Spectrophotometric Determination of Microamounts of Iridium(IV) with 3-Hydroxy-2-methyl-1-phenyl-4-pyridone or 3-Hydroxy-2-methyl-1-(4-tolyl)-4-pyridone

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Key words
extraction
iridium(IV)
4-pyridone
spectrophotometry
synthetic mixtures

A method for the extraction and spectrophotometric determination of iridium(IV) with 3-hydroxy-2-methyl-1-phenyl-4-pyridone (HX) or 3-hydroxy-2-methyl-1-(4-tolyl)-4-pyridone (HY) is described. The Ir^{IV}-HX(HY) complex was extracted from sulphuric or hydrochloric acid solutions into chloroform, dichloromethane, isobutyl methyl ketone or isoamyl alcohol. Absorbance was measured at 365 nm. The complex system conformed to Beer's law over the range of 1.9–38.5 mg cm^{-3} of iridium(IV). The relative standard deviations estimated from six independent determinations in samples of 3.84 and 15.36 mg cm^{-3} of iridium(IV) were 2 and 1.44 %, respectively. A derivative spectrophotometric method, based on digitized second-derivative absorption spectra, has been developed for the determination of microgram amounts of iridium(IV) with HX dissolved in chloroform in the presence of palladium(II). Iridium (1.9–34 μg cm^{-3}) was determined in the presence of palladium (1–8 μg cm^{-3}) with good precision and accuracy. The method was applied for the analyte determination in synthetic mixtures.

INTRODUCTION

Owing to their corrosion-resistant nature, iridium alloys have found a wide range of applications in both the chemical industry and manufacture. Iridium is also a minor constituent of most platinum metal deposits. Therefore, highly selective, sensitive, rapid and economical methods are needed for its trace determination. Neutron activation analysis,[1] atomic absorption spectrometry,[2] or inductively coupled plasma atomic emission spectrometry[3] may be used for this purpose. However, the necessary instruments are expensive, day-to-day maintenance costs are high and the methods are not free from various types of interferences.[4–7]

A wide variety of reagents have been proposed for the spectrophotometric determination of iridium.[8,9] Most available methods lack either sensitivity or selectivity, or both, and there are objectionable interferences from most noble metals. From the analytical point of view, the most closely associated platinum metal is rhodium. It is generally necessary to separate the two metals before determination.

Recently, gamma-pyridone derivatives, 3-hydroxy-2-methyl-1-phenyl-4-pyridone (HX) or 3-hydroxy-2-methyl-1-(4-tolyl)-4-pyridone (HY), have been reported as sensitive and selective complexing agents for the determination of titanium,[10] germanium,[11] vanadium,[12] and other

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metals. A systematic study on gamma-pyridone derivatives showed that HX and HY formed colourless species with iridium(IV) at room temperature, which could be extracted into chloroform. A detailed study of the Ir^{IV}-HX(HY) system has been carried out. In this paper, a method for the determination of iridium in various synthetic mixtures is described.

EXPERIMENTAL

Reagents

A stock solution of iridium(IV) chloride (5×10^{-3} mol dm^{-3}) was prepared by dissolving 0.02205 g of $(NH_4)_2IrCl_6$ (Aldrich, Chemical Company, Milwaukee, USA) in 10.00 cm^3 0.1 mol dm^{-3} solution of hydrochloric acid. The resulting solution was standardized gravimetrically using the dimethylglyoxime method.[13] A working solution was prepared daily by diluting the standard solution with deionized-distilled water.

3-Hydroxy-2-methyl-1-phenyl-4-pyridone (HX) and 3-hydroxy-2-methyl-1-(4-tolyl)-4-pyridone (HY) were synthesized as described previously.[14] Their solutions were prepared by dissolving weighed amounts of HX(HY) in chloroform (containing 0.5 % of ethanol and used without purification or drying).

Other standard solutions of different cations and anions, used to study the effect of various ions, were prepared by dissolving suitable quantities of their salts (Merck, Darmstadt, Germany; Fluka, Buchs, Switzerland) in deionized water or in dilute hydrochloric acid. Solutions of anions were prepared by dissolving the respective alkali metal salts in water. All the chemicals used were of analytical reagent grade.

