

Extractive spectrophotometric determination of palladium(II) with *o*-methyl phenyl thiourea from synthetic mixtures

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A novel method is proposed for the solvent extraction spectrophotometric determination of palladium(II) using low concentration of *o*-methylphenyl thiourea (OMPT). OMPT in chloroform quantitatively extracts trace concentration of palladium(II) at 0.8 mol dm⁻³ hydrochloric acid media which requires only 10 s equilibration time, the absorbance of yellow coloured palladium(II)-OMPT complex is measured at 340 nm. Complex is stable for more than 70 h. The composition of extracted species is 1:1, determined by mole ratio, job's continuous variation method and it is confirmed by log-log plot. Beer's law is obeyed in the range of 0.01 µg cm⁻³ to 15.0 µg cm⁻³. The molar absorptivity and sandell's sensitivity are 2.85 × 10³ dm³ mole⁻¹ cm⁻¹ and 0.037 µg cm⁻². Method is free from large number of interferences from cations and anions. The method is applied for separation of palladium(II) from multicomponent mixtures and hydrogenation catalyst.

Keywords: Palladium(II), *o*-methylphenyl thiourea, extractive spectrophotometry.

1. INTRODUCTION

Palladium(II) and its alloys have a wide range of applications in chemical industries. With the increasing use of palladium(II) in jewellery and cosmetic dentistry in the form of alloys [1, 2], the need arose for development of selective, sensitive, simple, rapid method for quantitative separation and determination of palladium(II). Several analytical techniques viz. AAS, ICP-MS, X-ray fluorescence and spectrophotometric methods are available for determination of palladium(II). Amongst the methods available for determination of palladium(II) the spectrophotometric methods are preferred because these are cheaper, easy to handle, requires less time and with desired accuracy.

2-hydroxy-1-naphthelene carboxaldehyde hydrazine carboxamide [3] has been used for spectrophotometric determination of palladium(II). Beer's law obeyed over the range of 0.55 µg cm⁻³ to 2.50 µg cm⁻³. A red colored complex of palladium(II) with PAR [4] is formed at pH 9.0 to 11.0 and quantitatively extracted into molten naphthalene. This method has interferences from cobalt(III), iron(II) and bismuth(III) and was removed by masking with EDTA. 1,3-

Bis(hydroxymethyl)benzimidazole-2-thione [5] was used for extractive spectrophotometric determination of palladium(II) from hydrogenation catalysts. palladium(II) complexed with 1-(2-pyridylazo)-2-naphthol (PAN) [6] in aqueous solution was extracted into chloroform. Components of nonferrous alloys interfere in determination of palladium (II). 3-phenoxybenzaldoxime [7] was used for extractive photometric determination of palladium(II) and method was applied for determination of palladium(II) from catalysts and synthetic mixtures. Hexane 2-5-dione-bis-(ethylenediamine) [8] forms pink colored complex with palladium(II), was quantitatively extracted at pH 3.5. Extractive spectrophotometric determination method was developed for palladium(II) using 2-hydroxy-3-nitro-5-methyl acetophenone oxime [9]. Palladium(II) was quantitatively extracted from pH 0.0-4.0. The proposed method for extraction spectrophotometric determination of palladium(II) using OMPT as a new reagent when compared with other extractive spectrophotometric methods it is found to be more sensitive and selective [10-25] (Table 1).

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Table 1. Comparison of present method with other extractive spectrophotometric determination methods of palladium(II)

