-2 – UHL 4.2 photometer (Zagorsky mechanical-optical plant, Russia) using 1–5 cm cuvettes. All absorbance measurements were performed at *ca* 20°C . pH measurements were carried out with a pH-meter, model pH–150M (Gomelsky Plant of Measuring Devices, Belarus), equipped with a glass electrode. pH of each solution was adjusted using diluted HCl and NaOH solutions.

Procedure for Os(IV) determination

Firstly, 5.0 mL of 1 mol L⁻¹ acetate buffer (sodium acetate – acetic acid) and 6.0 mL of 7.54×10^{-4} mol L⁻¹ OG solutions were placed in a 25 mL volumetric flask. Then, a sample solution containing 0.01–7.70 $\mu\text{g mL}^{-1}$ Os(IV) in the final volume was added. pH was adjusted to 5.80 and distilled water was added. The obtained solutions were heated on a boiling water bath ($\sim 98^\circ\text{C}$) for 30 min. Then, the solutions were cooled to the room temperature of *ca* 20°C. After cooling, the absorbance was measured at 540 nm in 5.0 cm cuvettes against blank solution. Os(IV) concentration was calculated using calibration curve and standard addition methods.

RESULTS AND DISCUSSION

Absorption spectra

According to the experimental results, Os(IV) ions interact with Orange G in weakly acidic media and form pink-orange coloured compound. Maximum absorbance signal of a dye appeared at 479 nm; for Os(IV)–OG compound the absorbance band was lower and exhibited a slight bathochromic shift ($\lambda_{\text{max}} = 498 \text{ nm}$) and a well-defined absorbance shoulder at $\lambda < 525 \text{ nm}$ (Fig. 1). That is why further investigations were carried out at 540 nm, *i.e.* in the spectral range, where the maximum difference between the absorbance of the reagent and the reaction product was observed.

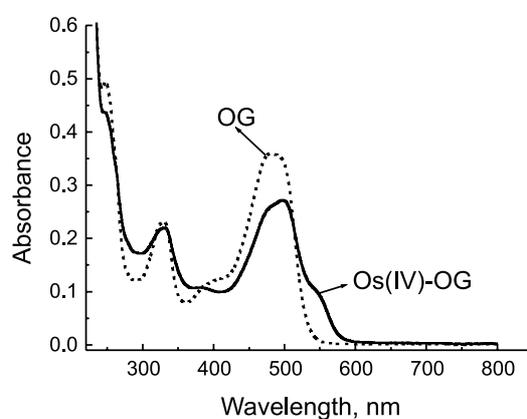


Figure 1. Absorption spectra of Orange G and osmium(IV)–OG compound; $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 1.51 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 1 \text{ cm}$

Effect of pH

In order to establish optimal reaction conditions, the investigations of the effect of pH on the magnitude of the analytical signal of Os(IV)–OG were carried out using sodium chloride and acetate buffer as background electrolytes (Fig. 2).

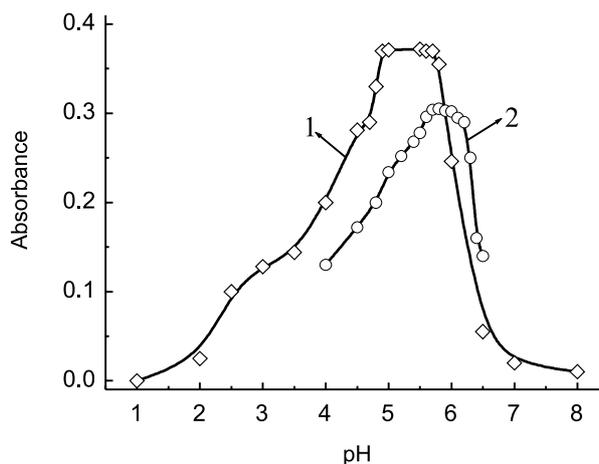


Figure 2. Effect of pH on the absorbance of osmium(IV)–OG complex; $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 4.52 \times 10^{-5} \text{ mol L}^{-1}$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$; 1 – $C(\text{NaCl}) = 0.10 \text{ mol L}^{-1}$; 2 – $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$

The Os(IV)–OG complex was formed in the sufficiently wide pH range. The maximum yield of the compound was achieved at pH 4.8–5.8 in the presence of NaCl and at pH 5.6–6.2 in the presence of acetate buffer solution. Possibly, in these media Os(IV) ions occurred in the form of mixed-ligand aquahydroxochloride complexes exhibited some acidity which more likely interacted with OG. All further investigations were carried out at $\text{pH} = 5.80 \pm 0.05$ using acetate buffer solution.