Procedure

To an aliquot of sample solution containing 1.2×10^{-4} mol dm^{-3} of iridium(IV), an adequate amount of sulphuric (8×10^{-3} mol dm^{-3} H_2SO_4, $c(H,Na)_2SO_4 = 0.8$ mol dm^{-3}) or hydrochloric acid (1×10^{-3} mol dm^{-3} HCl, $c(H,Na)Cl = 1$ mol dm^{-3}) was added, and the volume was made up to 5 cm^3. The solution was equilibrated with 5 cm^3 HX(HY) in chloroform (6×10^{-4} mol dm^{-3}), and the phases were shaken mechanically in a 50 cm^3 conical flask for 20 min. The absorbance of the organic phase was measured at 365 nm against a reagent blank.

Apparatus

A Varian double-beam spectrophotometer, Model Cary 3, equipped with 1-cm quartz cells, controlled by Matsuba 386/33DX computer in conjunction with HP Plotter-7574, was used for absorption measurements. The spectra were recorded at a scan rate of 600 nm min^{-1}. Derivative spectra of different orders were obtained from stored zero-order spectra using numerical differentiation ($\Delta\lambda = 4$ nm) with the Varian software program.

A Griffin flask shaker with a time switch served for extraction. The pH of the aqueous phase was measured with a Radiometer PHM 85 precision pH-meter.

Analysis of Synthetic Mixtures

Different amounts of metal salts, including those of iridium, were mixed so that the resultant mixtures corresponded to standard reference samples such as alloys. The mixture (0.04–0.1 g) was dissolved in aqua regia by heating and evaporating nearly to dryness.[15] Concentrated hydrochloric acid (2 cm^3) was then added and the solution was warmed. After cooling to room temperature, it was diluted with deionized water to 100 cm^3 in a calibrated flask. Aliquots of sample solution (10–30 µg) of Ir^{IV} were analyzed for iridium as described above.

RESULTS AND DISCUSSION

Optimization of Determination Conditions

Iridium(IV) was extracted with 3-hydroxy-2-methyl-1-phenyl-4-pyridone (HX) or 3-hydroxy-2-methyl-1-(4-tolyl)-4-pyridone (HY) into chloroform in the form of colourless complexes with maximum absorbance at 365 nm. The extractability and absorbance of the metal complexes were affected by the choice of sulphuric or hydrochloric acids and by the ionic strength of the aqueous phase (Figure 1). The extraction was the highest from hydrochloric or sulphuric acid solutions at pH > 2.7, without constant ionic strength. In the presence of an excess of chloride or sulphate ions, the absorbance-pH curves shifted to higher acidities and iridium(IV) was extracted at pH > 2.0. Accordingly, 8×10^{-3} mol dm^{-3} H_2SO_4 at $c(H,Na)_2SO_4 = 0.8$ mol dm^{-3} ($pH_{read} = 2.7$), or 1×10^{-3} mol dm^{-3} HCl, at $c(H,Na)Cl = 1$ mol dm^{-3}) ($pH_{read} = 2.4$) were used in all further work.

By varying the shaking period from 1 to 60 min, it was shown that at least 20 minutes of shaking was
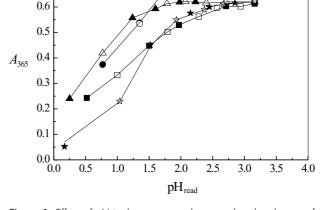

Figure 1. Effect of pH in the aqueous phase on the absorbance of Ir^{IV}-HX(HY) complexes in chloroform: $c(Ir^{IV}) = 1.2 \times 10^{-4}$ mol dm^{-3}; $c(HX) = 6 \times 10^{-4}$ mol dm^{-3} (■,★,●,▲); $c(HY) = 6 \times 10^{-4}$ mol dm^{-3} (□, ☆, ○, △). H_2SO_4 is varied without addition of Na_2SO_4 (■, □); HCl is varied without addition of NaCl (★, ☆); $c(H,Na)_2SO_4 = 0.8$ mol dm^{-3}, H_2SO_4 is varied (●, ○); $c(H,Na)Cl = 1.0$ mol dm^{-3}, HCl is varied (▲, △).

