



Research Article

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Extractive and spectrophotometric determination of palladium (II) using 2-(5-bromo-2-oxoindolin-3-ylidene)hydrazinecarbothioamide as an analytical reagent

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ABSTRACT

2-(5-bromo-2-oxoindolin-3-ylidene)hydrazinecarbothioamide, [5-Bromoisatin thiosemicarbazone (HBITSC)] extracts palladium (II) quantitatively (99.90%) into n-amyl alcohol from an aqueous solution of pH 0.0 - 4.0 and from 0.1 - 3.0M solution of hydrochloric acid. The n-amyl alcohol extract shows an intense peak at 520nm (λ max). Beer's law is obeyed over the Pd (II) concentration range 1.0 - 30 μ g/ml. The molar absorptivity is $7450 \text{ L mole}^{-1} \text{ cm}^{-1}$ at 520 nm and Sandel's sensitivity is 14.3 ng/cm^2 . The composition of extracted species is found to be 1:2 (Pd : HBITSC) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method has been applied for determination of Pd (II) in catalyst samples.

Key words: HBITSC, Palladium, Spectrophotometry.

INTRODUCTION

Various reagents¹⁻¹⁹ are available for the spectrophotometric determination of palladium. In the present communication, we describe the extractive spectrophotometric determination of Pd (II) with [5-Bromoisatin thiosemicarbazone (HBITSC)]

EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO LI-127 pH meter was employed for pH measurements. The reagent HBITSC was synthesized by condensations of 5-Bromoisatin and Thiosemicarbazide procedure recommended by Vogel¹⁸ and characterized by elemental and spectral analysis. Its solution was prepared in Dimethylformamide (DMF). A stock solution of Pd (II) was prepared by dissolving palladium chloride in water containing dilute hydrochloric acid; it was standardized by dimethylglyoxime method¹⁹. Working solutions of Pd (II) were made by suitable dilutions. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extractive Spectrophotometric Determination Of Pd (II):

To an aliquot of aqueous solution containing 10-300 μ g of Pd (II), 5ml of 2M hydrochloric acid, and 1ml of 0.5% solution of HBITSC prepared in DMF added. The volume of solution was made up to 10 ml with distilled water and then equilibrated for 1 min with 10 ml of n-amyl alcohol and the phases were allowed to separate. The n-amyl alcohol extract was collected in a 10 ml measuring flask and made up to mark with n-amyl alcohol. The absorbance

of n-amyl alcohol extract was measured at 520 nm against a reagent blank prepared under identical conditions. The Palladium content of the sample solution was determined from calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH or acidity.

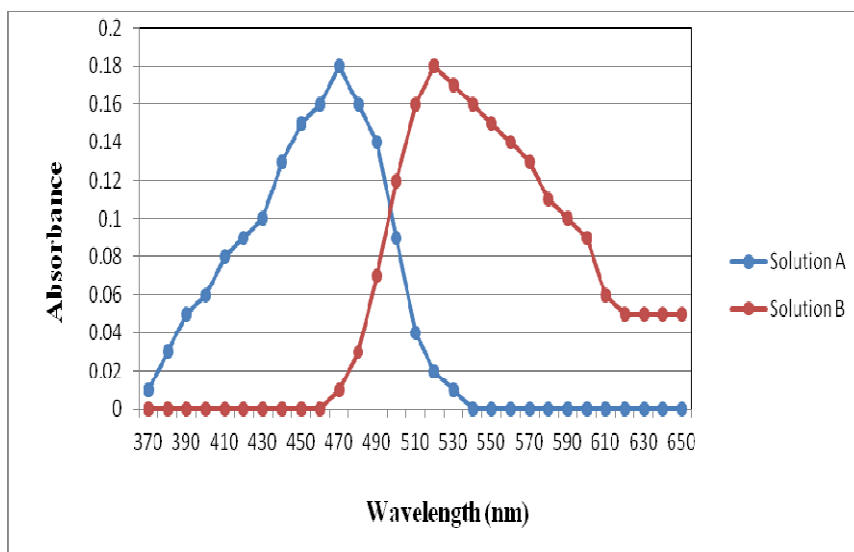
Determination of Pd (II) in catalyst sample (Pd-charcoal, Pd- BaSO₄ catalyst):

0.1 to 0.2 gm. sample of catalyst was dissolved in 10 ml of aquaregia. The solution was evaporated to dryness and the residue was dissolved in 10 ml of 1N HCl filter, if required and resulting solution was diluted to 100 ml with water. 1ml aliquot of this solution was analyzed for Pd (II) by the procedure as described earlier.

