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Solvent Extraction, Spectrophotometric determination of Iridium (III) using p-methylphenylthiourea as a chelating agent: Sequential Separation of Iridium(III), Ruthenium(III) and Platinum(IV)

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Abstract

A simple, sensitive and selective method for the solvent extraction and spectrophotometric determination of Ir(III) in various synthetic samples was reported. Ir(III) was extracted into chloroform in the form of its pink complex with p-methylphenyl thiourea (PMPT), and the absorption spectrum of the colored complex was acquired. Effects of acid concentration, reagent concentration and reagent concentration were examined to optimize sensitivity. The absorbance was linearly related to the concentration of Ir(III) in the ranges from 0 to 110 μgmL^{-1}, the correlation coefficient being 0.993. The molar absorptivity was calculated to be 0. 909×10^{3} L mol^{-1} cm^{-1} at 482 nm, whereas sandell's sensitivity was found as 0.21 μg cm^{-2}.Under the optimal conditions, the detection limit was 0.37 μgmL^{-1}. The proposed method was successfully applied for the sequential separation of iridium, ruthenium and platinum.

1. Introduction

The separation and determination of platinum group metals has attracted great interest due to increasing demand of these metals in the modern industries. Iridium is mostly found together with osmium in alloys like osmoiridium alloy, iridosmium alloy and neuynskite alloy. Due to its high density (22.56 g mL^{-1}) and higher melting point (2466°C) iridium has principle use as a hardening agent. Iridium finds applications in various fields like alloys for aircraft parts, pivots for surgical tools, dental alloy and electrical equipments. Iridium-192 is used in brachytherapy to treat various types of cancers [1,2]. It is also used industrially as a radiotracer and in gamma radiography to trace flaws in metal castings and welded joints. Literature survey reveals that many iridium complexes exhibits anti-tumeractivies [3–5].

Iridium and rhodium are used as a permanent chemical modifiers for determination of various metals by electrothermal AAS method[6–9]. Iridium (III) diimine complex shows electroluminescent properties which is used for the determination of tri-n-propylamine (TPA) and antibiotics [10] . Iridium-coated tungsten coil is used for the determination of bismuth in environmental samples by electrothermal vaporization AFS method [11]. Cyclometalated iridium complexes are used as micro-volume fiber-optic sensors for oxygen determination [12]. Determination of selenium content in cereals, bakery products and water samples is carried out by using iridium-treated graphite platforms by AAS [13,14]. Iridium and its compounds are used as a catalyst in various biological and chemical reactions [15–20].

Due to wide applications of iridium and its compounds in various fields, highly selective, sensitive, rapid and economical methods are needed for its trace and ultra trace determination. Various analytical techniques have been used for determination of iridium like controlled-potential coulometry [21,22] , glow discharge mass spectrometry [23], imprinted polymer-based method[24],radiochemical neutron activation analysis [25], adsorptive stripping voltammetry [26], negative thermal ionization mass spectrometry [27], double focusing magnetic sector field inductively coupled plasma mass spectrometry [28] . These are the advanced techniques but the high cost of instruments, high maintenance cost, and tedious experimental conditions limits the use of these methods in routine analysis.

Several extraction methods have been developed for extraction of iridium. Extraction of iridium(III) by ion-pair formation was reported [29] . A liquid-liquid extraction of iridium(IV) from chloride solution was studied under different condition of aqueous and organic phase concentration using alamine-336 as an extractant [30–32] . Solvent extraction of iridium(III) and iridium(IV) chloride complexes from hydrochloric acid and chloride solution by a sorbent MITKhAT containing sulfur and nitrogen was studied[33]. Extraction of iridium(IV) from hydrochloric acid media with crown ether in chloroform and its determination by ICP–AES was reported [34] .Co-extraction of platinum with iridium limits the applications of the method. Liquid-liquid extraction of Ir, Ru, and Rh from chloride solutions and their determination by ICP–AES technique were carried out [35].

Various reagents have been proposed for the spectrophotometric determination of iridium. Spectrophotometric determination of iridium has been reported using reagents like 3-hydroxy-2-methyl-1,4-naphthquinone-4-oxime[36] and 1,5-diphenylcarbazide[37] . 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) was described[38]. Extractive spectrophotometric determination of iridium(III) using 2-(5-bromo-2-oxoindolin-3-ylidene) hydrazine carbothioamide was reported[39] .

Catalytic effect of iridium on the oxidation of colored dyes or conversion of organic species into colored product forms the basis for spectrophotometric determination of iridium. Several reagents were reported for catalytic spectrophotometric determination of iridium. Three diantipyrylphenylmethane derivatives were oxidized by potassium periodate in hydrochloric acid media in presence of iridium[40]. Interference of ruthenium and heating at 90^{o}C were the demerits of the method. Iridium catalyzed oxidation of glycine by hexacyanoferrate (III) in aqueous alkaline medium method suffers from reaction time of 30 min and thermostating condition at 35^{o}C for 30 min.[41]. Solid-phase extraction of iridium from soil and water samples by using activated carbon cloth and spectrophotometric determination was reported[42]. Zn(II) and Cu(II) interferes in analysis.

