

Synergistic extraction and spectrophotometric determination of palladium(II), iron(III), and tellurium(IV) at trace level by newly synthesized *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix(4)arene

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Received: 4 October 2007 / Accepted: 22 February 2008 / Published online: 19 March 2008
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Abstract A spectrophotometric method for the determination of palladium, iron and tellurium from nitric acid media after extraction of their *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix(4)arene [DMIAPAC] complexes has been developed and possible synergistic effects have been investigated. Chloroform, carbon tetrachloride, cyclohexane, 1,2-dichloroethane, toluene and xylene were used as the diluents. The maximum enhancement was obtained in the presence of 30% 1,2-dichloroethane. The trace amounts of metals were determined spectrophotometrically. Beer's law obeyed in the concentration range of 5.0–95.0 µg, 8.0–120.0 µg and 10.0–140.0 µg/10 mL of the final solution of palladium, iron and tellurium, respectively. The molar absorptivities ($1 \text{ mol}^{-1} \text{ cm}^{-1}$) and Sandell's sensitivities ($\mu\text{g cm}^{-1}$) were calculated: Pd(II) = 1.73×10^4 and 0.0061; Fe(III) = 1.08×10^4 and 0.0052; Te(IV) = 1.67×10^4 and 0.0077. Ten replicate analyses containing 20 µg of Pd(II), 12.5 µg of Fe(III) and 32 µg of Te(IV) gave mean absorbance of 0.326, 0.242 and 0.418 with relative standard deviation of 0.36, 0.65 and 0.82% for Pd(II), Fe(III) and Te(IV), respectively. The interference of various ions was studied and optimum conditions were developed for the determination of these metals in certain alloys and synthetic mixtures.

Keywords *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix(4)arene · Palladium · Iron · Tellurium · Synergistic extraction

Introduction

The development of supramolecular chemistry has been dominated largely by the design and synthesis of macrocyclic compounds. The calixarenes, well known family of macrocyclic oligomers or oligophenols display interesting organizing properties for the building of various ligating sites to recognize various species including cations, anions and neutral molecules having unique three dimensional shapes and complexation ability [1–4]. They have found many applications in the design of various receptors and sophisticated molecular assemblies. These are basket shaped compounds of potential interest for host–guest complexation studies, new drugs delivery system, drug intermediate, metal cation separation which can incorporate the guest molecules in their molecular cavities or they aggregate to form molecular pore, tube, clusters or membranes etc. In recent years, a lot of papers and patents on the synthesis of cyclic phenol-formaldehyde oligomers have been published [5]. Furthermore, calixarenes which are accessible by the base-catalyzed condensation of para-substituted phenols with formaldehyde are ideal framework for the development of chromogenic ionophores in the molecular recognition of ionic species of the chemical and biological interest. The incorporation of suitable bathochromic moiety into the calixarene results in a designed chromogenic receptor. The chemistry of calix(*n*)arenes ($n = 4–8$) is well represented in the literature due to the availability of facile routes for their preparation. Extraction, transport, stability constant and spectrophotometric measurements augmented by spectral data and computer simulation studies provide evidence that many of its upper rim modified derivatives have many significant ionophoric properties for cations, several with good selectivity within groups of metals. Also, a number of appropriately designed calixarene exhibit a large variety of

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function such as inclusion compounds [6], selective complexing agents for metal ions [7, 8] and catalyst [9, 10]. Furthermore, azo compounds are the most widely used class of the dyes due to their versatile applications in various fields such as the dyeing of textile fiber, the coloring of different materials, and for plastics, biomedical studies and in organic synthesis [11, 12]. Moreover, azo group brings to calixarenes a chromogenic activity.