Effect of background electrolyte

In order to select a background electrolyte for stabilization of the reaction media, we studied how the nature and concentration of different sodium salts affected the interaction between Os(IV) and Orange G (Fig. 3).

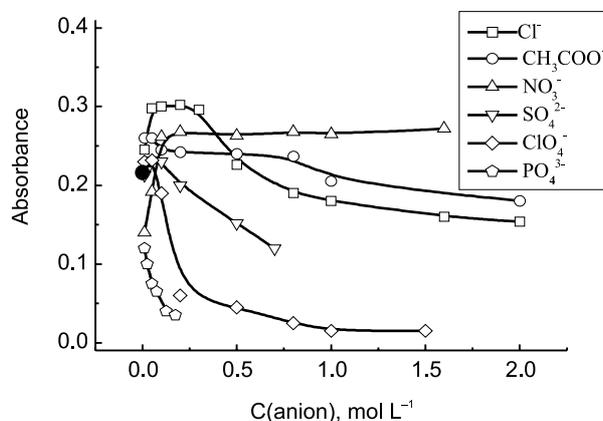


Figure 3. Influence of the concentration of various sodium salts on the absorbance of Os(IV)–OG complex $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 4.52 \times 10^{-5} \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$; filled symbol – without any additional background electrolyte

The presence of chloride, nitrate and acetate ions increased the absorbance of Os(IV)–OG compound. It was necessary to select an appropriate buffer solution to provide stable conditions of the reaction and to make the procedure of osmium(IV) determination with OG more rapid. According to the experimental results, the presence of 0.20 mol L^{-1} sodium acetate slightly increased the formation yield of Os(IV)–OG. 0.20 mol L^{-1} acetate buffer solution, in turn, had sufficient buffer capacity in the optimal pH range for the complex formation. Moreover, according to reference [1], the presence of carboxylic acids ions sufficiently improves the analytical characteristics of spectrophotometric reactions, *e.g.* repeatability. A decrease of sensitivity due to the lower absorbance of Os(IV)–OG compound in the presence of acetate buffer was rather small. Thus, acetate buffer solution was chosen as the reaction medium.

Effect of heating time

We investigated the kinetics of interaction between Os(IV) and OG at the room temperature ($\sim 20^\circ\text{C}$) and upon heating on a boiling water bath ($\sim 98^\circ\text{C}$), as well as time stability of the obtained complex. At the room temperature Os(IV) practically did not interact with OG. Os(IV)–OG compound was almost fully obtained after 30 min of heating on a boiling water bath (Fig. 4). This time of heating was applied in further studies; prolonged heating slightly increased the absorbance of the Os(IV)–OG system. Probably, when heated, the Os(IV)–OG complex was formed more rapidly, due to the decreased kinetic inertness of the initial OsCl_6^{2-} . The obtained Os(IV)–OG complex was stable for more than 24 h.

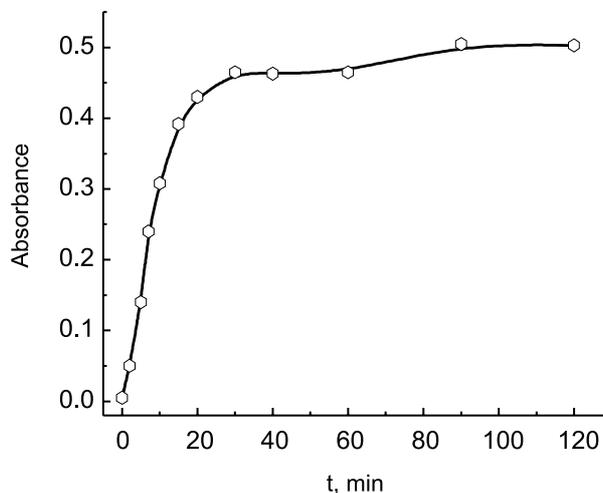


Figure 4. Influence of the heating time on Os(IV)–OG complex formation; $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 4.52 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$

Effect of addition order of reagents

Experimental results showed that the absorbance of the investigated system depended on the addition order of reagents. The following addition order of the reagents was optimal: acetate buffer, Orange G, and osmium(IV). Then pH of the media was adjusted using HCl and NaOH solutions, and the system was heated on a boiling water bath.