Reagents	Absorbance / nm	Acidity / pH	Beer's Law validity (range/µm)	Solvent	Molar Absorbivity (L/Mol cm)	M:L	Remark	Ref.No
di-2-pyridyl ketone benzoylhydrazone(DPKBH)	455	H ₂ SO ₄ media	0-0.01	benzene	9.38x10 ⁴	-----	Low beer's range	10
Pomissium, O,O'-Diethylthiophosphate(Sdtp)	298	1-13	0-3.4	chloroform	2.9x10 ⁴	1:2	Alternate shaking and standing 20 min	11
2,2'-Bipyridyl 2-Pyridylhydrazones(DPPH)	585	0.5-1.5	NM	chloroform	1.9x10 ⁴	1:1	10 min shaking	12
4-(3,5-Dichloro-2-pyridylazo)-1,3-diaminobenzene(3,5-Cl ₂ PADAB)	600	1-2 M H ₂ SO ₄	0.24-2.16	benzene	8.6x10 ⁴	NM	Low beer's range, 15 min shaking	13
2-(5-methyl-2-pyridilazo)-5-diethylaminophenol	553	0.1M H ₂ SO ₄	0.008-0.32	toluene	-----	-----	Low beer's range, Au(III), Pt(II), V(V), W(V), interfere	14
3-(4-dihydro-4,4,6-trimethyl-2(1H)-Pyrimidin-2-Thione(DTPP)	420	5.5	0.4-24.6	chloroform	3.90 x10 ⁴	1:2	CN ⁻ interfere	15
3,4,5-Trimethoxybenzaldehyde Thiosemicarbazone(TBTSO)	370	0.9M HCl	0-12	chloroform	8.35x10 ⁴	1:2	Twice extraction by 3 ml chloroform each	16
2,2'-dithionine (DTDA)	397	3.0	0.003-0.2	BMCK	1.47x10 ⁴	1:1	Low beer's range	17
3-Hydroxy-2-methyl-1-phenyl-4-pyridone(HX)	345	2.5	0.28-8.0	chloroform	1.89 x10 ⁴	1:2	35 min shaking	18
2-arythio-p-witracetophenone	730	7-8M Acetic acid	2.5-20	chloroform	1.61x10 ⁴	-----	Higher Acidity, 15 min shaking	19
Pyridoxal-4-phenyl-3-thiosemicarbazone(PPT)	460	3.0	0.4-6.4	benzene	2.2 x10 ⁴	1:1	Twice extraction by 10 ml chloroform each, interfering cations and anions masked	20
4-(2'-Furaldiminamino)-3-Methyl-5-Mercapto-1,2,4-triazole(TMAMT)	410	5.4	5-50	n-butanol	1.4x10 ⁴	1:1	Many interfering ions masked	21
Benzoyloxybenzylidene thiosemicarbazone(BBTSO)	365	5.0	5-60	cyclohexanol	4.0 x10 ⁴	1:1	Simple and sensitive	22
2-arythio-5-methyl-4-ethoxybenzoylthiosemicarbazone(BMALNH)	385	0.01-0.015M H ₂ SO ₄	2.0-9.0	chloroform	5.32 x10 ⁴	1:1	Few diverse ions studied, No application of the method	23
Monothiobenzoylthiosemicarbazone(3-mercapto-1,3-diphenyl-2-propen-1-one)(HSDBM)	470	3.5-6.5	0.6-3.6	benzene	NM	1:2	Low beer's range, 10 min shaking, interfering ions masked	24
Thioureacler's ketone(TMK)	518	2.5 x10 ⁻² M (C ₆ H ₅) ₂	0.002-0.1	chloroform	NM	NM	3 min homogeneous centrifugal extraction 4000 rpm, low beer's range	25
O-Methyl phenyl thiourea(OMP)	340	0.8M HCL	0-12.5	chloroform	2.85 x10 ⁴	1:1	Selective and sensitive	PM

NM : not mentioned, PM : present method.

2. EXPERIMENTAL

Instrumentation

A digital spectrophotometer model EL-159 [Elico Make] with matched 10mm quartz cells was used for absorbance measurements.

Reagents

All of the reagents used were of analytical reagent grade unless otherwise stated. A standard stock solution of palladium (II) has been prepared by dissolving 1.0 g palladium (II) chloride (PdCl₂)(Loba.Chem.) in 1.0 moldm⁻³ hydrochloric acid and diluted to 250 cm³ in a calibrated flask with water and was standardized by gravimetric method [27]. A working standard solution of palladium(II) 60 µgcm⁻³ was prepared by diluting the standard stock solution with water. *O*-methyl phenyl thiourea [OMPT] has been prepared using method reported by Frank and Smith [28]. The working reagent solution (1.0 x 10⁻⁴ moldm⁻³) of OMPT was prepared in chloroform. Other standard solutions of different metal ions were prepared by dissolving their respective salts in water and diluted suitably. Double distilled water was used throughout the work.

Recommended procedure

To an aliquot of solution containing 60 µg Pd(II) in a 25 cm³ calibrated flask, enough hydrochloric acid and water were added to adjust acidity 0.8 moldm⁻³ with respect to hydrochloric acid and diluted up to mark with distilled water. The aqueous solution was equilibrated with 10 cm³, 1.0 x 10⁻⁴ moldm⁻³ *o*-methyl phenyl thiourea [OMPT] in chloroform for 10 s, in a 125 cm³ separatory funnel. These two phases were allowed to separate and yellow colored palladium (II)-OMPT complex in organic phase was dried over anhydrous sodium sulphate. The total volume of organic phase was made 10 cm³ and absorbance of palladium(II)-OMPT complex in organic phase was measured at 340 nm against the reagent blank.