An extensive survey reveals that extraction behavior of transition metal ions have also been studied for calix(4)arenes [13, 14]. Interestingly, it is observed that azocalixarenes show remarkable change in the extraction of transition metals [15]. Deligoz et al. [16] have also reported that selective liquid–liquid extraction of various alkali, alkaline earth and transition metal cations from aqueous phase to the organic phase is carried out by using azocalixarenes. Thus, with these objectives in mind and in continuation of our recent work on the synthesis of calix(4)arenes [17], azo substituted heterocyclic systems [18, 19] and solvent extraction [20], efforts have been laid down to undertake comprehensive synergistic extraction of Pd(II), Fe(III) and Te(IV) using hitherto uninvestigated synthesized calix(4)arene containing azo functionality. The interference of various ions was studied and optimum conditions were developed for the determination of these metals in certain alloys and synthetic mixtures.

Experimental

Reagents and apparatus

A Shimadzu 1700 Pharmaspec UV–Vis Spectrophotometer with standard rectangular quartz cells (path length 10 mm) was used for the absorbance spectra of organic extractants. ^1H NMR spectra were recorded at room temperature on a 300 MHz Bruker advance DRX 300 instrument using TMS (tetramethylsilane) as an internal standard. Chemical shifts (δ) were recorded in ppm values relative to TMS. IR Spectra were run on a Perkin Elmer model 377 spectrophotometer in KBr pellets. Melting points were determined in open capillary tubes using an electric melting point apparatus and are uncorrected. Analytical thin layer chromatography was performed using E. Merck silica gel 0.50 mm plates Merck No. 5700 (Merck Darmstadt, Germany) using ethyl acetate/xylene (3:7 v/v) as eluent and solvents used were of AR grade.

Synthesis of *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix(4)arene

The typical synthesis of *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix(4)arene was proceeded in four reaction steps (Scheme 1).

- (i) Synthesis of 4-hydroxy-*p*-aminoazobenzene (**2**).
- (ii) Synthesis of 3-[4-(4'-hydroxyphenylazo)phenylazo]pentane-2,4-dione (**3**).
- (iii) Synthesis of 3,5-dimethyl-4-[4'-(4''-hydroxyphenyl)azophenylazo]isoxazole (**4**).
- (iv) Synthesis of *p*-[4-(3,5-dimethylisoxazolyl) azophenylazo]calix(4)arene (**5**).

Synthesis of 4-hydroxy-*p*-aminoazobenzene (**2**)