Determination of stoichiometric ratio in the complex compound

To establish the molar ratio of the components in the Os (IV)–Orange G complex, we used mole ratio method and method of continuous variations. The stoichiometric Os(IV):OG ratios estimated by both these methods were consistent and equalled to 2:1.

Effective molar absorptivity ϵ_λ of the Os(IV)–Orange G system was $1.1 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$; it was calculated using the results obtained in the method of continuous variation. According to the mole ratio curve, 2-fold reagent excess was necessary for the maximum formation yield of the Os(IV)–OG compound.

Effect of interfering ions

Investigation of selectivity of reaction between Os and OG was an important task in elaboration of a new spectrophotometric method for determination of osmium,

because its determination in real samples is generally complicated due to the presence of other metals, particularly PGEs. We studied the effect of several metals cations and typical masking agents on the results of spectrophotometric determination of Os(IV) in the presence of Orange G (Tab. 1). Larger than 10% deviation in the absorbance of the Os(IV)–Orange G system in the presence of a foreign ion was considered as the criterion of interference; the corresponding tolerance limits, expressed as Os(IV):ion molar ratios, are given in Table 1.

Table 1. Selectivity of spectrophotometric determination of Os(IV) using OG; $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 6.02 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$

Ion	$C_{\text{Os}}:C_{\text{ion}}$	Ion	$C_{\text{Os}}:C_{\text{ion}}$	Ion	$C_{\text{Os}}:C_{\text{ion}}$	Ion	$C_{\text{Os}}:C_{\text{ion}}$
Ni ²⁺	1:0.5	Pb ²⁺	1:5	Gd ³⁺	1:5	EDTA	1:1
Cu ²⁺	1:1.5	Cd ²⁺	1:1	Nd ³⁺	1:0.1	Cit ³⁺	1:20
Co ³⁺	1:200	Ce ³⁺	1:4	Ag ⁺	1:0.3	Tart ²⁺	1:5
Al ³⁺	1:300*	Ce ⁴⁺	1:0.1	Au ³⁺	1:<0.1	C ₂ O ₄ ²⁻	1:20
Fe ³⁺	1:3	Tb ³⁺	1:<0.1	Pt ⁴⁺	1:<0.1	P ₂ O ₇ ⁴⁻	1:50
Mg ²⁺	1:500*	Sm ³⁺	1:0.1	Pd ²⁺	1:<0.1	PO ₄ ³⁻	1:100
Ca ²⁺	1:500*	Ho ³⁺	1:5	Ru ⁴⁺	1:2	SiO ₃ ²⁻	1:40
Zn ²⁺	1:20	Eu ³⁺	1:20	Rh ³⁺	1:2	F ⁻	1:500*

* Higher concentrations of marked ions were not investigated.

According to the obtained results, large amounts of Ca²⁺, Mg²⁺, Co³⁺, Al³⁺, Zn²⁺ and Eu³⁺ and commensurable quantities of some rare-earth heavy metals, Rh(III) and Ru(IV) did not interfere with Os(IV)–OG complex formation. However, small amounts of other PGEs, Au³⁺, Ag⁺, Tb³⁺, Ce⁴⁺ and Sm³⁺ interfered strongly. Concerning the influence of possible masking agents, the presence of substantial quantities of organic anions hinder the complexation of Os(IV) with OG, however large excesses of F⁻, PO₄³⁻ and P₂O₇⁴⁻ almost did not effect. Hence, these reagents were utilized as the masking agents in spectroscopic determination of Os(IV) in the presence of OG.