Figure 2. Determination of Ir^{IV} to HX ratio by (a) Job's method, and (b) the mole-ratio method. c(HCl) = 1×10^{-3} mol dm^{-3}, c(H,Na)Cl = 1.0 mol dm^{-3}.

needed for equilibration of the Ir^{IV}-HX(HY) complex extraction. The absorbance of the organic phase was stable for at least 1.5 h.

The influence of different concentrations of the reagents in chloroform was also studied. The HX(HY) concentration was varied from 0.0 to 3×10^{-3} mol dm^{-3}. Maximum absorbance was observed at reagent concentrations higher than 2.4×10^{-4} mol dm^{-3} (a 2-fold excess of reagent regardless of iridium(IV) concentration). Higher reagent concentrations had no effect on the absorbance of the organic phase. Therefore, 6×10^{-4} mol dm^{-3} HX(HY) in chloroform was selected for the study.
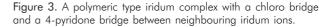
Composition of the Extracted Species

The ratio of iridium(IV) to HX(HY) in the extracted species was determined by Job's method, as modified by Vosburg and Cooper.[16] Absorbances were measured at four different wavelengths. For simpler illustration, Figure 2a presents the results obtained by extraction of iridium(IV) with HX from the hydrochloric acid solution into chloroform. Extraction from the sulphuric acid solution yielded identical results. Job plots were anomalous, having breaks in each curve at 0.6, regardless of reactant concentrations or the wavelength. This was an indication that the metal and the ligand, in the extracted species, were present in the ratio Ir^{IV}: HX = 1.5 : 1 or Ir^{IV}: HX = 3 : 2.

This was further confirmed by the mole ratio method shown in Figure 2b where the results obtained by extraction of iridium(IV) with HX from the hydrochloric acid solution into chloroform are presented. The mole ratio plots have an unusual shape, and the break points indicate the ratio Ir^{IV}: HX = 1 : 0.66 or Ir^{IV}: HX = 1.5 : 1.

Identical results were obtained by Ir^{IV} extraction with HY, regardless of the method used.

It was previously established[17–19] that iridium formed complex species where iridium ions were spanned with chloride ion bridges (*e.g.*, Ir(μ-Cl$_2$)Ir). By analogy with the work on copper complexes[20] and on palladium complexes[21] of some thiosemicarbazides, our results could be

Figure 3. A polymeric type iridum complex with a chloro bridge and a 4-pyridone bridge between neighbouring iridium ions.

accounted for by postulating formation of a polymeric type complex with a chloro bridge and a 4-pyridone bridge between the neighbouring iridium ions (Figure 3).
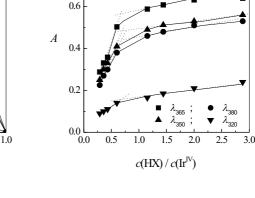
Calibration, Sensitivity and Reproducibility

A calibration curve was constructed under optimal conditions for Ir^{IV} at various concentrations, when the volume of the aqueous phase was equal to that of the organic phase. Beer's law was obeyed in the range of 1.9–38.5 μg cm^{-3} of iridium(IV). Molar absorptivities were 5.67×10^3 dm^3 mol^{-1} cm^{-1} (by extraction from sulphate solution) and 5.32×10^3 dm^3 mol^{-1} cm^{-1} (by extraction from chloride solution), regardless of the reagent used in the organic phase. Sandell's sensitivity of the systems was 0.034 and 0.032 μg cm^{-2}, respectively. The relative standard deviations evaluated from six independent determinations in samples of 3.84 and 15.36 μg cm^{-3} of iridium(IV) were 2 and 1.44 %, respectively.

The Ir^{IV}-HX(HY) complex was soluble in various organic solvents. Its molar absorptivities were also determined in dichloromethane, isobutyl methyl ketone, and isoamyl alcohol (Table I). The values varied about 8 % and increased in the order: isoamyl alcohol < chloroform < isobutyl methyl ketone < dichloromethane, regardless of the reagents used.