In continuation of our research on development of extraction, spectrophotometric determination methods for determination of various metal ions viz. palladium(II)[43] , osmium(IV)[44] , selenium(IV)[45] and rhodium(III)[46] using o-methylphenyl thiourea as a chromogenic reagent and cerium(IV)[47] and osmium(VIII) and ruthenium (IV)[48] using o-methoxyphenyl thiourea as a reagent, we have synthesized the chelating reagent p-methylphentl thiourea and develop a selective extraction and spectrophotometric method for the determination of iridium(III). The present method describes quantitative extraction of iridium(III) at low acid and reagent concentration. In addition, proposed method also elaborates the sequential separation of iridium(III), ruthenium(III) and platinum(IV). The comparison between earlier methods and present method is elaborated (Table 1).

2. Material and Methods

2.1 Instrumentation

Absorbance measurements were carried out on Systronics double-beam UV-visible spectrophotometer (AU-2701) with 1 cm quartz cells. Contech (CA-123) electronic balance was used for weighing purpose. Calibrated glasswares were used and were cleaned by soaking in dilute nitric acid followed by washing with liquid soap and rinsed twice with distilled water. All reagents were of analytical reagent grade, and doubly distilled water was used throughout the experiments.

2.2 Reagents

A stock solution of iridium(III) was prepared by dissolving 1 g of iridium trichloride hydrate ($IrCl_3.H_2O$) (Loba. Chemie, Mumbai, India) in hydrochloric acid (1 mol L^{-1}) and standardized gravimetrically[49].A working standard solution of lower concentration was prepared by suitable dilution of the stock solution.

The chromogenic reagent p-methylphenyl thiourea was synthesized according to the reported method [50]. 0.01 mol L^{-1} reagent solution in ethanol was used for color development. The reagent solution remains stable for a week.

Standard solutions of different metal ions used to study the effect of diverse ions were prepared by dissolving weighed quantity of their respective salts either in double distilled water or in dilute hydrochloric acid. Solutions of anions were prepared by dissolving their respective alkali metal salts in double distilled water.

2.3 Recommended Method

An aliquot of solution containing 400 µg of iridium(III) and AR grade concentrated perchloric acid were transferred to 10 mL volumetric flask to maintain the acidity of 0.1 mol L^{-1} on dilution up to the mark with distilled water. A pink color Ir (III)-PMPT complex was formed immediately after addition 0.01 mol L^{-1} PMPT in 20 % (V/V) ethanol. Pink colored complex was equilibrated with 10 mL chloroform for 10 seconds in a 125 mL separatory funnel. Two layers were allowed to separate. The organic layer containing iridium(III)-PMPT complex was dried over 1.0 g anhydrous sodium sulphate and diluted up to the mark in 10 mL volumetric flask with chloroform. The absorbance of iridium(III)-PMPT complex was measured at 482 nm against the reagent blank.

Table 1: Comparison of spectrophotometric determination methods for iridium

Reagent	Aqueous phase	Solvent	λ max, nm	Beer's law range,µg mL^{-1}	ε, L mol^{-1} cm^{-1}	Remark	Ref.
1-Phenyl-4,4,6-trirnethyl-(1H,4H)-pyrimidine-2-thiolates	pH 5.3-8.0	chloroform	430	3.8-42	3.879x10^{3}	Heating at 60^{o}C for 5 min	[51]
Rhodamine 6G	2.5 mol L^{-1}HCl	di-isopropyl ether	530		3.6x10^{3}	Heating on boiling water bath for 10-15 min	[52]
MolybdateButylrhodamine B	1.3 mol L^{-1}	Water	570	0.0-0.1	8.75x10^{4}	Sb(II), Ge(IV), Si(IV) interfères	[53]
2-(5-iodine-2-pyridylazo)-5-dimethylaminoaniline	pH 5.6-7.3	Water	475	0.0-0.7	1.01x10^{5}	Narrow Beer's Range	[54]
Alanine+ Hexacynoferrate (III)	0.4 mol L^{-1}NaOH	Water	420	2.99-19.98	1.3x10^{3}	Thermostating at 35^{o}C for 35 min	[55]
Xylene cynol FF	0.02 mol L^{-1} H_2SO_4	Water	615	0.00028-0.004	NR	Heating at 95^{o}C	[56]
m-acetylchlorophosphonazo	--	Water	580	0.06-0.6	NR	Os (IV),Ru(III) interferes	[57]
Tin (II) chloride	2.0 mol L^{-1} HCl	Water	358	0.001-0.008	1.23x10^{4}	Narrow Beer's Range	[58]
1-(2-pyridylazo)-2-napthol	pH 3.5-5.5	DMF	543,565	0.5-15	NR	Heating at 90^{o}C	[59]
p-methylphenylthiourea	0.1mol L^{-1} $HClO_4$	Chloroform	482	upto 110	0.909x10^{3}	>72 hrs stability, Free from interference, sequential separation of Ir(III), Ru(III)&Pt(IV)	Present Method

NR: Not Reported

3. Results and discussion

3.1 Absorption Spectra

The absorption curves of the iridium(III)–PMPT complex, extracted into chloroform from 0.1 mol L^{-1} perchloric acid solution using a general procedure shows that the complex had maximum absorption at 482 nm (Figure 1). The appearance of a single absorption peak and constant nature of absorption curves for different concentrations of iridium(III) indicates the formation of only one complex under the experimental conditions. Since the reagent blank shows negligible absorption at 482 nm, it was confirmed that the extracted species was Ir(III)-PMPT complex. Hence 482 nm was fixed for further study.

