Scientific paper

A New Reagent for Spectrophotometric Determination of Ir(IV): 5-[2-(4-Hydroxyphenyl) hydrazineylidene]-4-iminothiazolidin-2-one (HPIT)

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Abstract

The paper presents a new azolidone derivative – 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one (HPIT) studies and its interaction results with iridium(IV) ions. The Ir(IV) with this reagent in the pH = 5.0 without heating forms a stable complex (λ_{max} = 328 nm). The stoichiometric ratio of Ir(IV) to the reagent in complex is 1:1. The molar absorptivity and Sandell's sensitivity are 5.57×10^3 L mol^{-1} cm^{-1} and 0.034 μg cm^{-2} respectively. The calibration curve is linear in the range of 1.0–11.5 μg mL^{-1} of Ir(IV) (R = 0.9996). The limit of detection is 0.4 μg mL^{-1}. Based on the conducted investigation a rapid and simple, spectrophotometric method for the determination of Ir(IV) using 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one as a chromophoric reagent was developed. The iridium(IV) was determined in various synthetic mixtures and alloys.

Keywords: Iridium(IV); spectrophotometry; 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one; azolidones.

1. Introduction

The relevance of platinum metals and their compounds usage encourages the development of simple, rapid, inexpensive, selective, and sensitive methods for the determination of trace amounts of these metals in complex samples. Spectrophotometric methods of analysis are successfully used to solve this problem in analytical chemistry with the use of organic reagents containing functional-analytical groups.[1,2]

Spectrophotometric methods are one of the most widely used physical-chemical analysis methods in industrial and research laboratories. The main advantages of spectrophotometry are versatility, sufficient sensitivity to solve specific analytical problems, simplicity, the possibility of analysis automation, which provides it a leading place in the modern analytical chemistry. Spectrophotometric methods of platinoids determination are characterized by different sensitivity ($\varepsilon_\lambda \sim 10^3 – 10^5$ L mol^{-1} cm^{-1}) depending on the choice of reagent. Their sensitivity is significantly increased using organic reagents, namely heterocyclic azo derivatives and sulfur-containing compounds.[1–3] Among a large number of azo dyes, azolidones and their derivatives are of particular interest. The color reactions of noble metals with this group of reagents are highly sensitive ($\varepsilon = (0.4–1.5) \times 10^4$ L mol^{-1} cm^{-1}) and contrast ($\Delta\lambda$ = 70–80 nm). Azorodanines contain several functional-analytical groups in their molecules, which makes it possible to use them as group reagents for the precious metals determination.[3–6]

Our scientific group first investigated the analytical properties of several azolidone derivatives and successfully used them to determine the number of ions: Cu(II), Ni(II), Cd(II), Zn(II), Hg(II), Pd(II), Pt(IV), Rh(III), Ir(IV), Ru(IV).[7–21]

Due to the wide usage of iridium in various industries (such as jewelry, electrical equipment, dental alloys, automobile, chemical, and electronics industries, the field of photography and aviation), it is important to develop efficient analytical methods for this metal determination in various samples.[22,23] Most of spectrophotometric methods for the Iridium determination are not enough sensitive, selective and need heating or extraction.[1,2,4,11,24–27] For this reasons the goal of our research was to find the effective analytical reagents for spectrophotometric Ir(IV) determination using azolidones derivatives. One of them was a new reagent – 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one (HPIT).

2. Experimental

2. 1. Equipment

Spectrophotometric measurements were performed with a computerized spectrophotometer, model ULAB 108-UV, fitted with 1.0 cm quartz cells.

Computerized device MTech OVA-410 with a linear potential sweep was used for voltammetric measurements.[28] A three-electrode system including dropping mercury electrode (working electrode), a saturated calomel electrode (reference electrode), and platinum (counter electrode) were used.

The pH-150 M pH-meter equipped with a combined glass electrode was used to measure the pH values of solutions.

^{1}H NMR spectra were registered on the spectrometer Varian Mercury UX-400, DMSO-d_6 was used as a solvent, tetramethylsilane as a standard.