Effects of Diverse Ions

The effects of diverse ions on Ir^{IV} determination by liquid-liquid extraction with HX(HY) from chloride solu-

TABLE I. Effect of different solvents on molar absorptivity of Ir^{IV}-HX(HY)$^{(a)}$

Solvent	$10^{-3}\ \varepsilon$ / dm^3 mol^{-1}cm^{-1}	
	Chloride solution$^{(b)}$	Sulphate solution$^{(c)}$
Chloroform	5.32	5.67
Dichloromethane	5.88	6.08
Isobutyl methyl ketone	5.58	5.95
Isoamyl alcohol	4.94	5.26

$^{(a)}c$(HX or HY) = 6 × 10^{-4} mol dm^{-3}.
$^{(b)}c$(HCl) = 1 × 10^{-3} mol dm^{-3}, c(H,Na)Cl = 1.0 mol dm^{-3}.
$^{(c)}c$(H$_2$SO$_4$) = 8 × 10^{-3} mol dm^{-3}, c(H,Na)$_2$SO$_4$ = 0.8 mol dm^{-3}.

tions, were studied out at a fixed iridium concentration (1.2 × 10^{-4} mol dm^{-3}) with different amounts of foreign ions. The results are summarized in Table II. An error of ± 3 % in absorbance was considered tolerable. Metal ions were added as nitrate, chloride or sulphate, and anions as lithium, potassium or sodium salts. Most anions and cations were tolerated in large quantities. The results show that there was interference from anionic complexing agents such as thiocyanate, iodide and tartarate. Ruthenium(III) reacted with HX(HY), also giving a colourless complex. To eliminate its influence, an excess of the reagent had to be added. Platinum(IV) decreased, while molybdenum(VI) and iron(III) increased the absorbance. Rhodium(III) did not interfere in 25-fold amounts. It should be emphasized that identical analytical results were obtained with both investigated reagents.

Derivative Spectrophotometry

In our recent paper,[22] a satisfactory spectrophotometric method for the quantitative determination of palladium(II), using HX as reagent, was described. In iridium(IV) extraction with HX from sulphate solution, palladium(II) increased the absorbance. Under the present experimental conditions, the absorption maxima of the Ir^{IV}-HX and

TABLE II. Effect of foreign ions on the spectrophotometric determination of iridium(IV) with HX or HY dissolved in chloroform$^{(a)}$

Foreign ions	Tolerance limit [Ion] / [Ir^{IV}]
Sulphate, nitrate, chloride, Na^I, K^I, Mg^{II}	5000
Phosphate, perchlorate, Ni^{II}, Cu^{II}, Zn^{II}	500
Bromide, Co^{II}, Cd^{II}, Mn^{II}	100
Oxalate, Hg^{II}	50
Rh^{III}	25
Acetate	10
Carbonate, citrate, Os^{VIII}, Ag^I, Ga^{III}, Ru$^{III\ (b)}$	5
Tartarate, Mo^{VI}	1
Iodide, thiocyanate, Fe^{III}, Pd^{II}, Pt^{IV}	< 1

$^{(a)}c$(Ir^{IV}) = 1.2 × 10^{-4} mol dm^{-3}, c(HCl) = 1.0 × 10^{-3} mol dm^{-3}, c(H,Na)Cl = 1.0 mol dm^{-3}, c(HX or HY) = 6.0 × 10^{-4} mol dm^{-3}.
$^{(b)}c$(HX or HY) = 5.0 × 10^{-3} mol dm^{-3}.

Pd^{II}-HX complexes lay at 365 nm and 345 nm, respectively. Figure 4a shows the zero-order spectra of isomolar solutions of the Ir^{IV}-HX complex and the Pd^{II}-HX complex in chloroform (curves 1 and 2) and of their mixture (curve 3). Simultaneous spectrophotometric determination of iridium(IV) and palladium(II), when present in the same solution, is subject to considerable difficulties because of the considerable overlapping of the two spectra. The second-derivative spectrophotometry using HX as reagent can be a very useful tool for determining iridium(IV) in the presence of palladium(II), avoiding tedious and time-consuming separation procedures. The second-derivative spectra of the complexes and their mixture are shown in Figure 4b. The zero-crossing measurement technique was utilized to resolve the mixture of the complexes. Height H in the second-derivative spectra of the mixtures corresponding to values taken at 363.5 nm (zero-crossing point of the palladium complex) was proportional to iridium(IV)
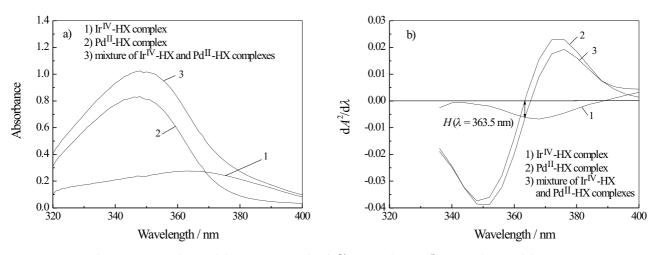

