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Synthesis of novel analytical reagent, 2,6-diacetylpyridine bis-4-phenyl-3-thiosemicarbazone and its analytical applications: Determination of Pd(II) in spiked samples

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ABSTRACT

New synthesized reagent 2,6-Diacetylpyridine bis-4-phenyl-3-thiosemicarbazone (2,6-DAPBPTSC) is proposed as a sensitive and selective analytical reagent for the separation and extractive spectrophotometric determination of palladium(II) at pH: 4.0 to form a yellowish orange colored 1:1 chelate complex, which is very well extracted in to isoamyl alcohol. The maximum absorbance was measured at 410 nm. This method obeys Beer's law in the concentration range 0.0 -12.6 μg mL^{-1} and the correlation coefficient of Pd(II)- 2,6-DAPBPTSC complex is 0.962, which indicates an adequate linearity between the two variables with good molar absorptivity and Sandell's sensitivity, 1.156 $\times 10^{4}$ L mol^{-1}cm^{-1} and 0.0092 μg cm^{-2}, respectively. The instability constant of complex calculated from Asmus' method is 1.667 $\times 10^{4}$ at room temperature. The precision and accuracy of the method is checked with calculation of relative standard deviation (n = 5), 0.371. The method was successfully applied for the determination of Pd(II) in spiked samples. The performance of present method was evaluated in terms of Student't' test and Variance ratio 'f' test which indicates the significance of present method over reported method.

Keywords: Palladium(II), 2,6-DAPBPTSC**,** Extraction, spiked samples, Water samples.

INTRODUCTION

The abundance of palladium in the earth's crust is 8.5 × 10^{-13} %. Palladium and its alloys have a wide range of applications both in the chemical industry and in instrument making[1]. Environmental pollution by the platinum group element (PGEs), mainly related to automotive catalytic converters, is exponentially increasing and reliable and accurate quantification is a mandatory task. The wide use of palladium not only in automotive catalytic converters but also as a drug and in food production has led to a more uncontrolled release of those metals in the environment, with respect to the one due to the traditional chemical industry [2]. Several alloys of palladium with cobalt and nickel have been constructed and studied. Palladium-cobalt alloys show catalytic activity in the hydrogenation of nitrile function [3]. Palladium is one of the precious metals. Owing to its corrosion resistant nature and alloying ability, palladium is an important element in metallurgy. Its alloys are used in dental and medicinal devices and in jewelry manufacture. Moreover, palladium is used in automobile catalysts together with Pt and Rh [4].

For the determination of palladium at micro levels, there are several frequently adopted methods using analytical techniques, such as AAS, ICP-AES, ICP-MS, X-ray fluorescence spectroscopy, spectrophotometry, spectrofluorometry and such other techniques. Among these, spectrophotometric methods are preferred because they are cheaper and easy to handle, with comparable sensitivity and also accuracy, good precision. Among various organic analytical reagents containing sulfur and nitrogen as donar atoms, thiosemicarbazones occupy a unique place. Although thiosemicarbazones are used as complexing agents in the spectrophotometric and extractive spectrophotometric determination of various metals ions[5-13], only a few thiosemicarbazones have been used for the extractive spectrophotometric determination of palladium(II) [14,15]. All of these methods are less sensitive, also the chloroform used for extraction in some of these methods is considered to be hazardous.

These are prompted us to develop a new extractive spectrophotometric method for the determination of Pd(II) by using a novel synthesized analytical reagent, 2,6-diacetylpyridine-bis-4-phenyl-3-thiosemicarbazone (2,6-DAPBPTSC). When compared with already reported extractive spectrophotometric methods, the present method is relatively more sensitive [16-23].

The method also has the advantage of higher selectivity in the presence of EDTA. A comparative account of 2,6-DAPBPTSC with other reagents for determination of palladium(II) is given in table1.