3.2 Effect of acid concentration

Extraction of iridium(III) was studied using different mineral acids namely hydrochloric acid, hydrobromic acid, perchloric acid and nitric acid, in the concentration range of 0.02 to 1.0 mol L^{-1} using 0.01 mol L^{-1} reagent in ethyl alcohol as a complexing reagent. Pink colored iridium(III)-PMPT complex was formed in all mineral acids studied except nitric acid. Complete complexation of iridium(III)-PMPT with maximum absorbance was observed in the range of 0.1 to 1.0 mol L^{-1}perchloric acid media (Figure 2).

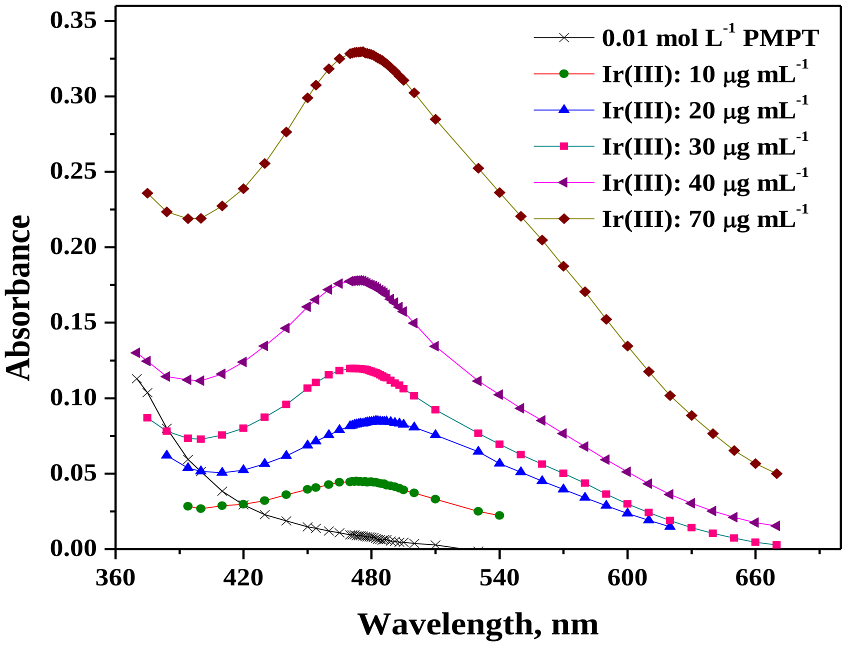


Figure 1: Absorption spectra of Ir(III)-PMPT complex

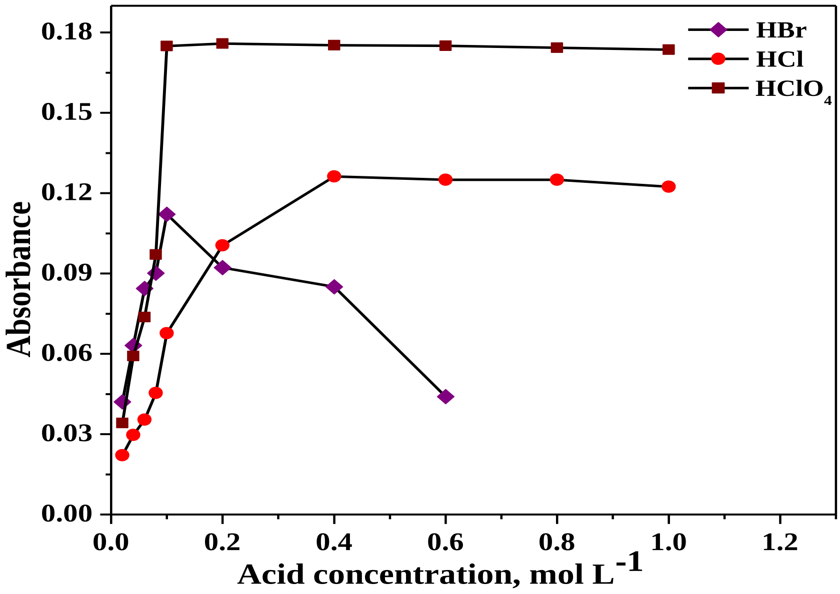


Figure 2: Effect of acid concentration on Ir(III)-PMPT complex formation

Therefore 0.1mol L^{-1} perchloric acid was fixed for further spectrophotometric study.

3.3 Effect of reagent (PMPT) concentration

To study the effect of reagent concentration, PMPT concentration was varied from 0.001 mol L^{-1} to 0.07 mol L^{-1} in ethyl alcohol. It was observed that the absorbance of iridium(III)-PMPT complex increases with increase in PMPT concentration from 0.001 mol L^{-1} to 0.01 mol L^{-1} then remains constant up to 0.05 mol L^{-1}. There was a small decrease in absorbance as reagent concentration further increase from 0.05 to 0.07 mol L^{-1} (Figure 3). Hence 0.01 mol L^{-1} PMPT in 20% (V/V) ethyl alcohol was used as the complexing reagent.

3.4 *Effect of reagent solvent concentration*

Iridium(III)-PMPT complex formation was investigated in ethyl alcohol in concentration range from 5 to 60 % (V/V). The pink colored Ir(III)-PMPT complex was formed in the range of 15 to 60 % (V/V) ethyl alcohol. To ensure complete complexation, 20% (V/V) ethyl alcohol was selected as the reagent solvent. (Figure 4).

