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NOTE

Extraction and Spectrophotometric Determination of Copper(II) with 4-Chloroisonitrosoacetophenone Semicarbazone

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Spectrophotometric method has been developed for the determination of copper(II) using 4-chloroisonitrosoacetophenone semicarbazone as a reagent. The reagent reacts with Cu(II) giving a slight blue coloured complex which can be quantitatively extracted into chloroform at pH 7.2–8.0. Molar absorptivity at 386 nm is 1.79517×10^5. Beer's Law is obeyed in the range 0.1 μg to 10 μg. The present method has been used for analysis of Cu(II) in real samples as well as in binary mixtures.

Several isonitroso compounds[1, 2] have been used for the extraction and spectrophotometric determination of metal ions. They reacts with transition metal ions to give coloured complexes[3, 4], which are extractable in organic solvents. In the present investigations the determination of Cu(II) with 4-chloroisonitrosoacetophenone semicarbazone (CINAPSC) has been reported. CINAPSC reacts with Cu(II) to give slightly blue coloured complex which is extractable into chloroform

A Shimadzu UV-Spectrophotometer with matched quartz cells of 1 cm optical path was used for absorbance measurements. The pH values were determined using an ELICO-LI-120 pH meter assembly.

Chemicals and solvents used were all of analytical reagent grade. A stock solution of copper having a strength of 1 mg/mL was prepared by dissolving copper sulphate in double distilled water containing dilute sulphuric acid and diluted to the desired volume with double distilled water. Copper sulphate solution was standardised by the benzoin oxime method[5]. The reagent CINAPSC was synthesized as per the procedure reported in the literature[6]. Standard solutions of diverse ions were prepared from their chloride, nitrate or sulphate or from sodium, potassium or ammonium salts using doubled distilled water.

1 mL aqueous solution containing 1 mg of copper, 2 mL of 1% solution of 4-chloroisonitrosoacetophenone semicarbazone and 2 mL of 0.5 molar solution of sodium acetate were mixed in a 25 mL beaker. The pH of the solution was adjusted to desired value using dilute solution of $NaOH/NH_4OH$. The resulting mixture was equilibrated for 2 minutes with 10 mL of chloroform. The organic

layer was separated and its absorbance was measured at 386 nm against the reagent blank prepared under identical conditions. Amount of copper in unknown solutions were determined from the standard calibration curve. To study the interference the respective foreign ions were added to the aqueous phase before the extraction and pH adjustment.

Copper can be quantitatively extracted by CINAPSC into chloroform from an aqueous solution at pH 7.2 to 8.0. Organic solvents were arranged in the following order on the basis of the extraction coefficient values:

Chloroform > Diethyl ether > Benzene > *n*-Butanol > Toluene > Ethyl methyl-ketone > Carbon tetrachloride > Ethyl acetate > Isoamyl alcohol > Nitrobenzene.

The absorption spectrum of Cu : CINAPSC complex in chloroform shows an absorption maxima around 386 nm. At this wavelength the Beer's law is found to be obeyed over a range of 0.1 μg to 10 ug of copper per mL and the molar absorptivity of the extracted species is 1.79517×10^5 lit mol^{-1} cm^{-1}, calculated on the basis of the total amount of copper taken.

The following ions when present in amounts indicated do not interfere in the spectrophotometric determination of Cu(II) 10 mg each of Li(I), Na(I), K(I), Sr(II), Mn(II); 5 mg each of Ba(II), Ca(II), Mg(II), Pb(II), Al(III), Bi(III), As(III), Mo(IV), V(V), W(VI), ; 10 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphate, persulphate, sulphite, nitrate, nitrite, cyanate, acetate, pyrophospate, perchlorate, thiourea and thiocyanate.

The interference by the ions Ag(I), Pd(II), Zn(II), Ce(IV), Zr(IV), Th(IV), V(VI), cyanide, citrate, tartarate, oxalate and EDTA can be removed by using appropriate masking agents. The composition of the extracted Cu(II) : CINAPSC complex species has been studied by Job's continuous variation and mole ratio methods. The result suggest 1:2 (metal:ligand) stoichiometry for Cu(II). The method has been successfully applied for the determination of copper from the binary mixtures nimonic 901 (BCS387) and from magnesium alloy (BCS-CRM307). The results of the analysis of the alloy samples are found to be in good agreement with those obtained by the standard method.

TABLE-1
ANALYSIS OF COPPER CONTAINING SAMPLES

S. No.	Sample/composition Alloys	Cu Found Present method	Cu Found Known method
1.	Nimonic 901 BCS CRM 387	0.290	0.310
2.	Magnesium (BCS CRM 387)	0.049	0.052
	Synthetic Binary Mixtures		
3.	Mixture No. 1	39.980	40.400
4.	Mixture No. 2	50.000	50.340
5.	Mixture No. 3	99.95	100.050

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