Figure 4. Zero-order spectra (a) and second-derivative spectra (b) of Ir^{IV}-HX complexes, Pd^{II}-HX complexes and their mixture. c(H$_2$SO$_4$) = 8 × 10^{-3} mol dm^{-3}, c(H,Na)$_2$SO$_4$ = 0.8 mol dm^{-3}, c(HX) = 2 × 10^{-3} mol dm^{-3}, c(Ir^{IV} or Pd^{II}) = 8 × 10^{-5} mol dm^{-3}.

TABLE III. Equations for calibration graphs and the correlation co-efficient for the determination of iridium(IV) with HX in mixtures with palladium(II) using second-derivative spectrophotometry[a]

γ (Pd) $\mu g\ cm^{-3}$	Regression equation[b]	Correlation coefficient
–	$D = 1.22 \times 10^{-3} - 1.45 \times 10^{-3}\ C$	–0.999
4.25	$D = -1.3 \times 10^{-4} - 1.46 \times 10^{-3}\ C$	–0.996
8.5	$D = 1.32 \times 10^{-3} - 1.48 \times 10^{-3}\ C$	–0.997

[a] γ(Ir) = 1.9–34 µg, λ = 363.5 nm.
[b] D, derivative value; C, concentration ($\mu g\ cm^{-3}$).

TABLE V. Determination of iridium in some synthetic mixtures

Sample no.	Mixture composition / %				Ir found[a] %
1	Ir 43.5,	Os 37.7,	Ru 11.3,	Pt 7.5	42.8 ± 0.9
2[b]	Ir 0.84,	Rh 21.2,	Cu 70.8,	Pd 7	0.81 ± 0.04
3	Ir 0.75,	Rh 1.6,	Ni 6.3,	Mn 21.1	0.84 ± 0.08
	Cd 12.7,	Pb 4.2,	Cr 0.6,	Mg 52.7	

[a] Average of four determinations.
[b] Determined by second-derivative spectrophotometry.

concentrations. A calibration graph for a simultaneous determination of iridium(IV) in the presence of palladium(II) using HX was obtained from H measurements in the second-derivative spectra for standards containing between 1.9 and 34 µg cm^{-3} of iridium(IV) in the presence of palladium (1–8 µg cm^{-3}) (Table III). To test the accuracy and precision of the method, six successive measurements were carried out with the standard solution containing 3.2 µg cm^{-3} of iridium(IV) and 7.1 µg cm^{-3} of palladium(II). The relative standard deviation for iridium(IV) for the second-derivative method was 1.2 %.

Comparison with Other Methods

Iridium can be determined spectrophotometrically at trace concentrations with a variety of reagents (Table IV).[23–28] Some methods are time-consuming because reactions develop at higher temperature[24,26,28] and are not selective

in the presence of rhodium(III), which is generally found to be associated with iridium.[23,24,28]

Compared with other spectrophotometric methods, the present approach is considerably less complicated, because the reaction takes place at room temperature. The proposed method is therefore time saving. Its other advantage is that iridium can be spectrophotometrically determined in the presence of a 25-fold excess of rhodium(III), without previous separation.

Application of the Method to Synthetic Mixtures

As no real samples were available to test the validity of the proposed method three synthetic mixtures with compositions corresponding to those of real samples were prepared. As shown in Table V, the results obtained were highly consistent with the amounts added, regardless of the reagent used in the organic phase.