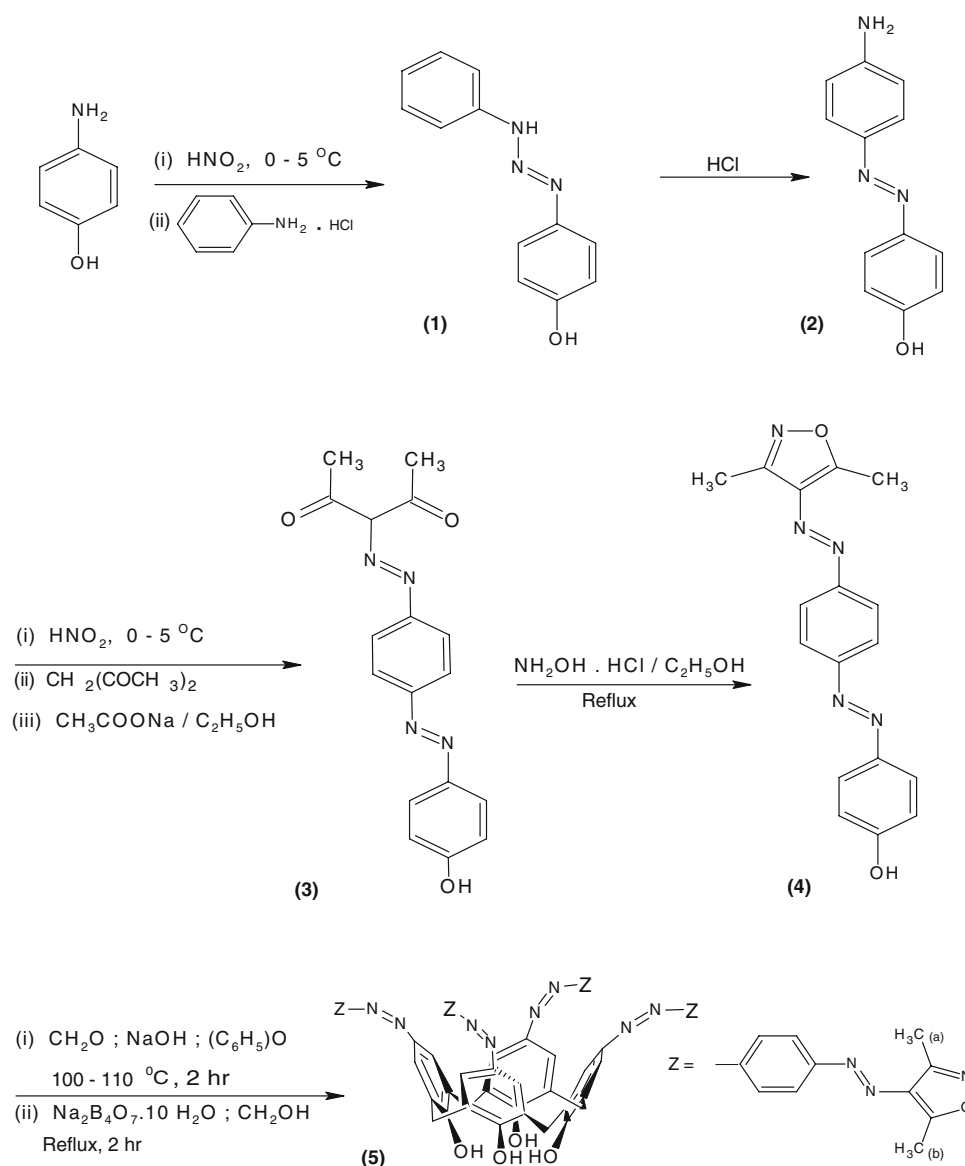
In a 250 cc beaker, water (75.0 mL), conc. hydrochloric acid (20.0 mL) and *p*-aminophenol (16.35 g) were taken. Contents were shaken vigorously and 50 g crushed ice was added to them. To this, a solution of 5.20 g of sodium nitrite in 12.0 mL of water was run with constant shaking during a period of 5–10 min and allowed to stand with frequent shaking for 15 min. Now a solution of 21.0 g of crystalline sodium acetate in 40.0 mL of water was added. A yellow precipitate of diazoaminobenzene (**1**) begins to form immediately; allowed to stand for 45 min below 20 °C. It was filtered by Buchner funnel and washed with 250 mL of cold water, drained and dried by pressing in between the filter paper sheets. Further, 5.0 g of finely powdered diazoaminobenzene was dissolved in 12–15 g of aniline in a small flask and 2.50 g of finely powdered aniline hydrochloride was added to it. Mixture was warmed with frequent shaking on a water bath at 40–45 °C for 1 h. Reaction mixture was then allowed to stand for 30 min. To this, 15 mL of glacial acetic acid diluted with equal volume of water was added, stirred to remove the excess of aniline. Mixture was allowed to stand with frequent shaking for 15 min. The *p*-aminoazobenzene (**2**) was filtered, washed with water and dried. It was recrystallized from 15 to 20 mL of carbon tetrachloride to obtain pure compound. Yield 80%, m.p. 125–127 °C.

IR (ν cm^{-1} , KBr) 3458 (O–H), 3040(C–H, sp^2), 1681 (N=N), 1645 (C=C), 1600, 1495, 1390 (C...C ring str.), 1184 (C–O), 910, 840, 750 (sub. phenyl); ^1H NMR (δ ppm) 5.1 (s, NH), 7.1–7.6 (m, 8H, Ar–H), 8.35 (bs, OH); FAB MS (m/z , % R.A.) 213 (M^+ , 45). Anal calc. (%) for $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}$: C, 67.60; H, 5.14; N, 19.72; Found C, 67.58; H, 5.06; N, 18.64.