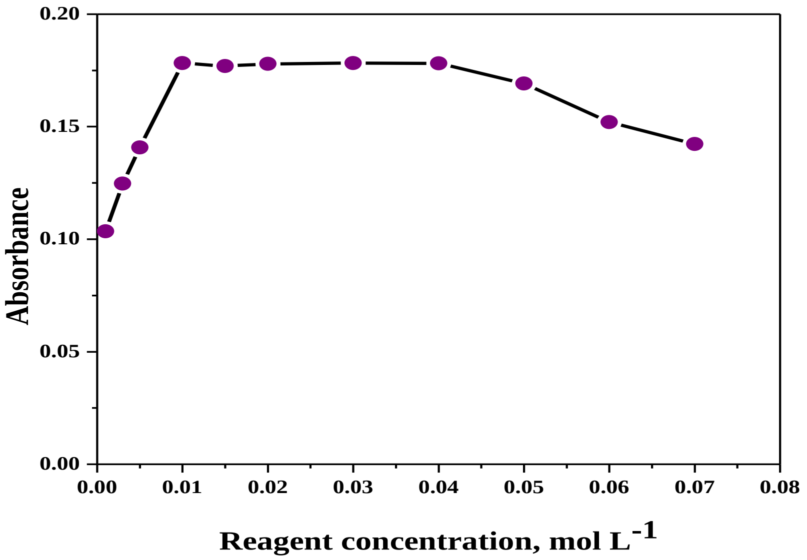


Figure 3: Effect PMPT concentration on Ir(III)-PMPT complex

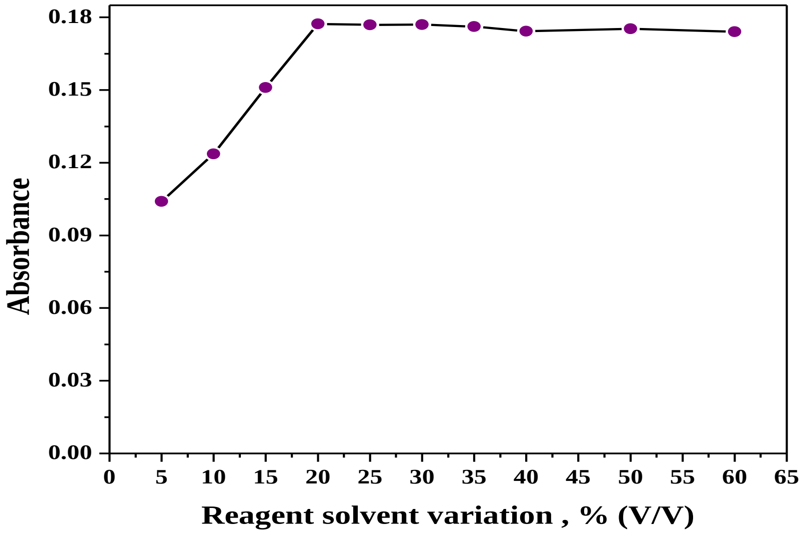


Figure 4: Effect of reagent solvent concentration on Ir(III)-PMPT complex

3.5 Effect of equilibration time and stability of the complex

The study of variation in absorbance with equilibration time was carried out over 3 seconds to 5 minutes. It was observed that extraction was completed in 10 seconds and there was no adverse effect of further equilibration on extraction of the iridium(III)-PMPT complex up to 5 minutes. Hence 10 second equilibration time was fixed. The stability of iridium(III)–PMPT complex was studied at room temperature by monitoring the absorbance value at regular time intervals of 1.0 h each and it was observed that the complex was stable for more than 72 hours.

3.6 Analytical figures of merit

The system obeyed Beer's law up to 110.0 µg mL^{-1} of iridium(III) at 482 nm (Figure 5).The molar absorptivity and Sandell's sensitivity were 0.909×10^{3} L mol^{-1} cm^{-1} and 0.21 µg cm^{-2}, respectively. The optimum range as defined by Ringbom's plot (Figure 6) was 40 to 110 µg mL^{-1}. The correlation coefficient value of iridium(III)-PMPT complex with an independent variable as concentration in µg mL^{-1} and a dependent variable as absorbance was found to be 0.993, indicating clear linearity between these variables. The slope and intercept for the best fitted lines were 0.0036 and 0.015 respectively. Therefore the content of iridium(III) in real samples can be determined using the straight line equation $y = 0.0036\ x + 0.015$. The analytical parameters obtained were summarized in Table 2.