2. 2. Reagents

The stock solution of iridium(IV) chloride was prepared by melting the exact mass of pure iridium (99.99%) with the oxidizing mixture of $NaNO_3$ + NaOH (1:3, *v/v*), and BaO_2 at 950 K for 45–60 min. Then the fusion was dissolved in 3.0 mol L^{-1} hydrochloric acid. The existence form of Ir(IV) ($[IrCl_6]^{2-}$) in the obtained solution was confirmed by comparing its absorption spectra with the transferred data.[29] The obtained solution of Ir(IV) was additionally standardized using the titration method – iodometry, due to possible losses during sintering.[1,2] Standard transferred solutions of Ir(IV) were prepared by diluting an aliquot of Ir(IV) initial stock solution in 1.0 mol L^{-1} HCl.

The solution of 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one was prepared by dissolving the exact mass of the pre-purified reagent in dimethyl sulfoxide. Working solutions of HPIT were prepared by diluting an aliquot of the stock solution in dimethyl sulfoxide. The 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one was synthesized by the following procedure: 0.01 mol of 4-aminophenol was dissolved in 3 ml of concentrated hydrochloric acid, after which 5 ml of water was added. The solution obtained at this stage, with cooling, was diazotized with 0.72 g of transferred nitrite dissolved in 3 mL of water. The resulting diazonium salt was added over 30 minutes to a solution of 0.01 mol of 4-iminothiazolidin-2-one previously dissolved in 80 ml of glacial acetate acid containing 4 g of anhydrous sodium acetate (pH = 4.5–5.0) with stirring and was cooled. The mixture was left at 12 h, after which it was poured into 200 mL of water. The precipitate was filtered, washed on the filter with water, dried, and recrystallized. ^{1}H NMR (400 MHz, DMSO-d_6; δ, ppm): 6.70 (d, *J* = 9.0 Hz, 2H, C_6H_4), 7.28 (d, *J* = 9.0 Hz, 2H, C_6H_4), 8.71 (s, 1H, NH), 8.97 (s, 1H, NH), 10.17 (s, 1H, NH). The purity of HPIT was determined by chromatography-mass spectrometry.

The solution of HCl was prepared by dilution of concentrated HCl. The solutions of sodium salts (to study the effect of anions), NaCl and NaOH were prepared by dissolving an appropriate amount of respective salts and NaOH in distilled water. The Britton–Robinson buffer (BRB) was prepared by mixing solutions of boric, phosphoric and acetic acids.[30] The solutions of various metals (to study the effect of cations) were prepared by dissolving the exact mass of the corresponding metal in HCl or HNO_3 acids either its mixture, or their salts in distilled water or dilute hydrochloric or nitric acids. The solutions of Rh(III) and Ru(IV) were prepared by sintering corresponding metal with the oxidizing mixture $NaNO_3$ + NaOH (1:3, *v/v*) with further dissolving the fusion in 3.0 mol · L^{-1} HCl.

All chemicals used were of analytical grade and distilled water was used for the preparation of the aqueous solution.

2. 3. Procedure

Research of the HPIT spectral characteristics

Aliquots of 2.0 mL HPIT working solution (2.5×10^{-4} mol L^{-1}) were transferred into a series of 25.0 mL transferred flasks, then 2.0 mL BRB (1.5 mol L^{-1}), 1.25 mL NaCl (2.0 mol L^{-1}) and water (~15–20 mL) were added to each flask. The pH values (2.0–12.0) were adjusted using NaOH (4.0 mol L^{-1}) and then diluted to volume with distilled water. The solution with pH = 1.0 was prepared as described above but without BRB addition and pH value was adjusted using HCl (6.0 mol L^{-1}). The absorption spectra were measured against distilled water as blank.

General procedure for the determination of Ir(IV) with HPIT

An aliquot of Ir(IV) solution (in the range of 1.0–11.5 μg mL^{-1} in the final volume), 2.5 mL (1.0×10^{-3} mol L^{-1}) HPIT, 1.0 mL (1.5 mol L^{-1}) BRB and 1.25 mL (2.0 mol L^{-1}) NaCl were placed into 25.0 mL calibrated flasks and then distilled water was added (~15 mL). The pH was adjusted to ~5.0 with NaOH solution and diluted to the mark with distilled water. The absorbance was measured at 328 nm against a reagent blank.