Synthesis of 3-[4-(4'-hydroxyphenylazo)phenylazo]pentane-2,4-dione (**3**)

In a 250 cc beaker, compound (**2**) (9.85 g, 0.05 M), conc. hydrochloric acid (10.0 mL) and water (35.0 mL) were taken. All the contents were kept in an ice-bath. To this, a solution of 2.25 g of sodium nitrite in 6.0 mL of water was added very slowly, keeping the temperature below 5 °C.

Scheme 1 An overview of synthetic pathway of *p*-[4-(3,5-dimethylisoxazolyl)azophenylazo] calix(4)arene



Meanwhile, in other beaker a mixture of pentane-2,4-dione (1.0 g, 0.5 M), sodium acetate (12.5 g) in 50 mL of ethyl alcohol was prepared and kept in a refrigerator for 30 min. To this, already prepared diazotized solution was added slowly under constant stirring. An orange product thus obtained was filtered, washed thoroughly with water and recrystallized from absolute alcohol to yield pure compound (3). Yield 72%, m.p. 171–173 °C.

IR ($\nu \text{ cm}^{-1}$, KBr) 3410 (O–H), 3074 (C–H, sp^2), 2850 (C–H, sp^3), 1676 (N=N), 1705 (C=O), 1660 (C=C), 1605, 1490, 1465 (C···C ring str.), 1166 (C–O), 740, 705, (sub. phenyl); $^1\text{H NMR}$ (δ ppm) 2.44 (s, 6H, $\text{CH}_3 \times 2$), 3.56 (s, H, CH), 7.33–7.11 (m, 4H, Ar–H), 7.45 (s, 4H, Ar–H), 8.46 (bs, OH); FAB MS (m/z , % R.A.) 325 ($\text{M}^+ + 1$, 35). Anal calc. (%) for $\text{C}_{17}\text{H}_{16}\text{N}_4\text{O}_3$: C, 62.96; H, 4.94; N, 17.28; Found C, 62.84; H, 4.90; N, 17.22.

Synthesis of 3,5-dimethyl-4-[4'-(4''-hydroxyphenyl)azophenylazo]isoxazole (4)

In 250 cc R.B. flask, equimolar quantity of compound (3) and hydroxylamine hydrochloride was taken together and refluxed for 4 h in alcoholic medium at 80 °C. Contents were allowed to keep for overnight period. Solid compound thus generated on standing was filtered, dried and recrystallized from carbon tetrachloride. Yield 68%, m.p. 225–227 °C.

IR ($\nu \text{ cm}^{-1}$, KBr) 3470 (O–H), 3042 (C–H, sp^2), 2870 (C–H, sp^3), 1691 (N=N), 1640 (C=C/C=N), 1580, 1475, 1395 (C···C ring str.), 1172 (C–O), 925, 870 (sub. phenyl); $^1\text{H NMR}$ (δ ppm) 2.36 (s, 6H, $\text{CH}_3 \times 2$), 7.35–7.08 (m, 4H, Ar–H), 7.53 (s, 4H, Ar–H), 8.38 (bs, OH); FAB MS (m/z , % R.A.) 322 ($\text{M}^+ + 1$, 38). Anal calc. (%) for

$C_{17}H_{15}N_5O_2$: C, 63.54; H, 4.71; N, 21.72; Found C, 63.48; H, 4.67; N, 21.68.

Synthesis of p-[4-(3,5-dimethylisoxazolylazo)phenylazo]calix(4)arene [DMIAPAC] (5)

Compound (4) (4.81, 0.15 M), *p*-formaldehyde (5.37 g, 0.18 M) and 0.27 g (0.0067 M) of solid 98% NaOH were dissolved in 25.0 mL diphenylether. The reaction mixture was heated at 100–110 °C under stirring for 2 h. It was then neutralized with hydrochloric acid and transferred to a round bottomed three neck flask equipped with a stirrer and a thermometer. Then 2.27 g (0.006 M) of sodium tetraborate decahydrate and 50 mL of hot ethylene glycol were added under stirring. The reaction mixture was refluxed for 2 h followed by cooling up to 80 °C, it was poured into 500 mL of cold water. The precipitate thus generated were filtered off and dried under vacuum at 40 °C. The dried product was recrystallized from a mixture of chloroform and methanol (3:7; v/v). Yield 68%, m.p. 225–227 °C.

