elements parameters x and u. However, the solution obtained in this case was in sharp disagreement with the modified B approximation. The solution obtained with $A = 2{\cdot}5$ was in general agreement with the modified B_1 approximation. The energy dependence of shape factor due to the best set of matrix elements reported by Bhattacharjee *et al.*[21], and due to the first set of matrix elements due to Manthuruthil[7] do not follow the present shape factor. Those due to Appalacharyulu[3] who employed the formalism of Buhring[19,20], in which the finite nuclear size effect and the higher order effects are included is in good agreement as shown in Fig. 5.

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SPECTROPHOTOMETRIC STUDY ON THE COMPLEXATION REACTION OF PALLADIUM(II) WITH BUTAPERAZINE DIMALEATE

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ABSTRACT

Butaperazine dimaleate forms a red coloured 1 : 1 complex with palladium(II) in hydrochloric acid-sodium acetate buffer. The complex exhibits absorption maximum at 490 nm with molar absorptivity $3{\cdot}5 \times 10^3$ litre mole^{-1} cm^{-1}. Beer's law is valid over the concentration range 0·2–17·0 μg/ml.

INTRODUCTION

IN the present communication, the authors propose the reaction of butaperazine dimaleate (BPDM) with palladium(II) for the spectrophotometric determination of palladium(II). The method offers the advantages of simplicity, rapidity, selectivity and wider range of determination without the need for extraction.

EXPERIMENTAL

Reagents

A stock solution of palladium(II) was prepared by dissolving 0·9980 g of palladium(II) chloride (M/s Johnson Matthey Chemicals, London) in 1 litre of 0·1 M HCl and was standardized gravimetrically by the dimethylglyoxime method[1]. A 0·2% solution of BPDM was prepared in hot double distilled water and stored in an amber bottle in a refrigerator. Sodium acetate-hydrochloric acid buffers were used. Beckman spectrophotometer Model DB was used for absorbance measurements.

Procedure for the Determination of Palladium(II)

An aliquot of the stock solution containing 5·0–425 μg of palladium, 5 ml of hydrochloric acid-

sodium acetate buffer (pH 1·5) and 2·0 ml of 0·2% BPDM were made up to 25 ml in volumetric flask with double distilled water. The solution was mixed well and the absorbance was measured at 490 nm against a corresponding reagent blank. The amount of palladium in the sample solution was then deduced from the standard calibration curve.

Results and Discussion

The aqueous solution of BPDM is light yellow having maximum absorbance at 375 nm. BPDM forms a red coloured complex with palladium(II) instantaneously at room temperature (26° C) in the presence of hydrochloric, sulphuric and phosphoric acids or with buffer solutions of low pH. The effective pH range where reproducible results are obtained is 1·3–2·2 as shown by the constancy of the λ and the absorbance. Below and above this pH range, maximum colour intensity of the complex is not obtained. A buffer medium of pH 1·5 has been selected for further studies. The red complex exhibits maximum absorption at 482–500 nm where the reagent and palladium(II) do not appreciably absorb. Absorption measurements are therefore made at 490 nm.

A six-fold molar excess of the reagent over palladium is required in order to obtain maximum absorbance. The absorbance readings were constant in the temperature range 5–45° C. Above 45° C, the absorbance decreased. The order of the addition of reagents was not critical. Beer's law is obeyed in the range 0·2–17·0 μg/ml of palladium. The optimum concentration range for the effective spectrophotometric determination evaluated by Ringbom's method is 2·0–16·6 μg/ml. The Sandell's sensitivity of the reaction as calculated from Beer's law data is 30 ng/cm² and the molar absorptivity is $3·53 \times 10^3$ litre mole^{-1} cm^{-1}. The mean value for eight different samples each containing 5 ppm of palladium has been found to be 4·974 ppm with a standard deviation of 0·0463. The accuracy of the method is about ± 2%.

Composition and Nature of the Complex

Job's method of continuous variation[3], mole ratio method[4] and slope ratio method[5] indicated the formation of 1 : 1 complex between the metal and the reagent. The value of the apparent stability constant, obtained by the mole ratio method for the Pd–BPDM complex at 26 ± 1° C, pH 1·5 ± 0·1 and ionic strength 0·1 M $NaNO_3$ was 5·3318. The complex was found to be cationic by ion exchange experiments.

Effect of Diverse Ions

In order to assess the possible analytical applications of this coloured reaction, the effect of foreign ions which often accompany Pd(II) has been studied with 8 ppm of Pd(II). The amounts of foreign ions which gave less than 2% error in absorbance reading are given in Table I.

Table I

Effect of diverse ions

[Amount of Pd (II) taken, 8 μg/ml]

Ion added	Tolerance limit (μg/ml)	Ion added	Tolerance limit (μg/ml)
Cs (VIII)	8	Chloride	3600
Pt (IV)	10	Bromide	3120
Ru (III)	1	Iodide	0·3
Rh (III)	4	Nitrate	2500
Ir (III)	5	Thiosulphate	0·1
Fe (III)	0·8	Sulphate	18700
Co (II)	350	Phosphate	1830
Ni (II)	600	Acetate	240
Cu (II)	500	Tartrate	600
Au (III)	0·2	Citrate	1000
Ce (IV)	0·2	Oxalate	960
U (VI)	500		
Fluoride	4000		

Acknowledgements

The authors thank M/s VEB Arzneimittelwerk Dresden, West Germany, for supply of pure BPDM. One of the authors (K. A. P.) is grateful to UGC, New Delhi and the Mysore University, for the award of Teacher Fellowship under F.I.P.

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