TABLE IV. Comparison of the present method for iridium determination with some of the methods reported in literature

Reagent	Medium	Heating: T / °C (t / min)	ε dm^3 mol^{-1} cm^{-1}	Linear range γ / µg cm^{-3}	Tolerance limits $c(Rh^{3+})$ / $c(Ir^{n+})$	Ref.
Piperazine dimalonate (PDM)	Aqueous	No heating	9.9×10^3	0.0–24.0 (Ir^{IV})	10	23
1,5-Diphenyl carbazide (DFC)	CHCl$_3$	100 (45)	1.06×10^4	0.53–3.00 (Ir^{IV})	<1	24
Difurfuryl thiocarbohydrazone (DCE)	Ethyl acetate	No heating	4.15×10^4	0.93–3.73 (Ir^{III})	—	25
1-Phenyl-4,4,6-trimethyl-(1H,4H)-pyrimidine-2-thiolate (PTPT)	CHCl$_3$	60 (5)	3.38×10^3	3.8–42.0 (Ir^{III})	40	26
2-Mercapto-4-methyl-5--phenylazopyrimidine (MFAP)	CHCl$_3$	No heating	9.5×10^3	0.6–9.0 (Ir^{III})	Not available	27
Bis(thiopheno-2-aldehydo)--thiocarbohydrazone (BTATCH)	Ethyl acetate	100 (30)	3.2×10^4	1.2–4.2 (Ir^{III})	<1	28
3-Hydroxy-2-methyl-1-phenyl-4-pyridone (HX)	CHCl$_3$	No heating	5.32×10^3	1.9–38.4 (Ir^{IV})	25	Present method [a]
3-Hydroxy-2-methyl-1-(4-tolyl)-4-pyridone (HY)	CHCl$_3$	No heating	5.32×10^3	1.9–38.4 (Ir^{IV})	25	Present method [a]

[a] Extraction from chloride solution.

CONCLUSION

A relatively simple and inexpensive method for the extraction and spectrophotometric determination of iridium(IV) was developed. 3-Hydroxy-2-methyl-1-phenyl-4-pyridone and 3-hydroxy-2-methyl-1-(4-tolyl)-4-pyridone were used as reagents for the extraction of iridium(IV) from sulphuric and hydrochloric acid media and for the spectrophotometric determination of iridium(IV) in the organic phase. The important features of the method described here are: i) the reaction takes place at room temperature; ii) a low reagent concentration is required; iii) iridium(IV) determination is possible in the presence of a 25-fold excess of rhodium(III); iv) the method is applicable to the analysis of iridium(IV) in synthetic mixtures with compositions similar to those of alloys.

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SAŽETAK

Spektrofotometrijsko određivanje mikrokoličina iridija(IV) 1-fenil-3-hidroksi-2-metil-4-piridonom ili 3-hidroksi-2-metil-1-(4-tolil)-4-piridonom

Vinka Drušković i Vlasta Vojković

Opisana je ekstrakcijsko-spektrofotometrijska metoda za određivanje iridija(IV) 1-fenil-3-hidroksi-2-metil-4-piridonom (HX) ili 3-hidroksi-2-metil-1-(4-tolil)-4-piridonom (HY). Kompleks Ir^{IV}-HX(HY) ekstrahira se u kloroform, diklormetan, izobutil-metil-keton ili izoamilni alkohol, i to iz otopina zakiseljenih dodatkom H_2SO_4 ili HCl. Apsorbancija se odčitava pri 365 nm. Sustav slijedi Beerov zakon u području 1,9–38,5 μg cm^{-3} iridija(IV). Relativne standardne devijacije dobivene iz šest neovisnih određivanja uzoraka koji su sadržavali 3,84 i 15,36 μg cm^{-3} iridija(IV) iznosile su ±2 % i ±1,44 %. Razvijena je spektrofotometrijska metoda za istodobno određivanje mikrokoličina iridija(IV) u prisutnosti paladija(II) s pomoću HX otopljenog u kloroformu, koja se zasniva na drugoj derivaciji apsorpcijskih spektara. Predložena je metoda točna i precizna za simultano određivanje iridija(IV) (1,9–34 μg cm^{-3}) u prisutnosti paladija(II) (1–8 μg cm^{-3}). Metoda je primijenjena za određivanje analita u sintetskim smjesama.