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

Reagent	pH	λ_{max} /nm	$\in_{max,} \times 10^4$/Lmol^{-1}cm^{-1}	M:L	Beer's law range/µg.mL^{-1}	References
5-methyl-2,3-hexanedione dioxime	0.5-1.5	379	0.389	1:2	50-9000	16
Glyoxalbis(4-Phenyl-3-thiosemicarbazone)	9.6	635	0.320	1:1	N.R	17
Phenanthraquinonemono thiosemicarbazone	N.R	540	0.420	N.R	1.3-6.7	18
5- Chlorosalicyladehyde guanylhydrazone	8.1	400	0.712	1:2	0-6.0	19
3-Phenoxybenzaldoxime	4.0	435	0.2434	N.R	0.4-4.0	20
O- Hydroxyacetophenone thiosemicarbazone	6.0	370	0.9	1:1	0.42-10.6	21
Furfurylacroline thiosemi carbozone	2.0-10.0	390	0.41	N.R	N.R	22
1,2-Cyclohexanedionedithio semicarcarbazonemono hydrochloride	6.2-8.0	590	0.53	1:2	1.85-6.75	23
2,6-Diacetylpyridinebis-4-phenyl-3-thiosemicarbazone	4.0	410	1.156	1:1	0-12.6	Present method

N.R: Not reported; M:L : metal : ligand

In the present paper a simple and highly sensitive method is reported for the extractive spectrophotometric determination of trace amounts of palladium(II) by complexion with 2,6-DAPBPTSC. The reagent forms a yellowish orange complex with palladium(II) in an acid medium pH (3.0-4.5), which is easily extractable into isoamylalcohol. Hence a systematic study has been made to develop an extractive spectrophotometric method for determination of micro amounts of palladium in spiked samples.

EXPERIMENTAL SECTION

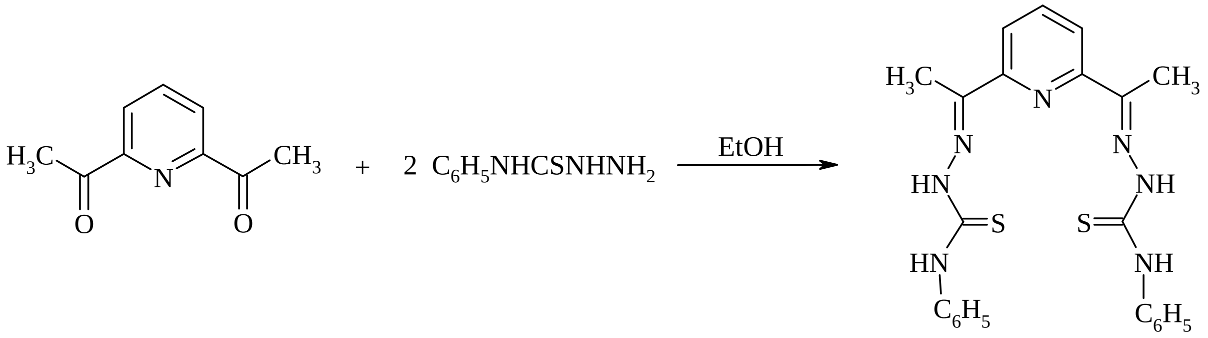
Apparatus: A Shimadzu 2450 UV-VIS spectrophotometer with 1.0 cm quartz cell is used for absorbance studies. An Elico LI-120 digital pH meter is used for pH adjustment. A Perkin-Elmer 170-30 atomic absorption spectrometer is used for the comparison of results. A Nicolet FT-IR 560 Magna spectrometer using KBr (neat) was used to obtain the infrared spectra of the 2,6-DAPBPTSC. The Bruker 300 MHz NMR spectrometer was used to obtain the ^{1}H NMR spectra of the ligand.

Reagents: All the reagents used were of AR grade unless otherwise stated. 2,6-diacetylpyridine-bis-4-phenyl-3-thiosemicarbazone (2,6-DAPBPTSC) is prepared as per the procedure: 1.60 g of 2,6-diacetylpyridine is dissolved in 30 mL of absolute ethanol and mixed in a round bottomed flask with 2.20 g of 4-phenyl-3-thiosemicarbazide is dissolved in 30 mL ethanol. The mixture was heated under reflux for 3 hours and then allowed to cool at room temperature for 12 hours. The crystals obtained for filtration and washed with cold ethanol and then recrystallized from ethanol (scheme 1)[24]. The melting point was 152^{0}C. 2,6-DAPBPTSC does dissolve in N,N-dimethylformamide (DMF), acetone and dimethyl sulphoxide. The characterization of 2,6-DAPBPTSC was carried out by IR and 1H NMR spectroscopy. The IR spectrum of 2,6-DAPBPTSC was shows absorption bands around 1485 cm-1 (C = S), 1540 cm-1(C = N) and 3300 cm-1 (-NH). The 1H NMR (DMSO, ppm): 9.85 (-NH), 2.6 (-CH3), 7.2-7.8 (Ph).