Masking of interfering ions

Since preliminary separation of small quantities of osmium from complex samples in a form of volatile tetraoxide can cause serious error in the analysis results due to probable losses of OsO_4 during distillation process, the use of appropriate masking agents can be very helpful in the analyses of different samples containing several concomitant and interfering agents. Table 2 presents the results of masking of some interfering ions using citrate, tartrate, EDTA, F^- , PO_4^{3-} , and $\text{P}_2\text{O}_7^{4-}$.

Table 2. $C_{\text{Os}}:C_{\text{Ion}}:C_{\text{Masking agent}}$ ratios tested during spectrophotometric determination of osmium(IV) in the presence of Orange G; $C(\text{Os(IV)}) = 3.01 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{OG}) = 6.02 \times 10^{-5} \text{ mol L}^{-1}$ $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$

Interfering ion	Masking agents ($C_{\text{Os}}:C_{\text{Ion}}:C_{\text{Masking agent}}$)					
	Cit ³⁻	Tart ²⁻	PO ₄ ³⁻	EDTA	P ₂ O ₇ ⁴⁻	F ⁻
Fe ³⁺	1:7:20	1:10:5	–	*	–	–
Ni ²⁺	1:1:20	1:4:5	–	*	–	1:1:500
Cu ²⁺	–	–	1:3:100	*	–	1:2:500
Zn ²⁺	1:100:20	1:200:5	–	*	–	1:70:500
Au ³⁺	–	–	–	*	1:0.5:50	–
Nd ³⁺	–	1:1:5	–	*	–	1:0.5:500
Ce ⁴⁺	–	1:10:5	1:2:100	*	1:0.5:50	1:2:500
Sm ³⁺	–	1:9:5	1:0.5:100	*	1:5:50	1:0.1:500
Tb ³⁺	–	1:20:5	1:0.5:100	*	–	1:0.5:500
Ru ⁴⁺	–	1:3:5	–	*	*	*
Pd ²⁺	1:0.3:30	1:2:10	–	1:0.2:2	1:0.3:100	–
Pt ⁴⁺	–	1:0.1:5	1:0.5:100	1:1:2	–	1:0.1:100

– No masking effect detected.

* Not investigated.

Thus, the investigated masking agents allowed for elimination of the effect caused by many interferents during spectrophotometric determination of osmium in the presence of Orange G in complex samples.

Sensitivity and linear range

Linear range of the proposed method for spectrophotometric determination of Os(IV) with Orange G was 0.01–7.70 $\mu\text{g mL}^{-1}$; regression equation of the calibration plot was $\Delta A = 0.038 + 0.126 \times C_{\text{Os}}$, correlation coefficient $R = 0.9991$. Limit of detection and limit of quantification equalled respectively to 0.02 $\mu\text{g mL}^{-1}$ and 0.07 $\mu\text{g mL}^{-1}$, and Sandell's sensitivity m_s was 0.03 $\mu\text{g cm}^{-2}$.

It was concluded that the developed spectrophotometric method for determination of Os(IV) has a wide linear concentration range and high sensitivity – it is more sensitive than most known methods for spectrophotometric quantification of Os in industrial laboratories, as well as the recently developed ones [4–6, 23–28].

Application to samples

The accuracy of spectrophotometric determination of osmium was tested on model solutions containing different noble metals ions. The obtained results are presented in Table 3. In case of EDTA and sodium tartrate used as masking agents, PGE ions almost did not affect the results of the analyses. The determined contents of Os correlated well with the added amounts. RSDs did not exceed typical values for spectrophotometric methods.

Table 3. Accuracy of spectrophotometric determination of Os(IV) in model solutions using Orange G ($n = 5$; $P = 0.95$); $C(\text{OG}) = 6.02 \times 10^{-5} \text{ mol L}^{-1}$; $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$

System	$C_{\text{Os}}:C_{\text{Ion}}:C_{\text{Mask.agent}}$	Added osmium, μg	Found osmium, $\bar{x} \pm \frac{S \cdot t_{\alpha}}{\sqrt{n}}$, μg	RSD, %
Os–Pd–EDTA	1:0.2:2	143.3	139.1 ± 5.3	3.7
Os–Pt–EDTA	1:1:2	143.3	147.6 ± 5.3	3.7
Os–Pd–Tart	1:0.5:10	143.3	148.9 ± 6.9	4.8
Os–Pt–Tart	1:0.1:5	143.3	142.5 ± 1.0	0.8

On the basis of the developed spectrophotometric method for determination of Os(IV) in the presence of OG, the content of osmium was estimated in intermetallides samples applying calibration curve method and using sodium tartrate as the masking agent.

