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NOTE

Spectrophotometric Determination of Nickel with 3-Hydroxy-3-*n*-Propyl-1-*p*-Carboxy-Phenyltriazene

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The reagent 3-hydroxy-3-*n*-propyl-1-*p*-carboxyphenyltriazene has been used for spectrophotometric determination of Ni(II). It forms a light green ethanol soluble complex with Ni(II) in the ratio 1 : 2 (NiR_2), with absorption maximum at 380 nm and optimum pH 6.8 to 8.0. The molar absorptivity and Sandell's sensitivity values are 7000 dm^3 mol^{-1} cm^{-1} and 8.3 ng/cm^2 respectively. Beer's law is obeyed over the concentration range of 0.3 ppm to 1.7 ppm of Ni(II). The stability constant has been worked out to be $\log \beta = 9.0$. Standard deviation (10 determinations) for 4.69 ppm of Ni(II) has been found to be ±0.007 ppm. Interference studies have also been done.

Hydroxytriazenes have been widely used for the spectrophotometric determination of a number of transition metals in our laboratory during last many years. Literature survey reveals that their utility as spectrophotometric reagents has been well established[1–6]. The present paper deals with the reagent 3-hydroxy-3-*n*-propyl-1-*p*-carboxyphenyl-triazene in the spectrophotometric determination of Ni(II).

Preparation of 3-hydroxy-3-*n*-propyl-1-*p*-carboxyphenyltriazene 3-Nitropropane (25 mL in 250 mL water) was reduced with zinc dust (30 g) in the presence of NH_4Cl (30 g) at 0–15°C to obtain *n*-propylhydroxylamine. The diazotized product was obtained by adding sodium nitrite (13.8 g) to *p*-aminobenzoic acid (34.28 g) dissolved in 50 mL HCl and 50 mL water in small lots at 0–5°C under constant mechanical stirring.

The diazonium compound was coupled with *n*-propylhydroxylamine at 0–5°C under mechanical stirring with occasional addition of sodium acetate solution for maintaining pH close to 5 during coupling process. The hydroxytriazene was obtained as yellowish brown micro-crystals after crystallization from ethyl alcohol.

TABLE-1
ELEMENTAL ANALYSIS OF LIGAND

	% Analysis		
	C	H	N
Theoretical	53.81	5.87	18.81
Experimental	53.69	5.88	18.89

The 1×10^{-3} M stock solution of reagent was prepared in ethanol while $NiSO_4 \cdot 6H_2O$ (AR) was used to prepare 5×10^{-2} M of Ni(II) in water. The latter was standardized with EDTA at pH 9 to 10 using murexide as indicator. Weaker solutions of Ni(II) were prepared by proper dilution. The pH was adjusted using tris-buffer or perchloric acid.

The pH measurements were made on a Systronics-324 pH-meter, and for absorbance measurements, a Beckman DU-2 and Bausch and Lomb Spectronic-21 spectrophotometer was used.

The reagent forms an ethanol-soluble light green coloured complex with nickel in the pH range 6.8 to 8.0. The λ_{max} and working wavelength for the complex is at 380 nm. Colour development is instantaneous and maximum colour development takes place when the reagent is in tenfold excess. Validity of Beer's law is observed in the range 0.3 ppm to 1.7 ppm of nickel. Molar absorptivity and Sandell's sensitivity values were found to be 7000 $dm^3\ mol^{-1}\ cm^{-1}$ and 8.39 ng/cm^2 respectively. Standard deviation was found to be ±0.007 ppm for 4.69 ppm of Ni(II) (10 determinations). Composition of the complex was found to be 1 : 2 (NiR_2) by Job's method[6], slope ratio method[7] and mole ratio method[8, 9]. The conditional stability constant was determined spectrophotometrically by Harvey and Manning method ($\log \beta = 9.0$).

As for interferences 4.69 ppm of nickel could be determined even in the presence of 100 ppm of Na(I), K(I), Ba(II), Mg(II), Cd(II), CH_3COO^-, F^-, Cl^-, Br^-, I^-, and SO_4^{2-}. However, 5 ppm of NH_4^+, Sn(II), Mn(II), Zn(II), Co(II), Th(IV), Mo(VI), oxalate, CO_3^{2-}, NO_3^-, NO_2^-, PO_4^{3-} tartarate ions were found to interfere with the determination of same quantity of nickel (4.69 ppm).

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