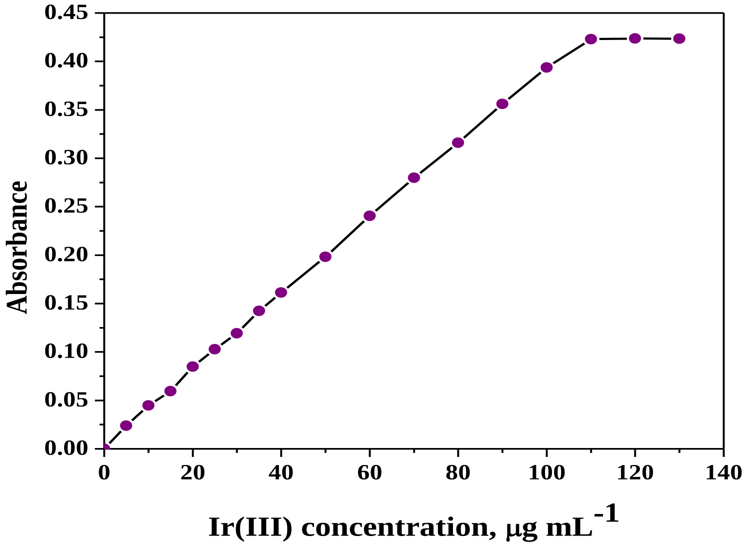


Figure 5: Beer's law range for Ir(III)-PMPT complex

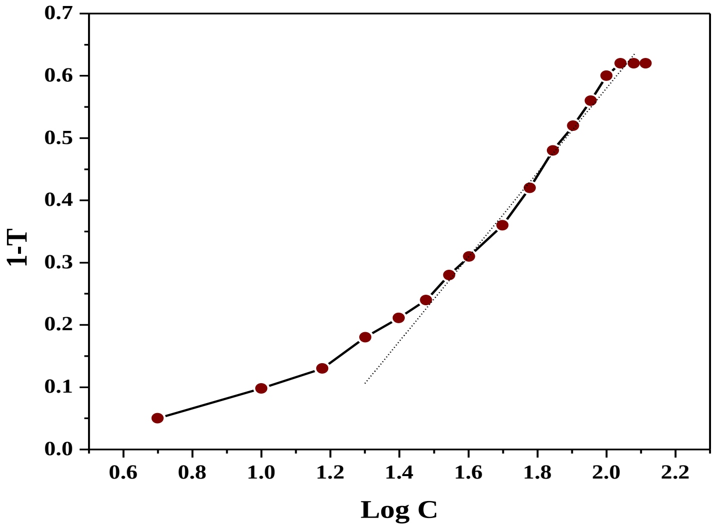


Figure 6: Ringbom's plot for Ir(III)-PMPT complex

Table 2: Spectral and physico-chemical characteristics of iridium (III) - PMPT complex

Parameter	Specification
Perchloric acid concentration	0.1 mol L^{-1}
Extraction solvent	Chloroform
Reagent concentration	0.01 mol L^{-1}
Equilibration time	10 seconds
λmax	482 nm
Molar absorptivity	0.909 x 10^3 L mol^{-1} cm^{-1}
Sandell's sensitivity	0.21μg cm^{-2}
Beer's law range	up to 110 μg mL^{-1}
Ringbom's optimum range	40.0 to 110 μg mL^{-1}
Correlation coefficient	0.993
Relative standard deviation	0.46 %
Stoichiometry of the complex	1:1 (Ir(VIII):PMPT)
Stability of complex	>72 hr.
Limit of detection(LOD)	0.37 μg mL^{-1}

3.7 Stoichiometry of complex

An attempt was made to ascertain the nature of the extracted complex species using log D - log C plot, job's method of continuous variation and the mole ratio method. The graph of log $D_{[Ir(III)]}$ against log $C_{[PMPT]}$ at 0.1 mol L^{-1} perchloric acid concentration was found to be linear and having slope value 1.10 (Figure 7).

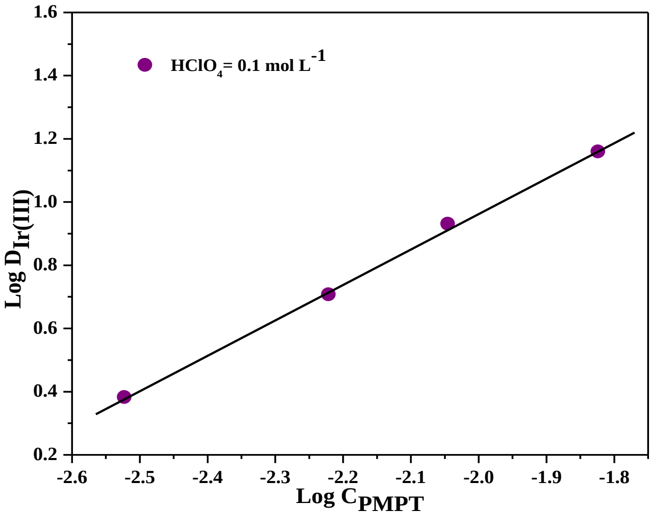


Figure 7: Plot of log $D_{Ir(III)}$ Vs log C_{PMPT} for Ir(III)-PMPT complex

Hence, the probable composition of the iridium(III):PMPT complex was 1:1. This composition was confirmed by the mole ratio method (Figure 8) and job's continuous variation method (Figure 9).