3. RESULTS AND DISCUSSION

Absorption Spectra

The palladium(II)-OMPT complex in chloroform shows the absorption maxima at 340 nm, whereas absorption spectrum due to reagent blank is negligible (Fig. 1) therefore, all the absorbance measurements were made at 340 nm against reagent blank for further spectrophotometric determination of palladium(II)

Effect of acidity

The extraction of palladium(II) was studied using different mineral acid media [hydrochloric, sulphuric, nitric and perchloric acid] using

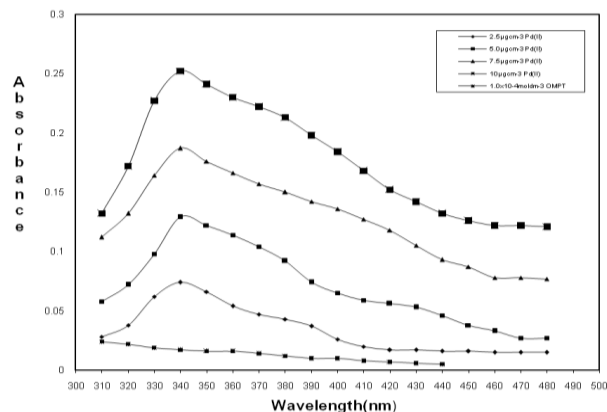


Fig 1. Absorption spectra of Pd(II)-OMPT Complex vs. OMPT reagent blank. Pd (II)-2.5 µg cm⁻³, 5.0 µg cm⁻³, 7.5 µg cm⁻³, 10.0 µg cm⁻³; OMPT- 1.0x10⁻⁴ mol dm⁻³; hydrochloric acid - 0.8 moldm⁻³, shaking time 10s.

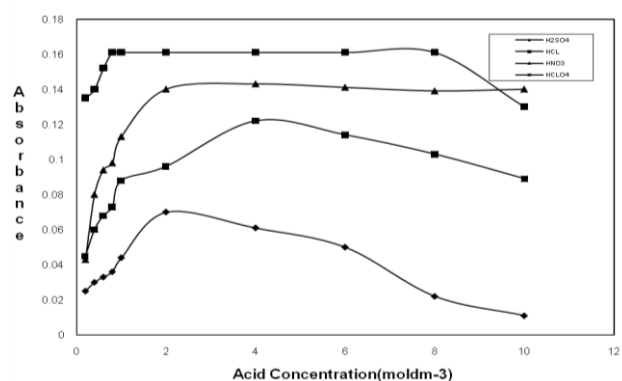


Fig 2. Effect of acidity on extraction of Pd(II)-OMPT complex. Pd(II)-6.0 µg cm⁻³, OMPT 1.0 x 10⁻⁴ mol dm⁻³, λ_{max}, 340 nm, shaking time 10s.

1.0 x 10⁻⁴ mol dm⁻³ reagent (OMPT) in chloroform, in a range of 0.1 to 10.0 mol dm⁻³ acid concentrations. Complexation was observed in all acids studied but complete complexation of palladium(II)-OMPT complex with maximum absorbance was observed in the range 0.8 – 8.0 mol dm⁻³ hydrochloric acid media (Fig. 2) Therefore 0.8 mol dm⁻³ hydrochloric acid concentration was used for further extraction study.

Choice of solvent

Toluene, xylene, benzene, n-hexane, n-butanol, n-butylacetate, chloroform were tried for the extraction of palladium(II)-OMPT complex (Fig.3). Amongst the solvents employed for solvent extraction spectrophotometric determination of

palladium(II) chloroform was selected with maximum extraction effectively.

Effect of reagent concentration

The effect of reagent concentration was studied by varying OMPT concentration from 1.0×10^{-5} mol dm^{-3} to 1.0×10^{-3} mol dm^{-3} in chloroform. It was observed that the absorption of palladium (II)-

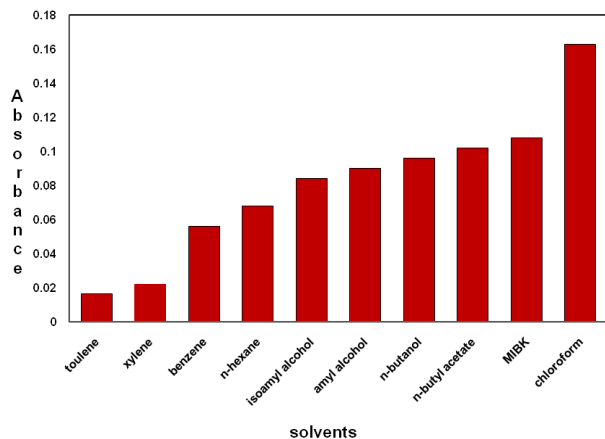


Fig 3. Effect of various solvents on extraction of Pd(II)-OMPT complex. Pd(II) $6.0 \mu\text{gcm}^{-3}$; OMPT- 1.0×10^{-4} mol dm^{-3} , hydrochloric acid 0.8 mol dm^{-3} , $\lambda_{\text{max}} = 340$ nm, shaking time 10s.