After 0.1mol L^{-1} sodium acetate and 0.1mol L^{-1} acetic acid were prepared in double distilled water, suitable portions of these solutions were mixed to obtain the desired pH.

A sample of 1.664 g of palladium chloride was dissolved in a minimum amount of 2.0 mol L^{-1} hydrochloric acid solution in a liter volumetric flask, and was finally made up to the mark with an identical normal hydrochloric acid solution. This stock solution was standardized gravimetrically using dithizone[25]. More dilute solutions were prepared by diluting the stock solution suitably.

Scheme 1



General analytical procedure: To an aliquot of a working standard solution containing 1× 10^{-4} to 1×10^{-5} mol L^{-1}. Palladium(II) are added pH 4.0 buffer (2 mL), reagent solution (1mL). The mixture is twice shaken with isoamylalcohol (4.0 mL) portions of each time for 1 min and allowed to stand for a few minutes. The organic phases are collected and made up to 25 mL with isoamylalcohol, while its absorbance is measured at 410 nm against the reagent blank.

Water samples: Choices of five water samples were collected in and around Nellore district, A.P, India. Each filtered environmental water samples is evaporated nearly to dryness with a mixture of 2 mL of concentrated H_2SO_4 and 4 mL of concentrated HNO_3 in a fume cupboard and then cooled to room temperature. The residue is then heated with 5 mL of double distilled water, in order to dissolve the salts. The solution is cooled and neutralized with dilute NH_4OH in the presence of 1-2 mL of 0.01% tartrate solution. The resulting solution was filtered and quantitatively transferred into a 10 mL volumetric flask and made upto the mark with double distilled water and analyzed by the recommended procedure [26].

Synthetic mixtures: Various synthetic mixtures were prepared and analyzed for Pd(II) by employing the proposed recommended procedure[26].

Catalysts samples: About 0.3 g of the catalyst sample was treated twice with 10.0 mL portions of aquaregia. The solution was then evaporated to 5.0 mL after cooling the solution was diluted with 20.0 mL of double distilled water and filtered into a 250mL calibrated flask. The residue was washed first with 20.0 mL of 2.0 mol L^{-1} nitric acid and then the solution was made upto the mark with double distilled water. Suitable aliquots of this solution were taken and analyzed for palladium(II), using the recommended procedure [26].

RESULTS AND DISCUSSION

2,6-diacetylpyridine-bis-4-phenyl-3-thiosemicarbazone(2,6-DAPBPTSC) forms a 1:1 (M:L) complex with palladium(II), which is extracted into isoamylalcohol from acetic acid-sodium acetate (pH 4.0) buffer. The yellowish orange Pd(II)-2,6-DAPBPTSC complex has a maximum absorbance at 410 nm and is stable for 46 hours. The conditions for effective extraction are established after studying the effects of various factors, such as pH, choice of the solvent, reagent concentration, salting-out agent and influence of diverse ions, in order to develop a rapid and sensitive extractive spectrophotometric method for the determination of palladium(II) at micro gram levels.

Absorption spectra of Pd(II)-2,6-DAPBPTSC complex: The absorption spectra of the reagent solution against the corresponding solvent as a blank and that of the solution containing palladium(II) complex against the reagent as a blank are recorded in the wavelength range 410 nm. The absorption spectra of the reagent and complex are shown in Fig.1. The spectra reveal that Pd(II)-2,6-DAPBPTSC complex and the reagent have maximum absorbance at 410 nm, respectively. Hence, further absorbance measurements of the Pd(II)-2,6-DAPBPTSC complex are recorded at 400 nm, against the reagent blank.