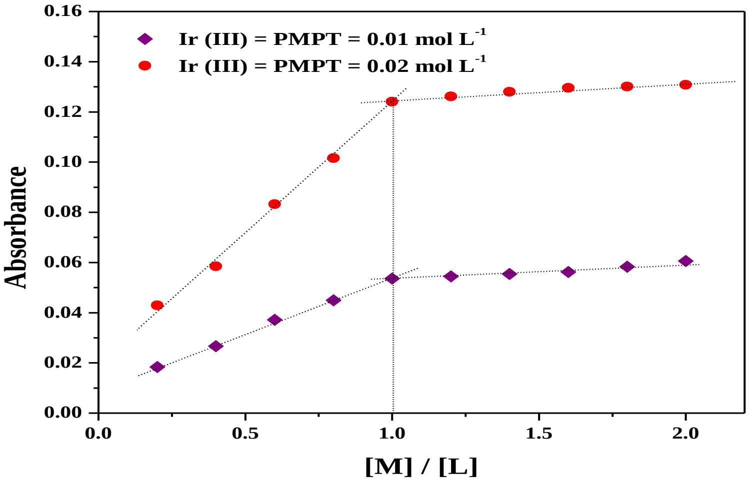


Figure 8: Mole ratio method for Ir(III)-PMPT complex

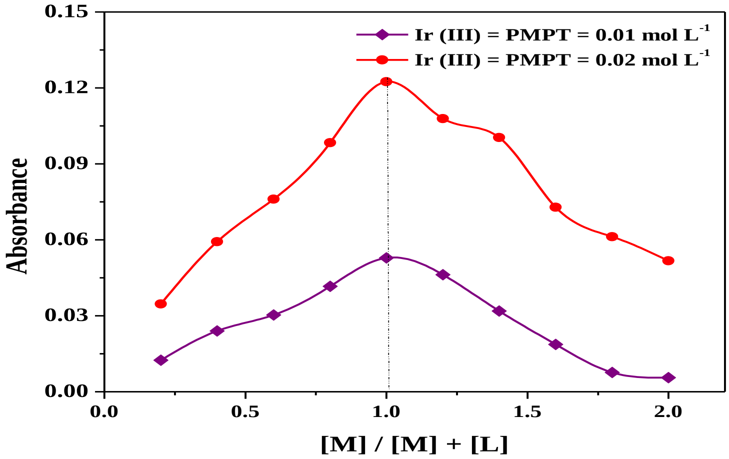


Figure 9: Job's continuous variation method for Ir(III)-PMPT complex

PMPT acts as a bidentate ligand, sulfur atom from thio group (-C=S) and nitrogen from amine group ($-NH_2$) coordinate with iridium(III) to form a 1:1 (iridium (III): PMPT) complex.

3.8 Interference Study

The effect of foreign ions on quantitative extraction of iridium(III) was studied. Variable amounts of ions were added to 40 µg mL^{-1} of iridium(III) in order to find the tolerance limits of these ions. Initially, the ions were added to iridium solution in large excess. When interference found to be intensive, the tests were repeated with successively smaller amounts. (An error of ± 2% in the absorbance values was considered to be tolerable). The interference of palladium(II) was removed by prior extraction from 0.05 mol L^{-1} hydrobromic acid media. Interference of mangenese(II) and copper(II) was eliminated by masking it with 100 mg EDTA.. Thus, the developed method was found to be free from interferences from large number of foreign ions (Table 3).

Table 3: Effect of foreign ions on extraction of Ir (III)-PMPT complex

Foreign ion	Added as	Tolerance limit, mg	Foreign ion	Added as	Tolerance limit,mg
As(III)	As_2O_3	7.00	Mn (II)[a]	$MnCl_2.6H_2O$	1.00
Al(III)	$AlCl_3.6H_2O$	0.75	Au (III)	$HAuClO_4$. H_2O	0.80
W (VI)	$Na_2WO_4.2$ H_2O	3.00	Fe (III)	$(NH_4)Fe(SO_4)_2.12H_2O$	0.60
Sn (II)	$SnCl_2.2$ H_2O	25.0	Cu (II)[a]	$CuSO_4.5$ H_2O	0.75
Li (I)	LiCl	35.0	Ca (II)	$CaCl_2.2$ H_2O	6.00
Co (II)	$CoCl_2.6$ H_2O	0.50	Pd (II)[b]	$PdCl_2$	2.2
Ni (II)	$NiCl_2.6$ H_2O	0.70	Rh (III)	$RhCl_3$	1.56
Pb (II)	$PbCl_2$	2.00	Ru (III)	$RuCl_3.6$ H_2O	1.60
Mg (II)	$MgCl_2.6$ H_2O	10.0	In (III)	$InCl_3.4$ H_2O	1.00
Cr (III)	$CrCl_3$	0.80	Ba (II)	$BaCl_2.6$ H_2O	40.0
Zn (II)	$ZnSO_4.7$ H_2O	8.00	Ga (III)	$GaCl_3$	4.50
Zr (II)	$ZrOCl_2.8H_2O$	6.00	Hg (II)	$HgCl_2$	10.0
Ag(I)	$AgNO_3$	1.00	Ti (III)	$(Ti_2SO_4)_3$	1.00
Cd (II)	$CdCl_2.2$ H_2O	8.00	Mo (VI)	$(NH_4)_5MO_7.2$ H_2O	1.00
La (III)	$LaCl_3.7$ H_2O	3.00	EDTA	Na_2EDTA	100
Se (IV)	SeO_2	9.60	Sulphate	K_2SO_4	100
Ce (IV)	$Ce(SO_4)_2.4$ H_2O	1.00	Succinate	$(CH_3COONa)_2.6$ H_2O	100
Fe (II)	$(NH_4)Fe(SO_4)_2$.12H2O	0.80	Acetate	$CH_3COONa.3H_2O$	100
Tl (III)	Tl_2O_3	0.50	Tartrate	$(CHOH:COOH)_2$	100
U (VI)	$UO_2(CH_3COO)_2$	9.00	Fluoride	NaF	50.0
Sr (III)	$SrCl_3.6$ H_2O	8.00	Bromide	KBr	100
V (V)	V_2O_5	5.00	Malonate	$(COOH)_2.2$ H_2O	100
Bi (III)	$BiCl_3$	2.50	Citrate	$C_6H_8O_7$. H_2O	100