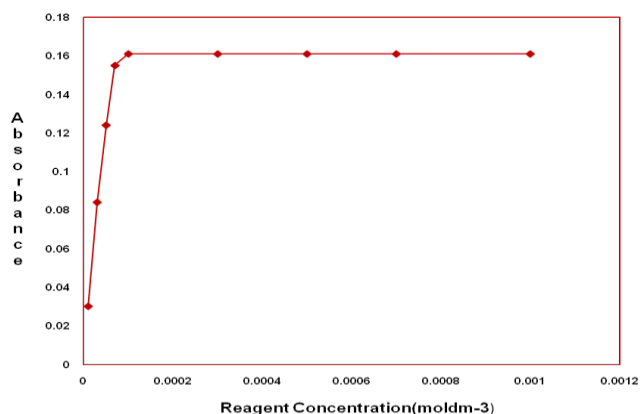


Fig 4. Effect of reagent concentration on extraction of Pd(II)-OMPT complex. Pd(II) $6.0 \mu\text{g cm}^{-3}$; OMPT 1.0×10^{-5} to 1.0×10^{-3} mol dm^{-3} , hydrochloric acid 0.8 mol dm^{-3} , shaking time 10s.

OMPT complex increases with increase in concentration of reagent from 1.0×10^{-5} mol dm^{-3} to 1.0×10^{-4} mol dm^{-3} after this range it become constant with no any adverse effect of excess reagent concentration on absorbance of palladium(II)-OMPT complex (Fig. 4).

Effect of equilibration time and stability of complex

The study of change in absorbance with variation in equilibration time was carried out over 5 s to 30 min. It has been observed that extraction was completed in 5s and there was no any adverse

effect of prolonged equilibration on extraction of palladium(II)-OMPT complex up to 30 min. Hence 10s equilibration time was fixed for further study, also absorbance of Pd(II)-OMPT complex was stable for more than 70h.

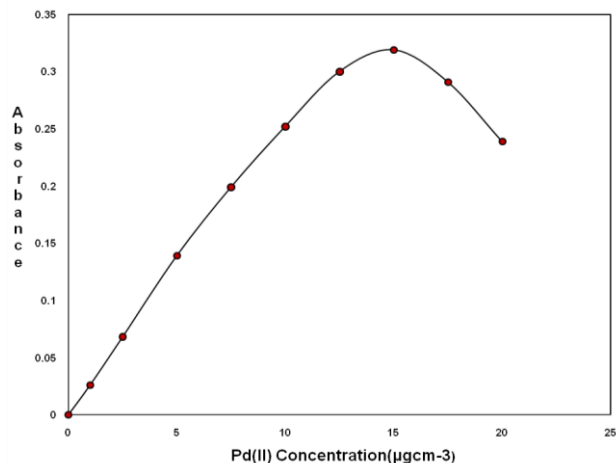


Fig 5. Applicability of Beer's law to Pd(II)-OMPT complex. Pd(II) 0.01 to $20.0 \mu\text{g cm}^{-3}$; OMPT- 1.0×10^{-4} mol dm^{-3} , hydrochloric acid 0.8 mol dm^{-3} , $\lambda_{\text{max}} = 340$ nm, shaking time 10s.

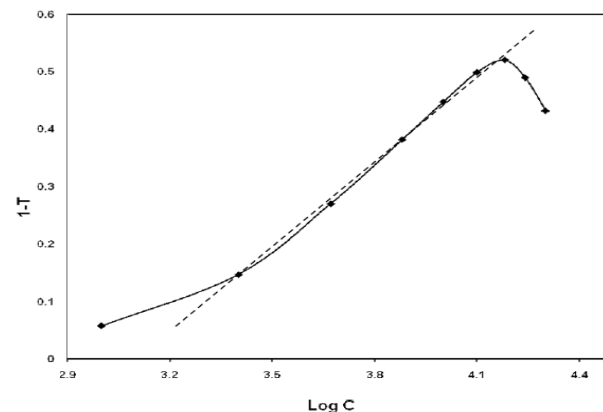


Fig 6. Ringbom's plot for Pd(II)-OMPT complex. Pd(II) 1000 to $22500 \mu\text{g dm}^{-3}$, OMPT 1.0×10^{-4} mol dm^{-3} , hydrochloric acid 0.8 mol dm^{-3} , $\lambda_{\text{max}} = 340$ nm, shaking time 10s.

