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Analytical Applications of Mixed Ligand Complexes. II. Separation-Gravimetric Determination of Cu(II) as Its Mixed Ligand Complex with 1,10-Phenanthroline and *p*-Cresotic Acid

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NOTE

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Abstract

A method is presented for the gravimetric determination of Cu(II) followed by its separation from Ni(II), Co(II), Mn(II), and other anions and cations by precipitating it as its mixed ligand complex with 1,10-phenanthroline and *p*-cresotic acid (5-methylsalicylic acid). The precipitate is equivalent to 0.184 times its weight of copper. The method has potential for use in the large-scale separation of copper.

The formation of mixed ligand complexes is known to often exert a "synergistic" effect on the solvent extraction of metal ions (*1*) and is also found to be responsible for the enhancement of the color of metal–ligand systems (*2, 3*). A survey (*2, 4*) of the literature reveals that although the realization of the importance of mixed ligand complexes in analytical chemistry has grown considerably in recent years, there are only a few reported analytical methods based on mixed complex formation, and gravimetric methods are fewer still. The present method is the first one

reported involving 1,10-phenanthroline (PHEN) and 5-methylsalicylic acid (MSA). MSA was chosen because the presence of the methyl substituent in salicylic acid enhances the molecular weight of the mixed complex with a consequent enhancement in the sensitivity of the gravimetric method.

EXPERIMENTAL

MSA was purified from iron and other impurities by the method of Chattopadhyaya (*5*). All other chemicals were of reagent grade. Conductivity water was used throughout. The metal ion solutions were standardized with the help of appropriate complexometric titrations (*6*). The pH adjustments were carried out with a radiometer pH meter PHM-29.

Procedure for Separation and Determination

The pH of the copper solution, containing 10 to 100 μg of Cu^{2+}/ml, was adjusted between 3.9 and 4.5 with dilute HNO_3 or NaOH. A water–ethanol solution (5% ethanol) having 2.0×10^{-2} *M* each of PHEN, monosodium salt of MSA, and NaOH was added dropwise to the copper solution until the precipitation of the bright green complex was complete. The precipitate and mother liquor were digested on a water bath for 20 min and then cooled to room temperature. The precipitate was filtered on glazed silica, dried at 35°C, and weighed (conversion factor 0.184).

RESULTS AND DISCUSSION

The fibrous precipitate forms rapidly and settles, leaving a clear supernatent liquid.

The tolerance limits for the determination of 10 ppm of Cu^{2+} were Ni^{2+} (30 ppm), Co^{2+} (30 ppm), Mn^{2+} (50 ppm), Zn^{2+} (50 ppm), Ca^{2+} (100 ppm), Ba^{2+} (100 ppm), Sr^{2+} (100 ppm), Na^{+} (5000 ppm), K^{+} (5000 ppm), Cl^{-} (1000 ppm), SO_4^{-} (2000 ppm), and NO_3^{-} (1000 ppm).

A great advantage of the proposed method is that the precipitate can be readily dissolved in 0.01 *N* HCl or H_2SO_4, and the mixture of PHEN and MSA can be recovered from the solution by solvent extraction with *n*-butanol to be reused for the extraction of more copper. The method thus holds promise for the large-scale separation of copper.

Composition of the Mixed Ligand Complex

The composition was established by potentiometric titrations with a standard KOH solution on a series of solutions containing (a) PHEN, (b) MSA, (c) Cu(II) + PHEN, (d) Cu(II) + MSA, and (e) Cu(II) + PHEN + MSA. An analysis of potentiometric titration curves revealed that a 1:1 Cu(II)–PHEN complex is formed at pH $\simeq$ 2, which adds an MSA molecule at pH $\geq$ 3.9 to form a 1:1:1 Cu–PHEN–MSA mixed complex. The titration curves were obtained and analyzed in a manner similar to the one adopted for Cu–2,2′-bipyridyl–salicylic acid systems (*7*).

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