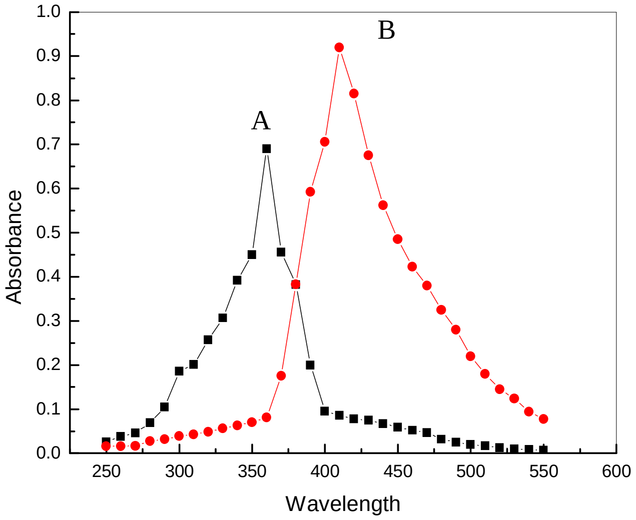


Fig.1. (A) Absorption spectra of 2,6-DAPBPTSC Vs blank, (B) Absorption spectra of Pd(II)-2,6-DAPBPTSC complex : 1 mL of (1.0 ×10^{-4} mol L^{-1}) Pd(II); 2.0 mL of pH 4.0; 1.0 mL of (1 × 10^{-3} mol L^{-1}) 2,6-DAPBPTSC

Effect of pH on extraction of Pd(II)-2,6-DAPBPTSC complex: To arrive at the optimum pH required for full color development, the effect of pH on the color intensity is studied. In each case, a mixture containing 1.0 mL of 1×10^{-4} mol L^{-1} palladium(II), 2.0 mL of suitable buffer, 1.0 mL of 1×10^{-3} mol L^{-1} 2,6-DAPBPTSC solution is taken and the volume of the aqueous phase is adjusted to 10.0 mL with double distilled water. It is shaken with 10.0 mL portion of isoamylalcohol for one minute. The organic phase is collected into a 25 mL standard flask and made up to the mark with isoamylalcohol. The same procedure is applied for buffers of different pH values, ranging from 1.0 to 6.0. The absorbances are measured at 410 nm, using their corresponding reagent blanks. A plot is executed between the pH and the absorbance, and the same is represented in Fig. 2. The plot shows that there is maximum absorbance and constancy in the pH range 3.5-4.5. Hence, pH 4.0 is chosen for further studies, considering this as an optimum pH.

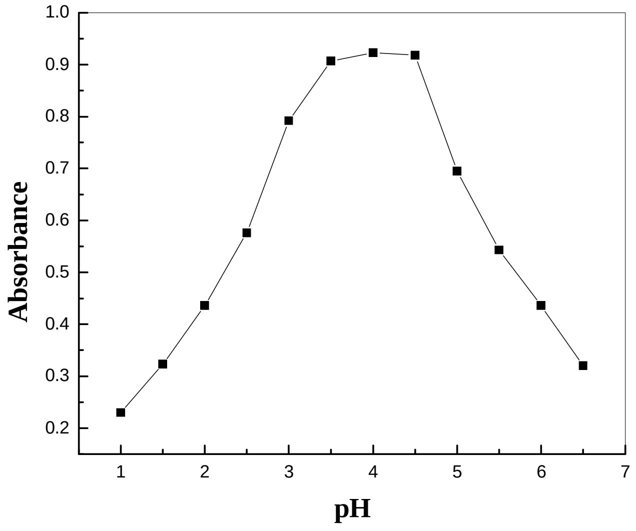


Fig. 2. Effect of pH on Pd(II)- 2,6-DAPBPTSC complex: 1 mL of (1.0 × 10^{-4} mol L^{-1}) Pd(II);1.0 mL of (1.0 × 10^{-3} mol L^{-1}) 2,6-DAPBPTSC; λ_{max}: 410 nm

Effect of solvents on extraction of Pd(II)-2,6-DAPBPTSC complex: The effect of various solvents such as isoamylalcohol, chloroform, toluene, benzene, n-butanol, carbon tetrachloride, ethyl acetate, butyl acetate, xylene, tributyl phosphate, n-propyl alcohol and methyl isobutyl ketone on the extraction of palladium(II) with 2,6-DAPBPTSC is studied adopting the standard procedure for measuring the effect of pH. Among the various solvents studied, isoamylalcohol is selected as the suitable solvent, because of its maximum absorbance and greater extraction ability.

Effect of reagent on extraction of Pd(II)-2,6-DAPBPTSC complex: The effect of reagent concentration is studied, using different aliquots containing constant volumes of 1.0 × 10^{-4} mol L^{-1} palladium(II) solution, 2.0 mL of pH 4.0 buffer solution and 1.0 mL of 2,6-DAPBPTSC solution containing different concentrations ranging from 1 ×1 0^{-4} to 20 × 10^{-4} mol L^{-1}, in order to obtain the maximum color formation. The total volumes of aqueous phases are brought to 10.0 mL with double distilled water. The aqueous phases are shaken with 10.0 mL of isoamylalcohol in each case and the organic phases are collected into 25.0 mL standard flasks. The organic phases were made up to 25.0 mL with isoamylalcohol and the absorbances of these phases were measured at 410 nm, against the reagent blank. It is clearly observed from the absorbance values, that a maximum sixteen-fold molar excess of the reagent is sufficient to get a maximum color formation of the complex.