Beer's law, molar absorptivity, sandell's sensitivity and correlation coefficient

Beer's law is obeyed over the concentration range of $0.01 \mu\text{gcm}^{-3}$ to $15.0 \mu\text{gcm}^{-3}$ for palladium(II)-OMPT complex at 340 nm (Fig. 5) The molar absorptivity and sandell's sensitivity are $2.85 \times 10^3 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and $0.037 \mu\text{gcm}^{-2}$ respectively. The optimum range as defined by Ringbom's [26] plot (Fig. 6) is 3.1 to $13.0 \mu\text{gcm}^{-3}$, slope of Ringbom's plot from fig.6 is 0.4655 . Hence, the ratio between the relative error in

concentration and photometric error is $p = 4.655$, and for one percent photometric error, $p = 0.01$, hence the relative error in concentration is 0.04655. The correlation coefficient value of Pd(II)-OMPT complex with an independent variable as concentration in $\mu\text{g cm}^{-3}$ and a dependent variable as absorbance, was found to be 0.99, indicates a clear linearity between these variables. The slope value

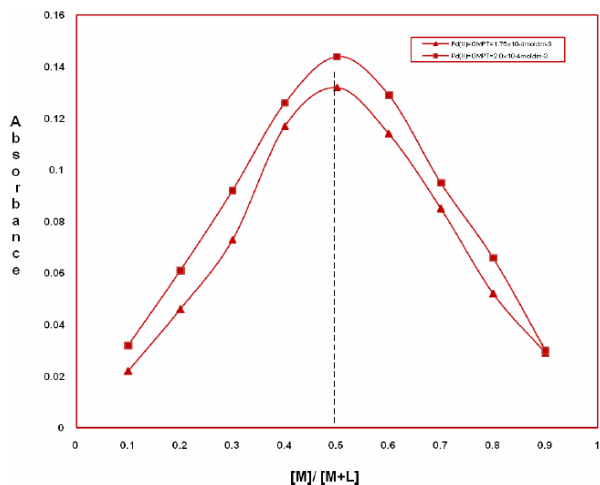


Fig 7. Pd(II):OMPT species by job's variation method. Pd(II)=OMPT= 1.75×10^{-4} (\blacktriangle) and 2.0×10^{-4} (\blacksquare) mol dm^{-3} , hydrochloric acid- 0.8 mol dm^{-3} , $\lambda_{\text{max}} = 340 \text{ nm}$, shaking time 10s.

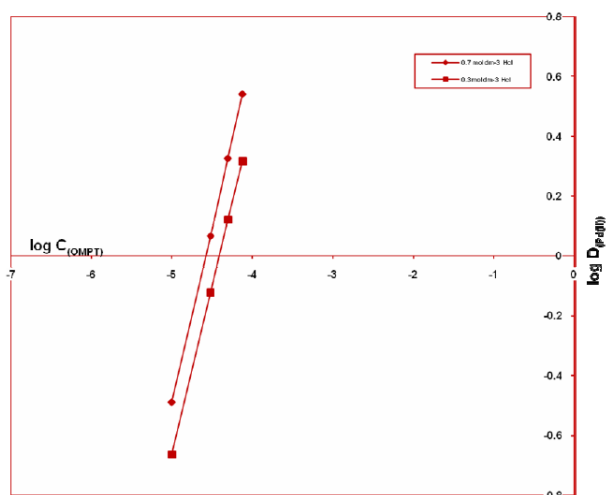


Fig 9. Plot of $\log D_{\text{Pd(II)}}$ vs. $\log C_{\text{OMPT}}$. Pd(II)- $6.0 \mu\text{g cm}^{-3}$, OMPT- $1.0 \times 10^{-4} \text{ mol dm}^{-3}$, hydrochloric acid - 0.7 mol dm^{-3} and 0.3 mol dm^{-3} , $\lambda_{\text{max}} = 340 \text{ nm}$, shaking time 10s.

and intercept for the best fitted line were obtained are 0.02394 and 0.01036. Therefore the content of palladium(II) in real samples can be determined using the straight line equation

$$Y = 0.02394X + 0.01036.$$

Stoichiometry of the complex

Probable composition of extracted species was ascertained by plotting graph of $\log D_{\text{Pd(II)}}$ against $\log C_{\text{OMPT}}$ at 0.7 mol dm^{-3} and 0.3 mol dm^{-3} hydrochloric acid concentrations. The plots are linear with slopes 1.14 and 1.11 respectively (Fig. 9). The probable composition was 1:1 (Pd(II):

