

## NOTES

### 3-Hydroxy-3-*p*-Tolyl-1-*o*-Nitrophenyl Triazene in Spectrophotometric Determination of Nickel

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Results of spectrophotometric determination of nickel with 3-hydroxy-3-*p*-tolyl-1-*o*-nitrophenyl triazene are reported here.

Preparation of 3-hydroxy-3-*p*-tolyl-1-*o*-nitro phenyl triazene has been reported by Gebhard and Thompson<sup>1</sup>. So far, this reagent has not been studied for its analytical utility. Results of spectrophotometric determination of nickel with this reagent are reported here. The reagent was prepared by reported method<sup>1</sup>.

Systronics 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)  $1 \times 10^{-2}$  M was prepared and standardized with EDTA using murexide<sup>2</sup> as indicator. Weaker solutions were prepared from it by proper dilution. The reagent solutions were prepared by dissolving the requisite quantity using the saturated aqueous sodium acetate and 1% ethanolic perchloric acid solutions.

The dark brown nickel complex exhibited max at 520 nm and the working wavelength was kept at 530 nm. Full colour developed immediately and remained stable. The solutions were made in ethanol. Maximum colour develops when eight fold excess of reagent was taken and pH was maintained between 6.7 to 6.9.

The four methods -Job's method<sup>3</sup>, slope ratio method<sup>4</sup> and mole ratio method of Yoe and Jones<sup>5</sup> and of Zolotov<sup>6</sup> gave the complex composition as 1 : 2 (Ni : R). Beer's law is obeyed in the entire concentration range studied *i.e.* 1.17 ppm to 7.04 ppm. Molar absorptivity value is found to be 7,46 l/mole and Sandell's sensitivity value is found to be 7.862 ng/cm<sup>2</sup>. Standard deviation using 5.87 ppm of nickel was found as 0.02 ppm from precision studies (10 determinations). The conditional stability constant was found from Harvey and Manning's curve as log B = 9.44 and from Job's curves as log B = 9.92.

Interference study revealed that in determination of 5.87 ppm of nickel, 5 ppm of Na (I), K (I), Ag (I), Ca (II), Mg (II), Ba (II), Hg (II), Cd (II), F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, CH<sub>3</sub>COO<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> do not interfere. Further 5 ppm of Mn (II), Co (II),

Zn (II), Cu (II), Cr (III),  $\text{CO}_3^{2-}$ ,  $\text{PO}_4^{3-}$ , oxalate and molybdate ions were found to interfere.

The solid complex 1 : 2 (Ni : R) was obtained as dark brown micro crystals (m.pt. 160°C). Analysis of C, H, N and Ni for the complex was found as:

	%C	%H	%N	%Ni
Experimental values	52.70	3.99	18.45	9.81
Theoretical values	52.04	3.68	18.63	9.76

The molecular formula ( $\text{NiC}_{26}\text{H}_{22}\text{N}_8\text{O}_6$ ) corroborates the composition of the complex found with solution studies.

### REFERENCES

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