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NOTE

Direct Spectrophotometric Determination of Copper with 3-Hydroxy-3-methyl-1-*p*-methoxyphenyltriazene

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3-Hydroxy-3-methyl-1-*p*-methoxyphenyltriazene was synthesised by Purohit[1]. The present communication deals with the spectrophotometric determination of copper with this reagent.

Results and Discussion

The reagent forms an ethanol-soluble brown complex with copper in the pH range 6–6.6. The λ_{max} for this complex is at 380 nm and the working wavelength was chosen as 430 nm such that subsequent absorbance measurements were made against the solvent blank. Colour development is instantaneous and all the solutions are made upto desired volume with ethanol. Maximum colour development takes place when the reagent is in eight-fold excess. Validity of Beer's law is observed in the range 6.35–38.1 ppm of copper. Molar absorptivity and Sandell's sensitivity were found to be 1340 dm^3 mol^{-1} cm^{-1} and 47.4 ng cm^{-2} respectively. Standard deviation was found to be ± 0.14 for 15.88 ppm of Cu^{II} (10 determinations). Composition of the complex was found to be 1 : 2 (Cu : R) by Job's method[3], slope-ratio method[4] and mole-ratio methods[5,6]. Spectrophotometrically conditional stability constant of the complex was found using Harvey and Manning mole-ratio curve as log β = 9.46.

It was found that 31.77 ppm of copper can be determined in presence of 10 ppm of Na^+, K^+, NH_4^+, chloride, bromide, iodide, acetate, nitrate, sulphate, carbonate, phosphate and molybdate. Further, Cd^{II}, Hg^{II}, Zn^{II}, Mg^{II}, Co^{III}, Ni^{II}, Ba^{II}, Cr^{III}, fluoride and oxalate were found to interfere in determinations of Cu (31.77 ppm) when present in 10 ppm concentration. Copper (31.77 ppm) could be determined in presence of 50 ppm each of the following ions : Na^+, K^+, NH_4^+, chloride, bromide. acetate, nitrate, sulphate, phosphate and molybdate and 100 ppm of the above ions except bromide.

The solid complex was crystallised from ethanol to give brown crystals, m.p. 158° (Found : C, 43.80; N, 19.04; H, 4.20. $C_{14}H_{22}N_8O_5$) calcd for : C, 43.60; N, 19.00; H, 4.70%).This is indicative of the presence of one water molecule in the solid complex. Ir spectra of the complex also exhibited the presence of one water molecule by the appearance of a new band at 3 400–3 500 cm^{-1}.

Experimental

The reagent was prepared as per the reported method[1] and its 1×10^{-2} *M* solution was prepared in ethanol. Solutions of 1×10^{-2} *M* of Cu^{II} was prepared by dissolving the requisite amount of $CuSO_4.5H_2O$ (AnalaR) in water and standardised with EDTA at pH 5–7 using murexide as indicator[2]. Weaker solutions were prepared by appropriate dilution. Sodium acetate (1%, w/v) and perchloric acid (1%, v/v) were used to adjust the desired pH.

A Systronics 108 spectrophotometer was used for absorbance measurements and a Systronics 324 pH meter for pH measurements.

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