Determination the stoichiometric ratio of the Ir(IV)-HPIT complex

The equimolar solutions of Ir(IV) and HPIT (1.0×10^{-3} mol L^{-1}) were used to determine the metal to ligand ratio by Job's method of continuous variation. The total concentration $C_{Ir(IV)} + C_{HPIT}$ was equal 1.0×10^{-4} mol L^{-1}

in a 25.0 mL volumetric flask. Then 1.0 mL (1.5 mol L^{-1}) BRB, 1.25 mL (2.0 mol L^{-1}) NaCl, and distilled water (~15 mL) were added. The pH was adjusted to 5.0 by NaOH and diluted with distilled water to the calibration mark. The absorbance values were recorded at 328 nm.

The mole-ratio method was performed in the transferred way: into a series of 25.0 mL volumetric flasks the Ir(IV) solution with fixed concentration (0.5 mL of 1.0×10^{-3} mol L^{-1}), an aliquot 0.10–10.0 mL (5.0×10^{-4} mol L^{-1}) HPIT, 1.0 mL (1.5 mol L^{-1}) BRB, 1.25 mL (2.0 mol L^{-1}) NaCl, and distilled water ~ 20 mL were added. The pH was adjusted to pH = 5.0 by adding NaOH and diluted up to the mark with water. Then, the absorbance at 328 nm was measured.

Alloys samples preparation

The alloys ($Gd_2Ir_3Al_9$, $Tb_2Ir_3Al_9$) were synthesized by arc melting of pure metals (Gd ≥ 99.86%, Tb ≥ 99.83%, Ir ≥ 99.9%, Al ≥ 99.998%) under an argon atmosphere and heated to 873 K, held at that temperature for 720 h and then cooled to room temperature.[31]

The solutions of alloys samples were prepared by dissolving of 0.05–0.1 g of sample in 10–20 mL of HCl and HNO_3 (10:1, *v/v*) mixture and heating for ~ 2 h. Then the black residue was filtered and filtrate transferred to a 200.0 mL volumetric flask. The residue was sintered with a $NaNO_3$ and NaOH (1:3, *v/v*) mixture at 950 K (60 min). The melt was dissolved in 3.0 mol L^{-1} HCl. The obtained solution was transferred to the previous filtrate and distilled water was added to the mark. The 0.4–1.5 mL of alloys aliquots were taken for Ir(IV) determination with HPIT as described above.[11]

3. Results and Discussion

3. 1. Research Spectral Characteristics of the HPIT

We researched a new reagent – 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one, which is a derivative of azolidone (Fig. 1). HPIT is the crystalline yellow powder poorly soluble in water and ethanol but well soluble in dimethylformamide and dimethyl sulfoxide. The melting point is 515 K.

In our previous work,[32] we investigated the effect of the medium acidity on the absorption spectra of HPIT over the pH range 1.0–12.0. As shown by the results of the experiment, the HPIT absorption maximum depends on the pH of the solution, which is associated with different existence forms depending on the medium acidity (Fig. 2). At pH = 1.0, the absorption spectrum is characterized by a maximum at a wavelength of 418 nm, which corresponds to the protonated form of the reagent; at pH 2.0–10.0, the maximum of absorbance shifts slightly to the region of smaller wavelengths (400 nm), and in the alkaline medium (pH>10.0) (Fig. 3), there is a course of hydrolysis reaction with the release of ammonia (Scheme 1). The molar absorptivity at λ = 418 nm is 2.04×10^4 L mol^{-1} cm^{-1} (pH = 1.0); at λ = 400 nm is $(1.14–1.85) \times 10^4$ L mol^{-1} cm^{-1} (pH 2.0–9.0) and 1.02×10^3 L mol^{-1} cm^{-1} (pH = 10.0).