IR (ν cm^{-1} , KBr) 3152 (O–H), 3042 (C–H, sp^2), 2870 (C–H, sp^3), 1672 (N=N), 1640 (C=C/C=N), 1580, 1475, 1395 (C...C ring str.), 1195 (C–O), 925, 870, (sub. phenyl); 1H NMR (δ ppm) 2.51 (s, 24 H, $CH_3 \times 8$), 4.46 (d, 4H, Hax of Ar– CH_2 –Ar), 3.38 (d, 4H, Heq of Ar– CH_2 –Ar), 6.92–7.2 (m, 8H, Ar–H), 7.45 (s, 16H, Ar–H), 9.8 (bs, OH); FAB MS (m/z , % R.A.) 322 ($M^+ + 1$, 38). Anal calc. (%) for $C_{17}H_{15}N_5O_2$: C, 63.54; H, 4.71; N, 21.72; Found C, 63.48; H, 4.67; N, 21.68.

Analytical procedure

Metal picrate

Metal picrate was prepared by neutralizing picric acid (Merck) with the appropriate metal salt in ethanol, according to the method of Wong et al. [21]. They were dried in vacuum and purified by recrystallization from absolute alcohol.

Extraction procedure

Stock solution of metal picrate (1.0×10^{-4} M) was prepared in double distilled water. The solution of *p*-[4-(3,5-dimethylisoxazolylazo)phenylazo]calix(4)arene (2.0×10^{-2} M) was prepared in 1,2-dichloroethane. These two solutions were mixed and centrifuged for an hour. In all the extractions, aqueous phase was adjusted to the desired nitric acid molarity (3 M). The mixture was then allowed to stand for ten minutes to complete the phase separation and assayed by spectrophotometrically. The concentration of picrate ion remaining in the aqueous phase was then determined against

the blank. Extraction is considerably enhanced by the presence of organic additive. It is observed that the maximum extractability was shifted towards lower acidity in the presence of organic additive and at higher concentration (>3.0 M), the extraction was less influenced by additive.

Results and discussion

Dependence of distribution on nitric acid concentration

The effect of water-miscible mixture on the extraction was investigated at increasing nitric acid molarities (Fig. 1) in the polar phase. The distribution ratio (D), defined as the ratio of the metal in the organic phase per mL and the concentration of the metal in polar phase per mL was calculated. From knowledge concerning ' D ', the volume of the aqueous phase (V_a) and that of the organic phase (V_o), the percentage of extraction ($E\%$) was calculated: $E(\%) = 100 D / (D + V_a / V_o)$. The distribution ratio (D) was considerably enhanced by the presence of organic additives. Higher D values were obtained by low acid concentration (2–3 M HNO_3). This phenomenon had been observed earlier for extraction from mixed aqueous organic media [22, 23].

Dependence of distribution on the DMIAPAC concentration

The dependence of D on the DMIAPAC concentration (0.01–1.0 M) was studied with 3M HNO_3 . Since 0.01 M DIMAPAC solution extracted more than 99% in a single step, 0.02 M solution was used for further studies. The extraction process follows the reaction.

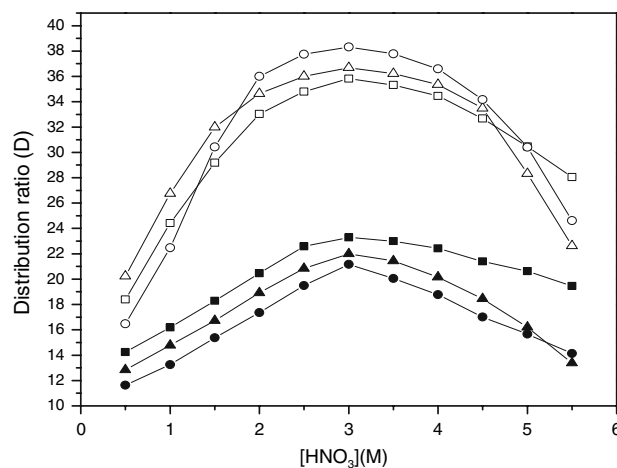
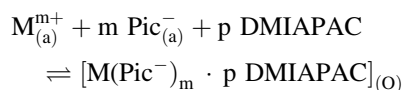