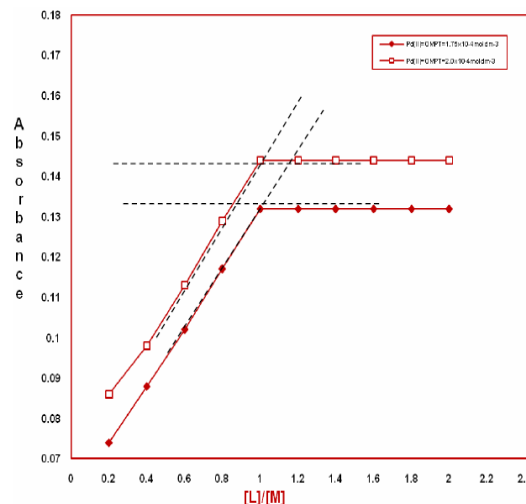


Fig 8. Pd(II):OMPT species by mole ratio method. Pd(II) = OMPT= 1.75×10^{-4} (\blacklozenge) and 2.0×10^{-4} (\square) mol dm^{-3} , $\lambda_{\text{max}} = 340 \text{ nm}$, hydrochloric acid 0.8 mol dm^{-3} , shaking time 10s.

OMPT). This composition was also verified by mole ratio method (Fig. 8) and also confirmed by job's continuous variation method (Fig. 7).

Influence of foreign ions

The effect of the various foreign ions was investigated in order to find tolerance limit of these ions in extraction spectrophotometric determination of Pd(II) (Table 2). The only interfering ion was silver (I) because of its precipitation as silver chloride.

Precision, accuracy and detection limit

To access the reproducibility and accuracy of the method, absorbance measurements with ten different identical solutions containing $60 \mu\text{g}$ palladium (II) were determined by proposed method. The average of these ten readings and standard deviation were determined. Standard deviation was found to be not more than 0.0013 and relative standard deviation was less than 0.82% respectively. It is evident from these results that the method is precise and accurate. The detection limit for palladium(II) for proposed method is

Table 2. Influence of foreign ion
Pd(II) $6.0 \mu\text{g cm}^{-3}$, OMPT $1.0 \times 10^{-4} \text{ mol dm}^{-3}$, hydrochloric acid 0.8 mol dm^{-3} ,
 λ_{max} 340 nm, shaking time 10s.

Foreign Ion	Added as	Tolerance limit(mg)	Foreign Ion	Added as	Tolerance limit(mg)
Mn(II)	MnCl ₂ .6H ₂ O	10	Ce(IV)	Ce(SO ₄) ₂ .4H ₂ O	0.075
Cd(II)	CdCl ₂ .2H ₂ O	100	Pb(II)	Pb(NO ₃) ₂	10
Fe(III)	(NH ₄)Fe(SO ₄) ₂ .12H ₂ O	10	V(V)	V ₂ O ₅	10
Hg(II)	HgCl ₂	5	U(VI)	UO ₂ (NO ₃) ₂ .6H ₂ O	1
Bi(III)	(BiNO ₃) ₃ .5H ₂ O	100	Co(II)	CoCl ₂ .6H ₂ O	10
Ni(II)	NiCl ₂ .6H ₂ O	10	Ba(II)	BaCl ₂ .6H ₂ O	100
Cu(II)	CuSO ₄ .5H ₂ O	100	Ca(II)	CaCl ₂ .2H ₂ O	100
Al(III)	AlCl ₃ .6H ₂ O	100	Sr(II)	Sr(NO ₃) ₂	100
Cr(III)	CrCl ₃	100	Tl(III)	TlNO ₃	0.4
Zn(II)	ZnSO ₄ .7H ₂ O	100	Bromide	KBr	100
Se(IV)	SeO ₂	80	Fluoride	NaF	80
La(III)	La(NO ₃) ₃ .5H ₂ O	10	Phosphate	Na ₃ PO ₄	60
Li(I)	LiCl	50	Sulphate	K ₂ SO ₄	40
Ti(III)	(Ti ₂ SO ₄) ₃	10	Succinate	(CH ₃ COONa) ₂ .6H ₂ O	100
Pt(IV)	H ₂ PtCl ₆	1	Citrate	C ₆ H ₅ O ₇ .H ₂ O	100
Ru(III)	RuCl ₃ .6H ₂ O	1	Malonate	CH ₂ (COONa) ₂	100
Mg(II)	MgCl ₂ .6H ₂ O	1	Tartrate	(CHOH.COOH) ₂	100
Sn(II)	SnCl ₂ .2H ₂ O	0.075	Acetate	CH ₃ COONa.3H ₂ O	100
Ga(III)	GaCl ₃	0.100	Iodide	KI	0.05
Au(III)	HAuClO ₄ .H ₂ O	0.100	Oxalate	(COOH) ₂ .2H ₂ O	10
Mo(VI)	(NH ₄) ₂ Mo ₇ .2H ₂ O	7	Thiocyanate	NH ₄ SCN	1
Sb(III)	Sb ₂ O ₃	5	E.D.T.A.	Na ₂ EDTA	0.5
Be(II)	BeSO ₄ .4H ₂ O	25	Nitrite	NaNO ₂	25
In(III)	InCl ₃ .4H ₂ O	0.15	Nitrate	NaNO ₃	30
Rh(III)	RhCl ₃	1	S ₂ O ₇ ²⁻	Na ₂ S ₂ O ₇	100
Os(VIII)	OsO ₄	0.03	S ₂ O ₈ ²⁻	K ₂ S ₂ O ₈	100
Ir(III)	IrCl ₃	0.1			