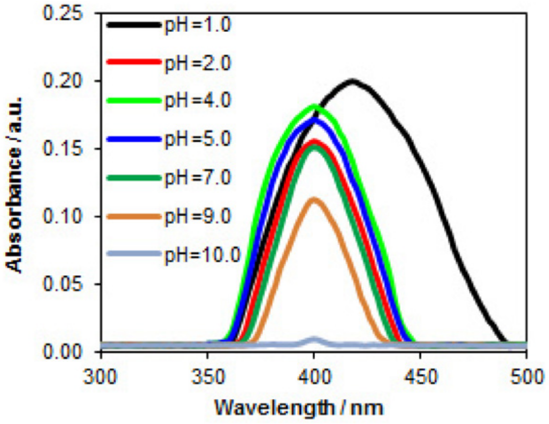


Fig. 2. Absorbance spectra of HPT at different pH

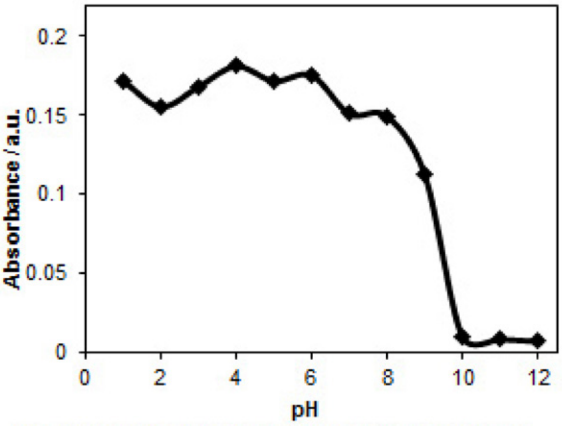


Fig. 3. Effect pH on the absorbance of HPIT at 400 nm

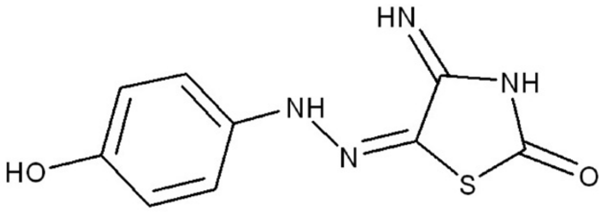
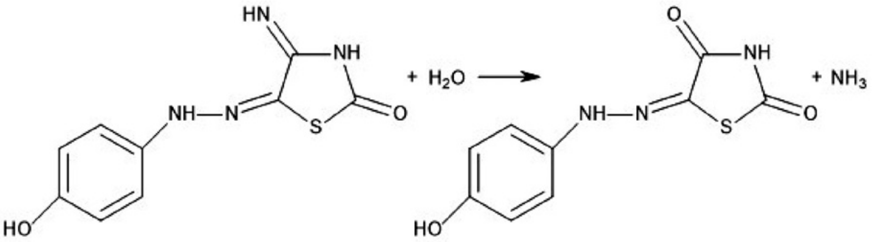


Fig. 1. Structual formula of the HPIT



Scheme 1. Hydrolysis of 5-[2-(4-hydroxyphenyl)hydrazineylidene]-4-iminothiazolidin-2-one

HPIT does not polymerize and does not form tautomeric forms at pH = 1.0 in the concentration range of 5.0 × 10^{-6}–8.0 × 10^{-5} mol L^{-1}, since in this range the Beer's law is applicable and only one maximum is observed.

3. 2. Investigation of the Interaction of Ir(IV) with HPIT

It was found that the Ir(IV) ions form a complex with HPIT (Fig. 4). The absorption spectra of HPIT, Ir(IV), and Ir(IV)-HPIT were recorded over the range 200 to 550 nm. As shown on Fig. 4 the maximum of the reagent at 400 nm (pH = 5.0) is reduced in the presence of iridium(IV) ions. Instead, there was an increase in the absorption of Ir(IV)-HPIT in the wavelength range from 250 to 360 nm compared to the absorption of the reagent, which indicates the interaction. The largest difference in absorption of the reagent and compound is at λ = 328 nm.