a: masked with 100 mg EDTA b: prior extraction

3.9 Precision, accuracy and detection limit

To check the reproducibility and accuracy of the method, absorbance of ten identical sample solutions containing 400 µg was measured by proposed method. The standard deviation and relative standard deviation for these ten measurements were determined which were found to be 0.00086 and 0.46 % respectively. These results indicate that the developed method is accurate and precise. The limit of detection (LOD), determined as amount corresponding to thrice the standard deviation blank value for iridium(III) by recommended method was found to be 0.37 µg mL^{-1}.

4. Applications

4.1 Separation and determination of iridium(III) from binary synthetic mixtures

The method permits the separation and determination of iridium(III) from binary mixtures containing tungsten(VI),copper(II), lead(II), nickel(II), magnesium(II), zinc(II) ,mercury(II) and cobalt(II). After quantitative separation of iridium(III) by proposed method, the aqueous phase was evaporated to dryness followed by 3.0 mL concentrated hydrochloric acid. The residue obtained was cooled, dissolved in water and the added metal ions were determined by standard methods[60] (Table 4).

Table 4: Separation and determination of Iridium (III) from binary synthetic mixtures.

Metal ion	Amount taken (µg)	Recovery ^{a} (%)	RSD (%)	Chromogenic reagent	Reference
Ir (III)	400	99.4	0.58	PMPT	PM
Mg(II)	100	99.8	0.24	permanganate	[60]
Ir (III)	400	99.4	0.73	PMPT	PM
Ni(II)	70	99.8	0.16	DMG	[60]
Ir (III)	400	99.1	0.47	PMPT	PM
W(VI)	100	99.4	0.38	thiocyanate	[60]
Ir (III)	400	99.6	0.21	PMPT	PM
Pb(II)	50	99.9	0.19	dithiozone	[60]
Ir (III)	400	99.4	0.73	PMPT	PM
Zn(II)	50	99.7	0.10	dithiozone	[60]
Ir (III)	400	99.5	0.31	PMPT	PM
Co(II)	70	99.2	0.14	thiocyanate	[60]
Ir (III)	400	99.8	0.31	PMPT	PM
Cu(II)^b	30	99.1	0.29	dithiozone	[60]
Ir (III)	400	99.7	0.43	PMPT	PM
Hg(II)	40	99.4	0.49	dithiozone	[60]

a: average of five determinations b: masked with 100 mg EDTA

4.2 Separation of iridium (III) from multicomponent synthetic mixtures

The proposed method allows the selective separation and determination of iridium(III) from different metal ions. Various multicomponent synthetic mixtures containing varying proportions of associated metal ions and fixed amount of iridium(III) (400 µg) were prepared. Iridium(III) was separated and determined by the recommended method. The results give quantitative recovery of iridium(III) in presence of associated metal ions. (Table 5)

Table 5: Separation and determination of Iridium (III) from multicomponent synthetic mixtures.

Ternary mixture composition (µg)	Recovery ^{a} (%)	RSD (%)
Ir (III) 400;Ni(II) 70; W(IV) 100; Pb(II) 50; Zn(II) 50	99.7	0.42
Ir (III) 400;Mg(II) 100; Co(II) 70; Cu(II)^b 30; Hg(II) 40	99.6	0.47
Ir (III) 400; Ni(II) 70; W(IV) 100; Cu(II)^b 30; Hg(II) 40	99.6	0.63
Ir (III) 400; Pb(II) 50; Co(II) 70; Zn(II) 50; Mg(II) 100	99.2	0.54
Ir (III) 400; Ni(II) 70; Pb(II) 50; Zn(II) 50; Hg(II) 40	99.5	0.61