RESULTS AND DISCUSSION

Palladium (II) could be extracted quantitatively (99.90%) by HBITSC into n-amyl alcohol from an aqueous solution of pH 0.0 to 4.0 and from 0.1 to 3.0M solution of hydrochloric acid. Organic solvents used for extraction of Pd (II) can be arranged on the basis of their extraction coefficient values as n-amyl alcohol > n-butanol > ethyl acetate > benzyl alcohol > chloroform > carbon tetrachloride > toluene > nitrobenzene.> xylene> chlorobenzene. n-amyl alcohol was found to be the best extracting solvent; hence, it was selected for extraction throughout the work. The n-amyl alcohol extract of Pd - HBITSC complex showed intense peak at 520 nm.(Fig.1). The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength. The result shows that the system confirmed to Beer's law at this wavelength over a palladium concentration range 1.0 to 30 ug/ml. The molar absorptivity of the extracted complex on the basis of Pd (II) content was calculated to be 7450 L mol⁻¹ cm⁻¹. The Sandel's sensitivity was found to be 14.3 ng/cm². It was found that 1 ml of 0.5% DMF solution of HBITSC was sufficient to extract 300 ug of Pd (II). The colour of the n-amyl alcohol extract was found to be stable at least 48 hrs at room temperature.

Figure 1



Solution A : Absorbance spectra of HBITSC
Solution B : Absorbance spectra of Pd - HBITSC Complex

Effect Of Other Ions:

Pd (II) (100 µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Pd (II) (100 µg) : 10 mg each of Ca(II), Mg(II), Ba(II), Sr(II), Li(I), Pb(II), Sn(II), Be(II), W(VI), Mo(VI), U(VI), V(V), Mn(II), Fe(II), Fe(III), Ni(II), Cu(II), Co(II), Zn(II). 5mg each of Hg(II), Cd(II), Al(III), As(III), Bi(III), Sb(III) and Cr(III). 2mg each of Ce(IV), Th(IV) and Zr(IV). 1mg each of Pt(IV), Ru(III), Rh(III) and Ir(IV). 0.5 mg of Os(IV) .

20 mg each of chloride, bromide, fluoride, sulphate, phosphates, acetate, oxalate, nitrate, citrate, persulphate and tartarate. Interference due to E.D.T.A., iodide, nitrite and thiosulphate were removed by boiling the solution with concentrated nitric acid before the adjustment of pH or acidity.

Composition Of The Extracted Complex:

The composition of the extracted complex was found to be 1:2 (Pd: HBITSC) by Job's continuous variation and Mole ratio methods.

Precision, Accuracy, Sensitivity and Application of Method:

The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Pd (II) following the recommended procedure. The average of 10 determination of 20 µg of Pd (II) in 10 cm³ solutions was 20.2 µg, which is varied between 20.057 µg and 20.343 µg at 95% confidence limit and standard deviation was ±0.2. **The proposed method has been applied for the determination of Pd (II) in catalyst.**

The results of the analysis of the samples were comparable with those obtained by the Dimethyl glyoxime method¹⁹ for Pd (II) (Table-1).

Table – 1 Determination Of Pd (II) catalyst samples

Sample	Pd (II) found % Present method	Dimethyl glyoxime Method ¹⁹
Pd charcoal catalyst	4.97%	4.993%
Pd - BaSO ₄ catalyst	0.88%	0.89%

*Average of three determinations

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