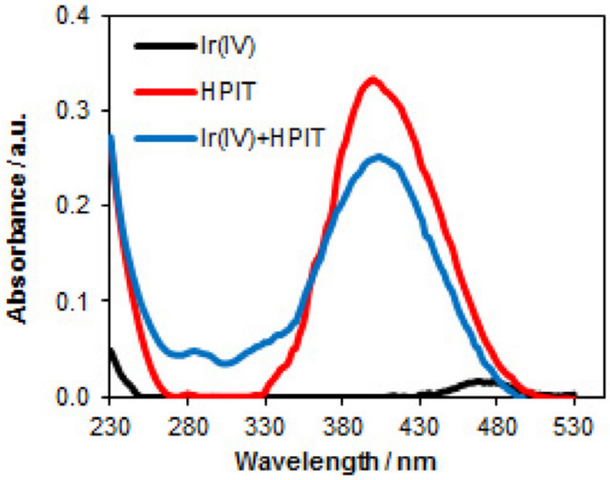


Fig. 4. Absorbance spectra of Ir(IV), reagent and complex Ir(IV) with HPIT (pH = 5.0, $C_{ir(IV)} = 8.0 \times 10^{-6}$ mol L^{-1}, $C_{HPIT} = 2.0 \times 10^{-5}$ mol L^{-1})

The effect of various parameters on the formed products absorption intensity was studied and the reaction conditions were optimized.

Effect of pH

The acidity of the medium is one of the important parameters that affect the complexation. Within the pH range from 2.0 to 10.0 Ir(IV) ions form a complex with HPIT (Fig. 5). The maximum yield of complex is at pH = 5.0. Hence this value of pH was selected for further studies.

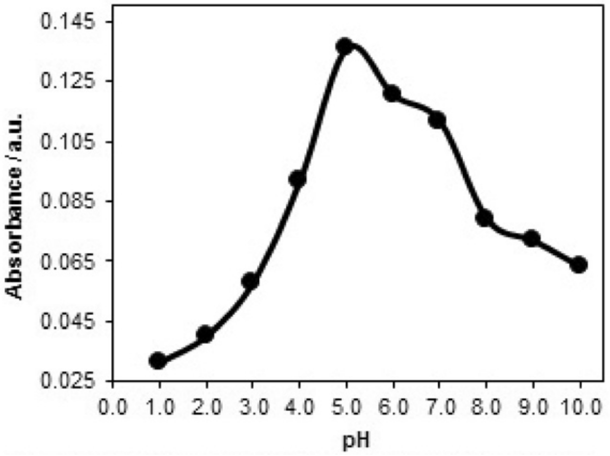


Fig. 5. Effect of pH on the maximum yield of Ir(IV) with HPIT complex (λ = 328 nm, $C_{ir(IV)} = 2 \times 10^{-5}$ mol L^{-1}, $C_{HPIT} = 4 \times 10^{-5}$ mol L^{-1})

Effect of time and temperature

The process of iridium(IV) ions complexation with HPIT occured at room temperature (~ 291–296 K) immediately after the acidity of the medium was established. The effect of heating time on the maximum yield of the colored compound was investigated. The solutions heating in a boiling water bath (~ 371 K) caused a decrease in the absorption of the solutions, but the complex compound Ir(IV)–HPIT was not destroyed even when heated for 60 min. Therefore it is recommended that the reaction should be carried out at room temperature. The absorbance of obtained Ir(IV)–HPIT complex was stable up to 72 h.

The stoichiometric ratio of complex

The Job's method of continuous variations (Fig. 6) and the mole-ratio method were used to determine the stoichiometric ratio of the complex. These methods indicate that complex with stoichiometry 1:1 was formed. A 1.5 fold excess of reagent is required for full complexation. The formal stability constant of the Ir(IV)–HPIT complex was calculated and it is equal to 8.9 × 10^5.