determined, as amount corresponding to thrice the standard deviation blank value and the limit of detection is $0.13 \mu\text{g cm}^{-3}$.

4.APPLICATIONS

Determination of palladium(II) in synthetic mixtures corresponding to alloys

Various synthetic mixtures were prepared in laboratory and palladium(II) was determined using recommended procedure. The results are in

agreement with those obtained by direct atomic absorption spectrometry. These results are reported in Table 3.

Determination of palladium(II) from hydrogenation catalyst

Proposed extraction spectrophotometric determination method was applied for the determination of palladium(II) in hydrogenation

Table 3. Separation of palladium (II) from synthetic mixtures corresponding to alloys

Composition of alloy (%)	Amount of palladium(II)		S.D	R.S.D(%)
	Taken (μg)	Found (μg) AAS PM ^a		
Oakay alloy (Pd 18.2; Pt 18.2; Ni 54.2; V 9.1)	60	59.96 59.94	0.345	0.58
Jewelry alloy (Pd 95.0; Rh 4.0; Ru 1.0)	60	59.95 59.92	0.896	1.50
Stibopalladinate alloy (Pd 75.0; Sb 25.0)	60	59.97 59.95	0.745	1.24
Pd-Cu alloy (Pd 60; Cu 40)	60	59.95 59.92	0.486	0.81

PM^a : Present method, average of five determinations.**Table 4.** Determination of palladium (II) in hydrogenation catalyst

Hydrogenation catalyst	Amount of palladium(II)		S.D.	R.S.D. (%)	C.L. $\alpha = 0.95$
	Taken (μg)	Found (μg) AAS PM ^a			
Palladium on activated charcoal (Pd-C, 30% Pd)	30	29.88 29.80	0.155	0.52	0.25
Palladium on activated charcoal (Pd-C, 5.0% Pd)	5.0	4.95 4.95	0.033	0.66	0.052
Palladium on barium sulphate (Pd-BaSO ₄ , 5.0% Pd)	5.0	4.94 4.93	0.020	0.41	0.032

catalysts (Table 4), Palladium on activated charcoal [29] (Pd-C,30%), palladium on barium sulphate [29] (Pd-BaSO₄,5%), palladium on activated charcoal [29] (Pd-C,5%). About 0.3 gm of the catalyst sample was treated twice with 10 cm³ portion of aqua regia, then evaporated to 5 cm³, and on cooling distilled water was added to it .it was filtered using whatmann filter paper no.41 and filtrate was transferred into 250 cm³ calibrated flask.

The residue was washed with 20 cm³ of 2.0 mol dm⁻³ nitric acid and then with distilled water. Finally the sample was made up to mark with distilled water and suitable aliquots of this solution were then analyzed as per recommended procedure for palladium (II) determination. The results obtained are in agreement with those obtained by direct atomic absorption spectrometry (Table 4).