Effect of salting-out agent on extraction of Pd(II)-2,6-DAPBPTSC Complex: Various salting-out agents, such as magnesium sulfate, lithium nitrate, lithium sulfate, lithium chloride, ammonium chloride and ammonium sulfate were attempted, in order to enhance the metal complex extraction into the organic phase in a single step. It was noticed that the addition of these salting out agents had no effect on its absorbance. The aqueous phase is tested for the compete extraction of Pd(II) by using dithizone, which confirmed the absence of Pd(II) in it.

Validity of beer's law, molar absorptivity, sandells' sensitivity and correlation coefficient for Pd(II)-2,6-DAPBPTSC complex: A well-known equation for extractive spectrophotometric analysis for the determination of palladium(II) at very low concentrations has been derived from Beer's Law. The effect of metal concentration has been studied in the range 0.0-12.6 μg mL^{-1} in Fig. 3. The molar absorptivity and the Sandell's sensitivity are found to be 1.156 × 10^4 L $mol^{-1}cm^{-1}$ and 0.0092 μg cm^{-2} of palladium(II), respectively. The correlation coefficient value of the Pd(II)-2,6-DAPBPTSC complex, with an independent variable as concentration in μg mL^{-1} and a dependent variable as absorbance, was found to be 0.962. This indicates a clear linearity between the two variables. The values of the slope and intercept for the best fitted line were obtained as 1.720 and -0.665, respectively. Thus, the content of Pd(II) in real samples can be determined using the following straight line equation: Y= 1.720 X + 0.665.

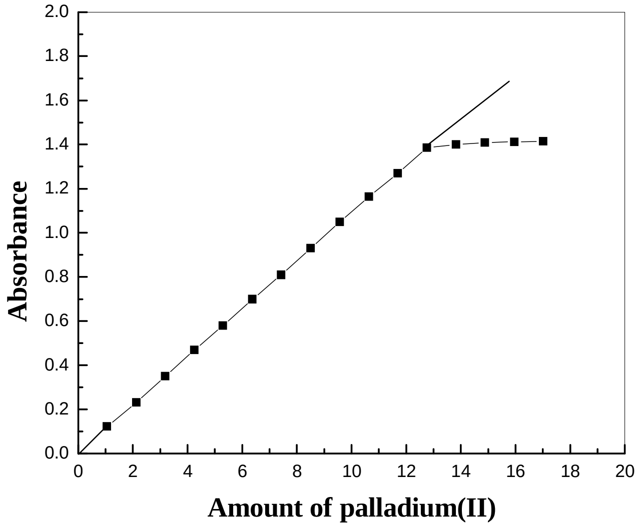


Fig. 3. Applicability of Beer's Law on Pd(II)- 2,6-DAPBPTSC complex:1.0 mL of (16 × 10^{-4} mol L^{-1}) 2,6-DAPBPTSC; 2.0 mL of pH 4.0; λ_{max}: 410 nm

Ringbom plot for Pd(II)–2,6-DAPBPTSC complex: The Ringbom plot is drawn between log C of palladium(II) and (1-T), where " T " is the transmittance, shown in Fig. 4. The plot has a sigmoid shape with linearity at intermediate concentration values from 3.02- 4.02 μgL^{-1}, which indicates that palladium(II) is precisely determined in the optimum concentration range 3.02- 4.02 μg L^{-1}. The slope of the Ringbom plot is 0.74. Based on this value at 1% photometric error, the ratio between the relative error in concentration and the photometric error is 3.11. Hence, the relative error in concentration is 0.031.