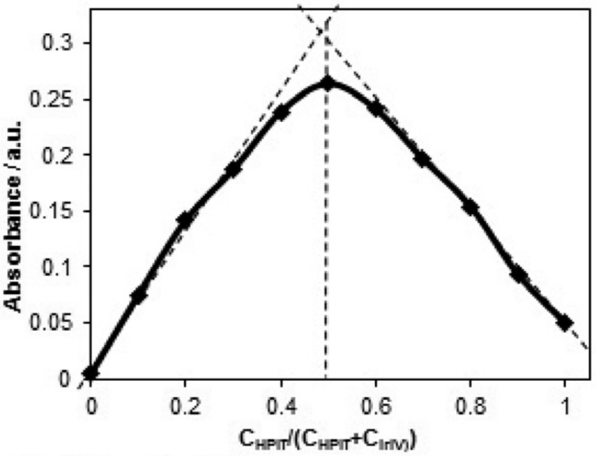


Fig. 6. The method of continuous variations

Calibration curve

The calibration graph for Ir(IV) determination with HPIT was constructed using the optimal conditions (pH = 5.0, λ = 328 nm, $C_{HPIT} = 1.0 \times 10^{-4}$ mol L^{-1}, $C_{NaCl} = 0.1$ mol L^{-1}, $C_{BRB} = 0.06$ mol L^{-1}) and showed that the system obeys Beer's law in the concentration range of 1.0–11.5 μg mL^{-1} of Ir(IV). The linear equation is $\Delta A = (0{,}011 \pm 0.004) + (0{,}0292 \pm 0.0007) \times C_{Ir(IV)}$ (ΔA – absorbance, C – concentration of Ir(IV) in μg mL^{-1}) with correlation coefficient equal 0.9996, N = 6, SD = 2.37×10^{-3}. The molar absorptivities and Sandell sensitivity are 5.57×10^3 L mol^{-1} cm^{-1} and 0.034 μg cm^{-2} respectively. The limit of detection is 0.4 μg mL^{-1}.

3. 3. Selectivity of the Ir(IV) Determination

The various cations and anions influence on the iridium(IV) determination were studied under the conditions of the standard procedure. The tolerance limits of tested interfering ions were calculated as the maximum concentrations that do not cause an error of more than ± 5% in an absorbance value. The tolerance limits for foreign ions are shown in Table 1. It has been found that the majority of cations do not interfere significantly. The iridium(IV) ions can be easily determined in the presence of Tb(III), Gd(II), Cd(II), Zn(II), Mn(II), Ni(II), Cu(II), Ca(II), Mg(II), Ba(II) and the studied anions. However, several ions such as Pd(II), Rh(III), Pt(IV), Ru(IV) interfere seriously. Their effect can be eliminated by using some of the studied anions as masking agents. For example, Pd(II) and Ru(IV) were masked using EDTA (the tolerance limits reach 1 for Pd(II) and 2 for Ru(IV)). This method has higher selectivity than most of the spectrophotometric methods of Iridium determination.[1, 2, 4, 11, 24–27]

3. 4. Analytical Application

The developed spectrophotometric method was applied to the determination of Ir(IV) in synthetic mixtures and alloys to validate it.

Analysis of Ir(IV) in synthetic mixtures

Different synthetic mixtures were prepared and analyzed using the proposed developed method in order to research the precision and accuracy (Table 2). As can be seen in Table 2, the obtained results are consistent with the

Table 1. Selectivity of Ir(IV) spectrophotometric determination ($C_{Ir(IV)} = 2.5 \times 10^{-5}$ mol L^{-1}; $C_{HPIT} = 1.0 \times 10^{-4}$ mol L^{-1}; $C_{NaCl} = 0.1$ mol L^{-1}; $C_{BRB} = 0.06$ mol L^{-1}; pH = 5.0; λ = 328 nm; *l* = 1.0 cm)

Foreign ion	Tolerance limit $C_{ion} : C_{Ir(IV)}$	Foreign ion	Tolerance limit $C_{ion} : C_{Ir(IV)}$	Foreign ion	Tolerance limit $C_{ion} : C_{Ir(IV)}$
Pd(II)	0.1	Ni(II)	150	Ca(II), Mg(II)	>200
Pt(IV)	0.25	Fe(III)	15	$C_2O_4^{2-*}$	100
Rh(III)	0.1	Cd(II)*	75	F^{-*}	100
Ru(IV)	0.25	Pb(II)*	75	EDTA*	100
Tb(III)	100	Mn(II)*	100	Sal^{-*}	100
Gd(III)	75	Zn(II)	75	$Citr^{3-*}$	100
Cu(II)	50	Al(III)	8	$Tart^{2-*}$	100
Co(II)	30	Ba(II)	75	PO_4^{3-*}	100

*These ions decrease the absorbance value by ±5%, and the others all increase.