Fig. 1 Effect of the nitric acid concentration on the extraction of Pd(II), Fe(III) and Te(IV) with 0.02 M DMIAPDA from a pure nitric acid solution [Pd(II): ▲, Fe(III): ■, and Te(IV): ●]; a solution containing 30% (v/v) 1,2-Dichloroethane [Pd(II): △, Fe(III): □ and Te(IV): ○]



where, $p = 1, 2, \dots$ and the subscripts (a) and (o) refer to the species present in aqueous and organic phase, respectively for which the equilibrium constant (K_{ex}) is given by:

$$K_{\text{ex}} = \frac{[M \cdot (\text{Pic}^{-})_m \cdot p \text{DMIAPAC}]_{(o)}}{[M^{m+}]_{(a)} [\text{Pic}^{-}]_{(a)}^m [\text{DMIAPAC}]_{(o)}^p}$$

By introducing the distribution ratio D as

$$D = [M(\text{Pic}^{-})_m \cdot p \text{DMIAPAC}]_{(o)} / [M^{m+}]_{(a)}$$

We obtained

$$\log D = \log K_{\text{ex}} + m \log [\text{Pic}^{-}]_{(a)} \\ + p \log [\text{DMIAPAC}]_{(o)}$$

A plot of $\log D$ vs. $\log [\text{DMIAPAC}]_{(o)}$ in the organic phase for studied different metal ion yielded the slope of 1.10, 1.01 and 0.95 for Pd(II), Fe(III) and Te(IV), respectively indicating that metal ion and ligand form 1:1 inclusion compound and the extracted molecular species would be $\text{Pd}(\text{Pic})_2 \cdot \text{DMIAPAC}$, $\text{Fe}(\text{Pic})_3 \cdot \text{DMIAPAC}$ and $\text{Te}(\text{Pic})_4 \cdot \text{DMIAPAC}$.

Influence of diluents

The extractability and selectivity for the extraction of metals by organic extractants are greatly affected by the nature of solvents. To study the influence of several typical

commonly available aliphatic and aromatic organic solvents on the efficiency of extraction by DMIAPAC, the extractability of the metals from 3 M HNO_3 was investigated with 0.02 M DMIAPAC dissolved in various solvents (diluents). Although non-polar diluents such as xylene and benzene yielded less extraction than that of polar diluents, a significant enhancement was obtained in extraction by introducing polar organic additives having a high dielectric constant to the aqueous phase. The addition of the polar additive decreased the dielectric constant of the aqueous phase. The maximum enhancement in extraction was obtained in the presence of 30% 1, 2-dichloroethane.

Beer's law and sensitivity

Under optimum conditions described above, calibration graphs for the metals were obtained at 340, 592 and 425 nm against reagent blank for Pd(II), Fe(III) and Te(IV), respectively. Beer's law holds in the calibration ranges: Pd, 5–95 μg ; Fe, 8–120 μg and Te, 10–140 μg /10 mL of the final solution. The molar absorptivities ($\text{l mol}^{-1} \text{cm}^{-1}$) and Sandell's sensitivities ($\mu\text{g cm}^{-1}$) were calculated Pd(II) = 1.73×10^4 and 0.0061, Fe(III) = 1.08×10^4 and 0.0052, and Te(VI) = 1.67×10^4 and 0.0077, respectively. Ten replicate analyses containing 20 μg Pd(II), 12.5 μg Fe(III) and 32 μg Te(VI) individually gave mean absorbance of 0.326, 0.242 and 0.418 with relative standard deviation of 0.68, 0.85 and 1.02%, respectively. The proposed method is found to be more sensitive and selective than many of the recent spectrophotometric methods (Table 1). It is probably because of