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REFERENCES

- P. S. More, A. D. Sawant, *Analyt. Lett.*, **27**, 1737 (1994).
- S. Ichinoki, S. Otani, Y. Fujii, *J. Liqui. Chrom. Rel. Techni.*, **29**, 2457 (2006).
- R. S. Lokhande, N. Saini, *Asian. J. Chem.*, **19**, 159 (2007).
- D. Yanjie, G.Ke, *Bull. Korean. Chem. Soc.*, **26**, 943 (2005).
- S.H. Gaikwad, M.A. Anuse, *Ind J. Chem. Technol.*, **10**, 447 (2003).
- T.V. Popova, V.L. Tolmachev, S.V. Al Ansari, N.V. Sheheglova, *J. Anal.Chem.*, **56**, 364 (2001).
- R.S. Lokhande, H.G. Namade, A.B. Chaudhari, D.G. Hundiwale, *Asian J. Chem.*, **13**, 596 (2001).
- R.S. Lokhande, Y.R. Bambardekar, *J.Ultra.Chem.*, **3**, 35 (2007).
- R.S. Lokhande, A.D. Sawant, V.D. Barhate, *Res.J. Chem.Env.*, **11**, 7 (2007).
- T. Nakanishi, M. Otomo, *Anal. Sci.*, **1**, 161 (1985).
- K. Hayashi, Y. Sasaki, S. Tagashira, M. Nakashima, *Anal. Sci.*, **1**, 51 (1985).
- J.A. Stratis, A.N. Anthemidis, G.S. Vasilikiotis, *Analyst.*, **109**, 373 (1984).
- C.A. Fontain, C.B. Marone, R. Olisina, *Bull. Chem. Soc. Jap.*, **61**, 4121 (1988).

14. I.K. Sahara, I. Tachi, E. Tsuda, N. Hata, S. Taguchi, K. Goto, *Analyst.*, **114**, 1479 (1989).
15. A. Safavi, A. Massouni, *Microb. Acta.*, **42**, 314 (1990).
16. A.M.A. Kader, K.S. Prasad, *Tr. J. Chem.*, **20**, 222 (1996).
17. M.B. Gholivand, N. Nozari, *Talanta*, **52**, 1055 (2000).
18. V. Vojkovic, V. Druskovik, *Croat. Chem. Acta.*, **76**, 87 (2003).
19. P.T. Gojare, S.H. Gaikwad, M. A. Anuse, *Res. J. Chem. Env.*, **5**, 3 (2001).
20. L.R. Sharma, J.R. Kumar, K.J. Reddy, A.K. Kumar, A.V. Reddy, *Anal. Sci.*, **18**, 1257 (2002).
21. S.H. Gaikwad, T.N. Lokhande, M.A. Anuse, *Ind. J. Chem.*, **44**, 1625 (2005).
22. S.L. Narayana, K.J. Reddy, S.A. Narayana Reddy, J. R. Kumar, A.V. Reddy, *J. Chinese. Chem. Soc.*, **54**, 1233 (2007).
23. G.B. Pethe, S.G. Bhadange, M.D. Joshi, A. . Aswar, *Pelagia. Res. Lib.*, **1**, 58 (2010).
24. B.N. Prabhu, S.M. Khopkar, *Bull. Chem. Soc. Jap.*, **49**, 2139 (1976).
25. M.R. Jamali, Y.A.. Sadi, R.R. Kozani, F. Shemirani, *E. J. Chem.*, **6**, 1077 (2009).
26. A.Z. Ringbom, *Z. Anal. Chem.*, **115**, 332 (1939).
27. N.H. Furman, *Standard Method of Chemical Analysis*, 6th edition, Malabar Florida, 1962.
28. R. L. Frank, P. V. Smith, *Org. Synth.*, **3**, 735 (1955); *ibid*, **28**, 89 (1948).
29. A.I. Vogel, *A Textbook of Practical Organic Chemistry*, 5th edition, Pearson Education, Ltd, South Asia, 2006.

ЕКСТРАКЦИОННО СПЕКТРОФОТОМЕТРИЧНО ОПРЕДЕЛЯНЕ НА ПАЛАДИЙ (II) С О-МЕТИЛ-ФЕНИЛКАРБАМИД В СИНТЕТИЧНИ СРЕДИ

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(Резюме)

Предложен е нов метод за екстракционно спектрофотометрично определяне на паладий, използвайки ниски концентрации на о-метил-фенилкарбамид (ОМРТ). Последният реактив извлича количествено из хлороформ следи от паладий (II) в среда на 0.8 mol dm^{-3} солна киселина. Равновесието се достига само за 10 секунди. Абсорбцията на жълто-оцветения комплекс паладий (II)-ОМРТ се определя при 340 nm. Комплексът е стабилен за повече от 70 часа. Моларното съотношение на екстрахираните компоненти е 1:1 и е потвърдено в двойно-логаритмични координати. Законът на Беег се спазва в интервала от 0.01 до $15.0 \text{ } \mu\text{g cm}^{-3}$. Моларната абсорбция и чувствителността по Сандел са $2.85 \times 10^3 \text{ dm}^3 \text{ mole}^{-1} \text{ cm}^{-1}$ and $0.037 \text{ } \mu\text{g cm}^{-2}$. Методът не се влияе от голям брой катиони и аниони. Той може да се прилага за разделяне на паладий (II) от много-компонентни смеси и катализатори за хидриране.