Table 2. Determination of Iridium(IV) in synthetic mixtures, n = 3; P = 0.95

Composition of synthetic mixture	Added Ir(IV), μg	Found Ir(IV), $\bar{x} \pm \frac{s \times t_\alpha}{\sqrt{n}}$, μg	RSD, %
12.6 μg Ru(IV), 0.7 mg Co(II)	96	98±5	2.0
3.3 μg Pd(II), 2.8 mg Ni(II), 9.7 mg Pb(II)	96	101±7	2.8
24.4 μg Pt(IV), 3.4 mg Mn(II)	96	100±8	3.2

Table 3. Results of the determination of Ir(IV) in alloys, n = 3, P = 0,95

Alloy	ω_{Ir}^{calc},	Spectrophotometry		Voltammetry	
		$\overline{\omega}_{Ir}^{pr} \pm \frac{S \times t_{\alpha}}{\sqrt{n}}$, %	RSD, %	$\overline{\omega}_{Ir}^{pr} \pm \frac{S \times t_{\alpha}}{\sqrt{n}}$, %	RSD, %
$Gd_2Ir_3Al_9$	50.9	51.5 ± 1.8	1.4	51.2 ± 1.4	1.1
$Tb_2Ir_3Al_9$	50.7	51.0 ± 1.5	1.2	50.8 ± 1.6	1.3

added amounts of Ir(IV). The calculated values of the relative standard deviation and the absence of a significant systematic error indicate a good reproducibility and accuracy of this spectrophotometric method.

Analysis of Ir(IV) in the alloys

The results of Iridium determination in the alloys are given in Table 3. The data obtained by the spectrophotometric method were compared with Ir(IV) contents determined by the voltammetric method. As seen, the results of both methods are agreemented. The results in Table 3 show that the relative error and relative standard deviation do not exceed 1.5%.

4. Conclusions

The results of this research indicate that the developed spectrophotometric method based on the complexation of Ir(IV) with the new azolidone derivative (5-[2-(4-hydroxyphenyl)-hydrazineylidene]-4-iminothiazolidin-2-one) can be successfully used for the determination of iridium(IV) in different samples. This method is simple, sensitive, selective towards many ions, reproducible, and rapid because it does not require heating or separation from a large number of foreign ions (such as REE, Cu(II), Ni(II), Mn(II), Zn(II), Cd(II), et al.), which are associated with Ir(IV) in its objects. The time needed for analysis about 30 min, which makes this method much faster than other spectrophotometric methods for determining Iridium, which are described in the literature.

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Povzetek

V prispevku je predstavljen nov derivat azolidona – 5-[2-(4-hidroksifenil)hidrazineiliden]-4-iminotiazolidin-2-on (HPIT) in rezultati njegove interakcije z ioni iridija(IV). Ir(IV) ion s tem reagentom pri pH = 5,0 brez segrevanja tvori stabilen kompleks (λ_{max} = 328 nm). Stehiometrično razmerje Ir(IV) in reagenta v kompleksu je 1:1. Molarna absorptivnost in Sandellova občutljivost sta $5{,}57 \times 10^3$ L mol^{-1} cm^{-1} in 0,034 µg cm^{-2}. Kalibracijska krivulja je linearna v območju 1,0–11,5 µg mL^{-1} Ir(IV) (R = 0,9996). Meja zaznave je 0,4 µg mL^{-1}. Na podlagi izvedene raziskave je bila razvita hitra in enostavna spektrofotometrična metoda za določanje Ir(IV) z uporabo 5-[2-(4-hidroksifenil) hidrazineilidena]-4-iminotiazolidin-2-ona kot kromofornega reagenta. Ir(IV) je bil določen v različnih sintetičnih mešanicah in zlitinah.