The procedure of dissolution of intermetallide samples was as follows: 0.01–0.05 g of the intermetallic alloy was dissolved in 10–20 mL of a mixture of concentrated hydrochloric and nitric acids (10:1) and boiled in a beaker on a sand bath for 1–2 h. Under these conditions, grey-coloured residue (elemental silicium) was formed on the bottom of the beaker. After quantitative transfer of the solution to a 100.0 mL volumetric flask, the residue was washed with 1 mol L⁻¹ NaOH solution for a few times. The washing liquid was placed in the same volumetric flask and distilled water was added to complete the volume to 100.0 mL. For the analytical procedure, 0.5–2.0 mL aliquots were taken. The results of osmium determination in intermetallides are presented in Table 4. Other elements present in samples solutions did not interfere with the results of analyses. The obtained data correlated well with theoretical contents of osmium in the examined intermetallides samples. Standard deviations did not exceed the values typical for spectrophotometric methods.

Table 4. Results of spectrophotometric determination of osmium in intermetallides using Orange G ($n = 5$; $P = 0.95$); $C(\text{OG}) = 1.81 \times 10^{-4} \text{ mol L}^{-1}$; $C(\text{Tart}) = 1.52 \times 10^{-4} \text{ mol L}^{-1}$; $C(\text{CH}_3\text{COONa} + \text{CH}_3\text{COOH}) = 0.20 \text{ mol L}^{-1}$; $\text{pH} = 5.80$; $l = 3 \text{ cm}$; $\lambda = 540 \text{ nm}$. Calibration plot equation: $\Delta A = 0.04224 + 0.00211 \times m_{\text{Os}}$, m_{Os} – mass of osmium in the aliquot of intermetallide sample solution, μg ; $R = 0.9987$

Intermetallide	$C_{\text{Os}}^{\text{teor}}$, %	Mass of the sample, g	$C_{\text{Os}}^{\text{teor}}$, $\mu\text{g mL}^{-1}$	$\bar{C}_{\text{Os}}^{\text{pract}}$, $\mu\text{g mL}^{-1}$	$\bar{C}_{\text{Os}}^{\text{pract}} \pm \frac{S \cdot t_{\alpha}}{\sqrt{n}}$, %	RSD, %
Nd ₂₀ Os ₁₅ S ₆₅	37.7	0.0136	51.3	52.9	38.9 ± 1.4	3.8
		0.0230	86.8	83.7	36.8 ± 1.7	4.6
Nd ₁₀ Os ₃₀ S ₆₀	64.6	0.0173	111.8	107.0	61.8 ± 3.5	5.4
		0.0494	319.3	308.1	62.4 ± 2.8	4.4

Hence, it was concluded that the elaborated spectrophotometric method for Os(IV) determination using OG azo dye exhibits good reproducibility and rapidity of analysis. It competes favourably with other methods widely used for spectrophotometric determination of osmium.

CONCLUSIONS

Optimum conditions for the formation of coloured complex compound between Os(IV) and acidic monoazo dye Orange G were established. The Os(IV):OG stoichiometric ration in the complex was 2:1. Effective molar absorptivity was $\epsilon_{\lambda} = 1.1 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. Moreover, a new sensitive spectrophotometric method for determination of Os(IV) was developed. The proposed procedure allowed for determination of Os(IV) in a wide range of concentrations: 0.01–7.70 $\mu\text{g mL}^{-1}$ (LOD = 0.02 $\mu\text{g mL}^{-1}$; LOQ = 0.07 $\mu\text{g mL}^{-1}$). Selectivity of spectrophotometric determination of Os(IV) in the presence of noble metal ions and other interferents was studied. The effect of the interferents was eliminated using the masking agents. The proposed technique was found more sensitive than most other spectrophotometric techniques for determination of osmium and can be recommended for determination of osmium in various samples.

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