Table 1 Comparative study of the present method with some of the existing methods

Metal	Reagents	Molar absorptivity (ϵ) $\text{l mol}^{-1} \text{cm}^{-1}$	Reference
Palladium(II)	1-Phenyl-4,4,6-trimethyl(1H,4H)-pyrimidine-2-thiolates	4.06×10^3	[24]
	Potassium ethylxanthate	1.20×10^4	[25]
	4-(2'-Thiazolylazo) resacetophenoneoxime	2.20×10^3	[26]
	1-Phenyl-1-hydrozonyl-2-oximino-1,2-ethanedione	6.81×10^3	[27]
	Nioxime	3.46×10^3	[28]
	Present method	1.73×10^4	
Iron(III)	Morpholine-4-carbodithioate	2.50×10^3	[29]
	1-Nitroso-2-naphthol-3,6-disulphonate (nitroso-R)	7.58×10^3	[30]
	4- <i>p</i> -Nitrobenzoyl-2,4-dihydro-5-methyl-1-phenylpyrazol-3-one	9.46×10^3	[31]
	Indone-1,2,3-trionetrixime	7.80×10^3	[32]
	1-(2-Thiazolylazo)-2-naphthol	1.01×10^4	[33]
	Trifluoroacetylacetone	3.77×10^3	[34]
	Cyanex 301	5.05×10^3	[35]
Present method	1.08×10^4		
Tellurium (IV)	4- <i>p</i> -Nitrobenzoyl-2,4-dihydro-5-methyl-1-phenyl-pyrazol-3-one	1.68×10^3	[31]
	Present method	1.67×10^4	

Table 2 Effect of divers ions in the determination of palladium, iron and tellurium

Ions	Absorbance		
	Palladium at 340 nm	Iron at 592 nm	Tellurium at 425 nm
Acetate	0.326	0.242	0.418
Citrate	0.326	0.242	0.418
Chloride	0.326	0.242	0.418
EDTA	0.868*	1.042*	1.826*
Iodide	0.326	0.242	0.418
Thiocyanate	0.108*	0.242	0.418
Bismuth(III)	0.326	0.242	0.418
Copper(II)	–*	2.802*	2.002*
	0.326 ^a	0.242 ^a	0.418 ^d
Cobalt(II)	0.326	–*	–*
		0.242 ^c	0.418 ^c
Chromium(III)	0.326	0.242	0.418
Indium(III)	0.326	0.242	0.418
Iron(III)	1.862*	–	0.418
	0.326 ^a	–	–
Magnesium(II)	0.326	0.242	0.418
Mercury(II)	0.326	0.242	0.418
Molybdenum(VI)	0.326	0.242	0.418
Nickel(II)	0.326	–*	0.418
		0.242 ^c	
Palladium	–	0.242	0.418
Rhodium(III)	0.326	0.242	0.418
Tellurium(IV)	0.326	0.242	–
Vanadium(V)	0.326	0.242	0.418
Zinc(II)	0.326	0.242	0.418

* Interfered seriously

^a Masked by 1 mg of thiourea^b Masked by 2 mL of 5% NaF and 2 mL of 2% trisodium citrate^c Masked by 5 mL of 1% hydroxyl amine^d Masked by 4 mL of 2% KI^e Masked by 5 mL of 5% sodium cyanide solution

effective ion pair formation between big size inclusion compounds of metal ion and large charge diffused picrate anion. Furthermore, both picrate and ligand has quite enough lipophilic character which facilitates extraction from aqueous to organic phase. This method has been employed for the determination of palladium, iron and tellurium in various complex materials.