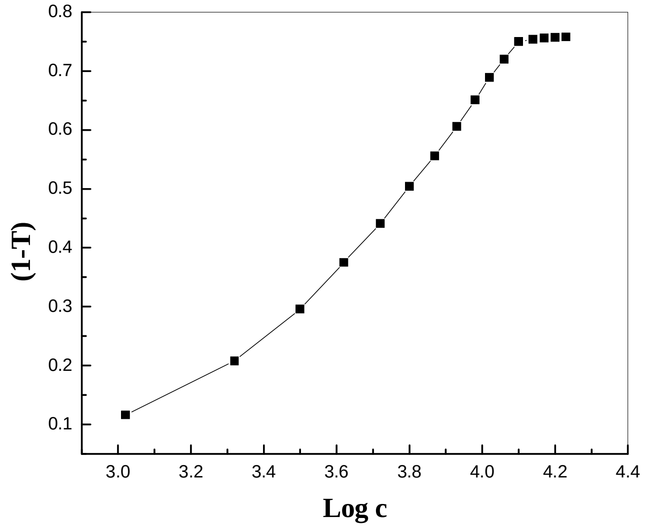


Fig 4. Ringbom plot of Pd(II)- 2,6-DAPBPTSC complex: 1.064- 17.02 μg L^{-1} Pd(II); 1.0 mL of (16 × 10^{-4} mol L^{-1}) 2,6-DAPBPTSC; 2.0 mL of pH 4.0; λ_{max}: 410 nm

Precision, accuracy and detection limit of the method: To assess the precision and accuracy of the method, determinations were carried out for a set of five measurements, with different concentrations of Pd(II), under optimum conditions. The results show that the standard deviation of the method is not more than 0.001 and relative standard deviation is less than 0.371 percent. These results indicate that this method has good precision, besides being accurate. The detection limit, C_{min} is determined as the amount of Pd(II) corresponding to three times the standard deviation of the blank values and a value of 0.00785 μg mL^{-1} is obtained.

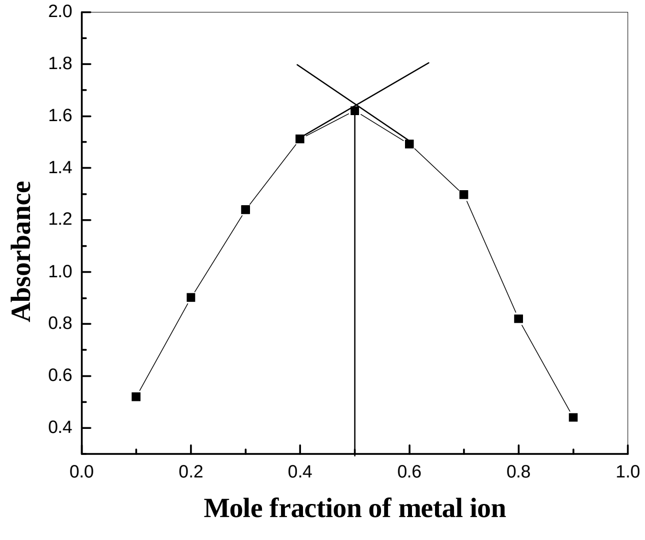


Fig. 5. Job's method of continuous variation of Pd(II)- 2,6-DAPBPTSC Complex: 1.0 mL of (16 × 10^{-4} mol L^{-1}) Pd(II) and 2,6-DAPBPTSC; 2.0 mL of pH 4.0; λ_{max}: 410 nm

Determination of the composition of Pd(II)-2,6-DAPBPTSC complex: Job's method of continuous variation, mole ratio methods were employed to elucidate the composition of the complex. Equimolar solutions of palladium(II) and 2,6-DAPBPTSC (16 × 10^{-4} mol L^{-1}) were used to determine the metal to ligand ratio by job's method of continuous variation. The absorbance values were recorded at 410 nm against the reagent blank. A plot (Fig. 5) was drawn between the absorbance and V_M / V_L+V_M, where V_L and V_M are the volumes of the reagent and the metal, respectively. The obtained curve indicates that 1:1 (M: L) stoichiometry in the extracted species. This was further confirmed by the mole ratio method by using the solution containing 1.0 mL of palladium(II) (1.12 × 10^{-3} mol L^{-1}) 2.0 mL of buffer (pH 4.0) and 1.0 mL of 2,6-DAPBPTSC (1.0 × 10^{-3} mol L^{-1}) solution. The absorbance values were recorded at 410 nm against their respective reagent blanks. A plot (Fig. 6) was drawn between the absorbance and the volume of the reagent. From the curve it is noticed that one mole of the palladium(II) reacts with one mole of 2,6-DAPBPTSC.