a: average of five determinations b: masked with 100 mg EDTA

4.3 Sequential separation of Iridium(III), Ruthenium(III) and Platinum(IV)

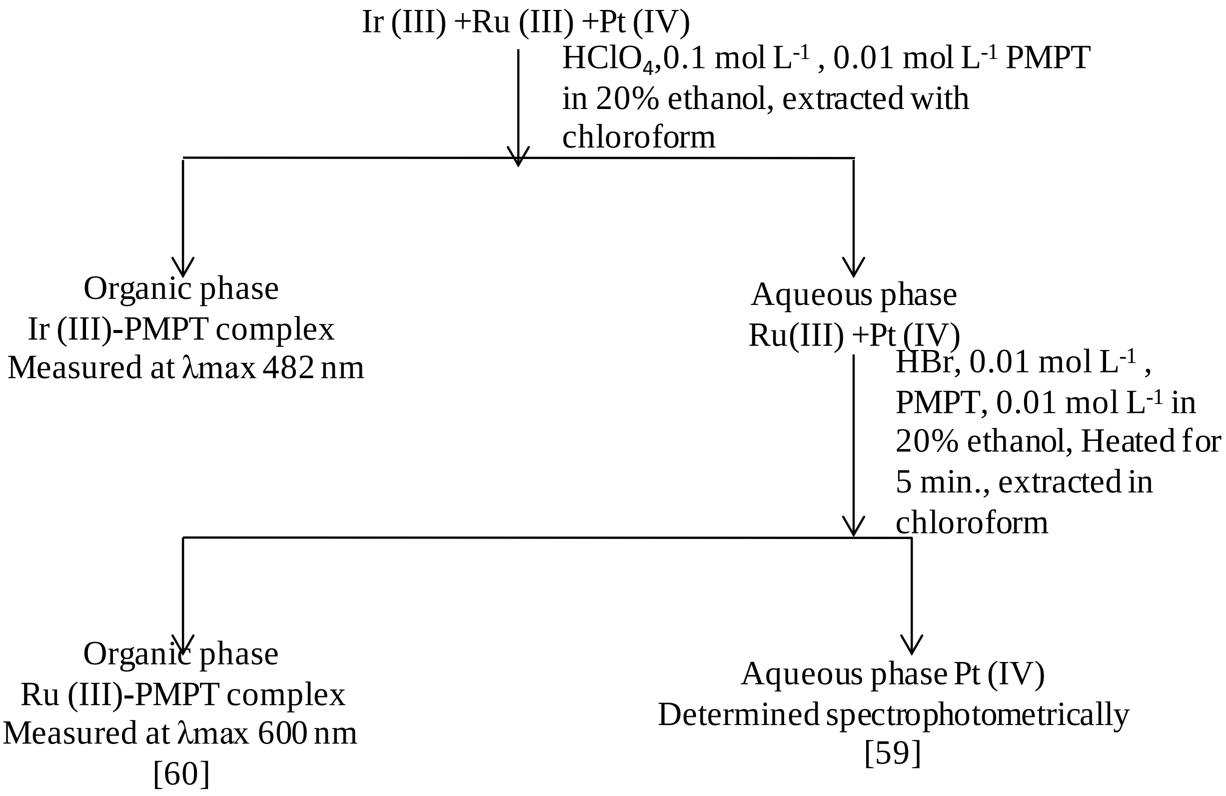
The selectivity of proposed method was verified by applying it to the separation and determination of iridium(III), ruthenium(III) and platinum(IV) from synthetic mixtures. The separation of these metal ions was resolved by taking advantage of different acid concentrations (Scheme 1). Iridium(III) was extracted in chloroform from a mixture using 0.01 mol L^{-1} PMPT from 0.1 mol L^{-1}perchloric acid media. Aqueous solution containing ruthenium(III) and platinum(IV) was evaporated to dryness; residue was dissolved in distilled water. Aqueous phase was then made 0.01 mol L^{-1} by addition of hydrobromic acid; followed by addition of 0.01 mol L^{-1} PMPT in 20 %(V/V) ethyl alcohol. Resulting solution was then heated on water bath for 5 min. Bluish green colored Ru(III)-PMPT complex was formed which was cooled and equilibrated for 5 seconds with chloroform. The absorbance of the chloroform layer was measured at 600 nm[61]. The raffinate containing platinum(IV) was evaporated to dryness, cooled, dissolved in 10 mL of distilled water and platinum(IV) was determined spectrophotometrically[60] (Table 6).

Table 6: Sequential separation of Iridium (III) Ruthenium (III) and Platinum (IV)

Mixture	Amount taken (µg)	Chromogenic ligand	Recovery [a]	RSD %
Ir (III)+ Ru (III)+Pt (IV)	Ir (300) Ru (50) Pt (40)	PMPT PMPT $SnCl_2$	99.1 99.5 99.7	0.21 0.34 0.11
Ir (III)+ Ru (III)+Pt (IV)	Ir (400) Ru (50) Pt (40)	PMPT PMPT $SnCl_2$	99.2 99.3 99.7	0.24 1.43 0.16
Ir (III)+ Ru (III)+Pt (IV)	Ir (500) Ru (50) Pt (40)	PMPT PMPT $SnCl_2$	99.3 99.4 99.7	0.89 0.31 0.22

a: average of five determinations

Scheme 1: Sequential Separation of iridium(III),ruthenium(III) and platinum(IV)



Conclusion

The chromogenic reagent reported in present work is found to be sensitive and selective for the solvent extraction and spectrophotometric determination of iridium. Literature survey shows that varieties of organic reagents are reported for the spectrophotometric determination of iridium. Comparison of the proposed method with reported methods shows that has relatively high sensitivity, selectivity, low acidic condition, no need of use of buffer solution and heating, quantitative extraction with short equilibration time. Proposed work has following merits

- Method is sensitive and selective for determination of iridium(III).
- Low reagent concentration required.(0.01 mol L^{-1}) ,wide Beer's Range (upto 110 µg mL^{-1}), good stability (>72 hrs), lower acid concentration for full color development (0.1 mol L^{-1})
- Pink color complex formed and extracted into chloroform within 10 seconds.
- No need of multiple extractions.
- 0.37 µg mL^{-1} of iridium can be successfully determined with high accuracy.
- The numerical values of relative standard deviation (0.46%), correlation coefficient (0.99) proves the accuracy and reproducibility of method.
- Method is successfully applied for separation and determination of iridium (III) from binary and multicomponent mixtures.
- The applicability of the method is improved by development of Sequential separation and quantitative determination of iridium(III),ruthenium(III) and platinum(IV).

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