Effect of foreign ions

The interference from foreign ions was studied. In general, 1000 µg amount of salt of the studied anions and amounts equivalent to 500 µg metal ions were added individually to an aliquot containing 20 µg Pd(II), 12.5 µg Fe(III) and 32 µg Te(VI). Among the anion examined (Table 2), EDTA interfered in the determination of all the metals and thiocyanate interfered in the determination of palladium. The non-interference may be attributed to their low stability constants compared to the metal complexes. Among the metal ions studied Cu(II), Fe(III) interferes in the determination of palladium. The interference of Cu(II) was eliminated by adding 1 mg of thiourea during extraction and interference of Fe(III) was masked by 2 mL of 5% Na F and 2 mL of 2% trisodium citrate. The interference of Ni(II) and Cu(II) was masked by adding 5 mL of 1% hydroxylamine and 1 mg of thiourea solutions, respectively in the estimation of iron. Cu(II) and Co(II) interfere in the determination of tellurium which is masked by KI and NaCN, respectively. The interference due to various ions was studied and optimum conditions were developed for the determination of three metals in certain alloys and synthetic mixture.

Determination of iron in alloy

A sample of alloy (0.1–1.0 g) was dissolved in 10–15 mL of aquaregia on a water bath. After cooling, 3–5 mL of hydrogen peroxide (30%) was added. The excess of peroxide was decomposed by heating the solution on a water

Table 3 Determination of iron in some alloys

Sample	Certified values (%)	Amount of iron (µg)		RSD (%)
		Taken	Found ^a	
NBS, SRM-85b Aluminium alloy	Cu: 3.99 ^b , Mg: 1.94, Mn: 0.61, Cr: 0.21, Si: 0.18, Fe: 0.24, Ti: 0.020, Pb: 0.021, Ga: 0.019, V: 0.066, Ni: 0.084 ^c	2.16	21.52 ± 0.02	0.36
NBS, SRM-94c Die casting alloy	Cu: 1.01 ^b , Al: 4.13, Sn: 0.06, Fe: 0.018, Cd: 0.002, Pb: 0.002, Mg: 0.042, Ni: 0.006 ^c	16.20	16.02 ± 0.01	0.65

^a Average of seven determinations ± Standard deviation^b Masked by 1mg of thiourea^c Masked by 2mL of 5% NaF + 2 mL of 2% trisodium citrate

Table 4 Sequential determination of palladium and tellurium in synthetic samples

Composition of the mixture (%)	Taken (μg)		AAS (μg)		Parent method (μg)		RSD (%)	
	Pd	Te	Found		Found		Pd	Te
			Pd	Te	Pd	Te		
Pd(II): 8.50 Ir(III): 17.00 Rh(III): 20.00 Mn(II): 24.00 Cu(II): 5.00 ^a Te(IV): 14.00	8.50	14.00	8.46	14.00	8.40	13.92	0.65	0.82
Pd(II): 6.00 Ir(III): 21.00 Ni(II): 5.00 Co(II): 15.00 ^b Te(IV): 9.60	6.00	9.60	5.98	9.60	9.52	9.48	1.02	0.75

^a Masked by 1 mg of thiourea^b Masked by 5 mL of 5% NaCN solution

bath. The mixture was cooled and then filtered and then diluted to 250 mL with distilled water in a calibrated flask. An appropriate volume of this solution was taken and iron was determined by the general procedure. Sequential determination of palladium and tellurium in synthetic mixture were prepared containing palladium and tellurium together with other metals. These metals were determined by a general procedure after masking the interference of the metal ions by using suitable masking agents. The results are presented in Tables 3 and 4.

Conclusion

As discussed in this paper, a new chromogenic *p*-[4-(3,5-dimethylisoxazolylazo)phenylazo]calix(4)arene has been synthesized and a simple method is suggested for enhancing the extraction of palladium, iron and tellurium and their determination at trace level spectrophotometrically. The method proved to be convenient and highly selective. In addition, results achieved were highly accurate.

Acknowledgements We are thankful to the Department of Science and Technology (DST) Govt. of India, New Delhi for providing financial support for the research project (SR/S-1/IC-17/2006) and Head, Sophisticated Analytical Instrumentation Facility (SAIF), CDRI, Lucknow for spectral measurement.

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