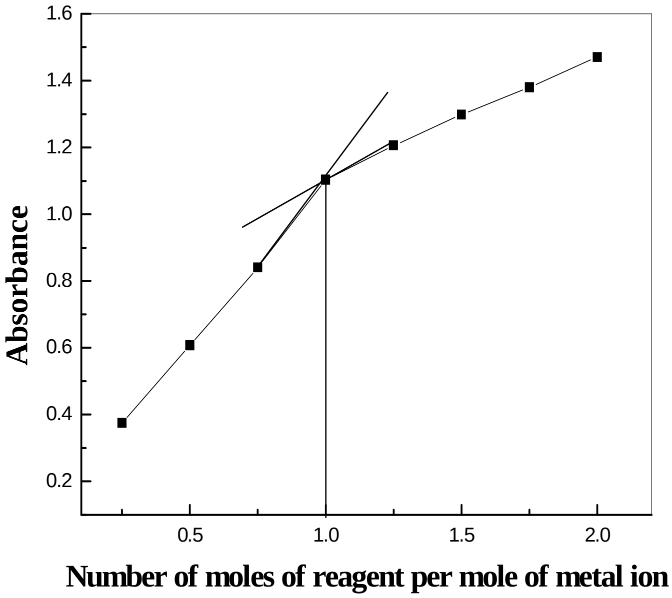


Fig. 6. Mole ratio method Pd(II)- 2,6-DAPBPTSC complex: 1.0 mL of (1.12 × 10^{-3} mol L^{-1}) Pd(II); 1 mL of (1.0 × 10^{-3} mol L^{-1}) 2,6-DAPBPTSC; 2 mL of pH: 4.0; λ_{max}: 410 nm

Finally, the composition of the complex was verified by Asmu's method. The experimental part of Asmu's method is similar to that of the mole ratio method. A liner plot was obtained between 1/m and 1/v, where 'm' is the extraction modules, which is calculated by dividing the optical density with the cell width and 'v' is the volume of the reagent. This plot also confirms the metal to ligand ratio to be 1:1.

Determination of instability constant of Pd(II)-2,6-DAPBPTSC complex: The instability constant of Pd(II)- 2,6-DAPBPTSC complex was calculated using as Asmus' method. The absorbance values of organic extracts were obtained at 410 nm the solutions containing fixed volumes of palladium(II) (1.0 mL of 1.12 × 10^{-3} mol L^{-1}) and pH : 4.0 buffer with different known volumes of 0.25 - 2.0 mL of 1.0 × 10^{-3} mol L^{-1} of 2,6-DAPBPTSC in the presence isoamylalcohol. The instability constant of Pd(II)-2,6-DAPBPTSC complex was calculated to be 1.667 × 10^{-4} at room temperature.

Effect of foreign ions on extraction of Pd(II)- 2,6-DAPBPTSC complex: Interference of a number of cations and anions is studied is the color absorbance of the Pd(II)-2,6-DAPBPTSC complex. A change in absorbance of ± 0.0025 is taken as the tolerance limit for interference. Cations like Be(II), Mg(II), Mn(II), Ti(IV) and Cr(IV) which do not interfere, even when present up to 5000 µg . Al(III), Fe(II), W(IV) and Zr(IV) do not have any effect when present up to 1000 µg , where as Pb(II), Hg(II) Cd(II), Ni(II), Cu(II), Zn(II), U(VI), Se(IV) and Ce(IV) interfere seriously, even when present at trace levels. Anions like ascorbate, tartrate, citrate, and carbonate do not interference when present up to 5000 µg, while fluoride, chloride, bromide and iodide tolerate up to 2500 µg. The interference of Cu(II), U(VI) and Ce(IV) was suppressed by using 1.0 mL of 2 % oxalate solution as a masking agent. Ni(II), Zn(II), tolerate with 1.0 mL of 2 % EDTA as a masking agent, where as Cd(II) was masked with 1.0 mL of 1% thiocyanate solution. Pb(II), Hg(II) and Se(IV) were masked with 2 .0 mL of 1 % phosphate solution (Table 2).

Applications of developed method: The developed sensitive extractive spectrophotometric method for Pd(II) was successfully applied for its determination in spiked samples and results were presented in Tables 3, 4 and 5 the obtained results were compared with reported method [27] in terms of student's "*t* " test and various ratio "*f* " test.

