

NOTES

**Direct Spectrophotometric Determination of Nickel with
3-Hydroxy-3-Isopropyl-1-*p*-Chlorophenyl Triazene**

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In the present note, the spectrophotometric determination of nickel(II) with 3-hydroxy-3-isopropyl-1-*p*-chloro phenyl triazenes has been described.

The synthesis of 3-hydroxy-3-isopropyl-1-*p*-chloro phenyl triazene has been reported by Purohit and Golwalkar¹ and it has been used for the spectrophotometric² and complexometric³ determination of iron. The IR spectral studies⁴ and determination of pK value⁵ of this reagent has also been reported. The present communication deals with spectrophotometric determination of nickel with this reagent.

Systronic UV-VIS spectrophotometer-108 has been used for absorbance measurements and Systronics pH meter-324 has been used for measuring pH.

Solution of Ni(II), 1×10^{-2} M, was prepared and standardized with EDTA using murexide⁶ as indicator. Weaker solutions were prepared from it by proper dilution. The reagent solutions were prepared by dissolving the requisite quantity in ethanol. The desired pH was adjusted using 1% aqueous tris buffer and 1% ethanolic perchloric acid.

The leaf-green nickel complex exhibited λ_{max} at 395 nm and the working wavelength was taken as 415 nm. Full colour develops after 3/4 hrs of mixing Ni(II) and reagent solution. The solutions were made up to the desired volume in ethanol. The colour was stable for 3 hrs. Maximum colour develops when eightfold excess of reagent was taken and pH was maintained between 8.0 to 8.4. Absorbance was taken against reagent blank.

The four methods—Job's method⁷, Slope ratio method⁸ and Mole ratio method of Yoe and Jones⁹ and of Zolotov¹⁰—gave the complex composition as 1 : 2 (Ni : R). Beer's law is obeyed in the entire concentration range studied i.e. 2.93 ppm to 17.61 ppm. Molar absorptivity value is 2,387 l/mole cm and Sandell's sensitivity value is 24.59 ng/cm². Standard deviation using 14.67 ppm of nickel was found as 0.05 ppm (10 determinations) from precision studies. The conditional

stability constant was found using Harvey and Manning's curve as 10.72 and from Job's curves as 10.83.

The interference studies revealed that 14.67 ppm of nickel can be determined in presence of 5 ppm of Na(I), K(I), Ag(I), Ca(II), Mg(II), Ba(II), Mn(II), Hg(II), Cd(II), F⁻, Cl⁻, Br⁻, I⁻, NO₃⁻, CH₃COO⁻, CO₃²⁻, and SO₄²⁻. Further, 5 ppm of Co(II), Cu(II), Zn(II), Cr(III), PO₄³⁻, oxalate and molybdate were found to interfere.

The solid complex was obtained as leaf green microcrystals, m.pt. 115°C. Analysis for C, H, N, Cl and Ni elements of the complex [molecular formula, Ni (C₁₈H₂₂N₆O₂Cl₂)] was obtained as:

	% C	% H	% N	% Cl	% Ni
Experimental values	43.66	5.43	17.29	15.69	12.09
Theoretical values	44.73	4.58	17.38	14.65	12.12

The molecular formula corroborates the composition of the complex found with solution studies.

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