Table 2. Effect of Foreign Ions on the extraction of Pd(II)- 2,6-DAPBPTSC complex

Foreign Ion	Tolerance Limit, µg/ml	Foreign Ion	Tolerance Limit, µg/ml
Be(II)	5000	Se(IV)	-
Mg(II)	5000	Ce(IV)	-
Mn(II)	5000	Carbonate	5000
Ti(IV)	5000	Ascorbate	5000
Cr(IV)	5000	Tartrate	5000
Al(III)	1000	Citrate	5000
Fe(II)	1000	Fluoride	2500
W(IV)	1000	Chloride	2500
Zr(IV)	1000	Bromide	2500
Pb(II)	-	Iodide	2500
Hg(II)	-		
Cd(II)	-		
Ni(II)	-		
Cu(II)	-		
Zn(II)	-		
U(VI)	-		

Table 3. Determination of Pd(II) in water samples

S. No	Palladium (II) added µg.mL^{-1}	Palladium(II)[a] found		S.D.	R.S.D, %	T-test	F-test
		Present method	Reported method				
Sample 1	6.0	5.69	5.67	0.02	0. 342	7.86	4.89
Sample 2	8.0	7.62	7.56	0.032	0.418	4.92	2.59
Sample 3	10.0	9.83	9.75	0.036	0.363	2.00	5.25
Sample 4	11.0	11.92	11.85	0.032	0.271	2.20	3.82

[a] *Average of five determinations*

Table 4. Determination of Pd(II) in synthetic mixtures

Synthetic mixtu- re composition (µg mL^{-1})	Palladium (II) added µg mL^{-1}	Palladium(II)[a] found		S.D.	R.S.D, %	T-test	F-test
		Present method	Reported method				
25 Fe(III) + 12.5 Co(II) + 25 Cu(II)	3.00	2.98	2.99	0.008	0.27	1.17	4.67
	4.00	3.98	3.99	0.010	0.25	0.89	5.33
	5.00	4.99	4.98	0.012	0.24	1.73	4.97
2.0 Fe(III) + 10 Ru(III) + 5 Pb(IV) +10 Ir(IV)	3.00	2.97	2.98	0.012	0.41	0.85	3.58
	4.00	3.98	3.97	0.016	0.39	0.42	1.19
	5.00	4.98	4.97	0.016	0.32	0.59	4.79
20 Ni(II) + 10 Co(II) +5.0 Au(II)	10.0	9.98	9.96	0.012	0.12	1.71	3.58
25 Pt(IV) +10 Ru(III) +20 Ir(IV) +20 Rh(III) + 10 Zr(IV)	4.00	3.97	3.98	0.018	0.45	0.66	2.55
	5.00	4.97	4.98	0.016	0.32	0.59	4.79
	6.00	5.96	5.97	0.016	0.27	0.51	5.99
2.0 Ni(II) +12 V(V) + 5 Pb(IV)	6.00	5.98	5.97	0.017	0.29	0.51	5.45
	7.00	6.97	6.99	0.021	0.30	1.10	2.66
	8.00	7.95	7.98	0.026	0.32	1.27	3.18

[a] *Average of five determinations*

Table 5. Determination of Pd(II) in hydrogenation catalysts

Catalyst	Palladium(II) Claimed,%	Palladium(II)[a] found		S.D.	R.S.D, %	T-test	F-test
		Present method	Reported method				
Pd-CaCo$_3$	5.00	4.95	4.98	0.021	0.43	1.31	4.54
Pd-BaCo$_3$	5.00	4.96	4.99	0.015	0.31	2.03	3.34
Pd-BaSo$_4$	5.00	4.98	4.97	0.019	0.38	0.56	3.21
Pd-Activated charcoal	10.00	9.95	9.98	0.021	0.21	1.20	5.71

[a] *Average of five determinations*

CONCLUSION

The present investigations proved that 2,6-DAPBPTSC is a Promising complexing agent for Pd(II) and its subsequent determination by extractive spectrophotometry was rapid and precise. The method has good sensitivity when compared to other existing extractive spectrophotometric determination methods. The method was more selective in presence of EDTA, Oxalate and phosphate because cations like Ni(II), Zn(II), Cd(II), Cu(II), U(VI), Pb(II), and Hg(II), if present, do not interfere with extraction of Pd(II). It has been found that only a sixteen-fold excess of reagent is sufficient for the quantitative extraction of palladium(II). A further advantage of 2,6-DAPBPTSC over other available spectrophotometric reagents is the determination of Pd(II) in the presence of other noble metals. The method has been successfully applied for the determination of Pd(II) in spiked samples.

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