Volumetric determination of copper by precipitation with ammonium thiocyanate

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Abstract

Potassium dichromate was employed for the determination of copper by precipitation as cuprous thiocyanate. 1.0-10.0 mg of copper was accurately determined by this procedure. The method was applied for the analysis of copper in Devarda's alloy and chalcopyrites.

1. Introduction

Volumetric methods for copper via oxidation of thiocyanate part in cuprous thiocyanate are very few.1-3 Raju and Reddy4 described a potentiometric method for the estimation of thiocyanate using dichromate. This procedure has been made simple by Muralikrishna et al.,5 who have estimated thiocyanate to a visual end point. We have made use of this reagent, viz., potassium dichromate, for the accurate determination of copper by precipitation as cuprous thiocyanate.

2. Experimental

All the reagents used were of AR quality. The stock solution of copper was prepared by dissolving a known weight of copper sulphate in distilled water and diluting to a known volume. The solution was standardised iodometrically.6a

Procedure

An aliquot of the solution containing 1.0-10.0 mg of copper was pipetted out and the copper was precipitated as cuprous thiocyanate using a freshly prepared 10% w/v ammonium thiocyanate solution, either by the conventional method using sulphur dioxide6b or by the procedure described by Raju et al.7 The precipitate was filtered through a No. 4 sintered crucible and washed successively with cold water and 20% ethanol till it was free from soluble thiocyanate. The precipitate in the crucible was quantitatively dissolved with about 15-20 ml of liquor ammonia and the solution was
allowed to drop into a clean buchner flask containing 10 ml of the standard dichromate solution (0·025 M) and 45–50 ml of 9 M sulphuric acid. The crucible was washed with distilled water and the washings collected into the same buchner flask. The flask was allowed to cool and stand at room temperature for about 5–10 min and the excess dichromate was titrated with mohr salt solution using 2–3 drops of 0·025 M ferroin indicator solution. A 10 ml microburette graduated in 0·02 ml was used in these titrations.

By this procedure, 1–10 mg of copper could be conveniently determined with an accuracy of ±0·56%. The reverse addition of dichromate solution to the ammoniacal copper thiocyanate solution resulted in low value of copper.

**ANALYSIS OF COPPER IN DEVARDA'S ALLOY**

The alloy (0·2848 g) was treated with a mixture of sulphuric, nitric and hydrochloric acids and the solution was evaporated to dense white fumes. The residue was cooled, dissolved in just the requisite quantity of dilute sulphuric acid, filtered and made up to 100 ml in a volumetric flask. The copper content in the alloy solution was determined by the iodometric procedure. Several aliquots of the alloy solution were analysed for their copper content using the procedure described above. The percentage of copper in Devarda's alloy as obtained by the present method (44·89) is comparable to that obtained by the iodometric method (44·98).

**ANALYSIS OF COPPER IN CHALCOPYRITES**

The finely powdered ore (0·3309 g) was dissolved in 25 ml of concentrated nitric acid and the solution evaporated to about 5 ml. 10 ml of concentrated hydrochloric acid and 10 ml of sulphuric acid (1:1) were then added and the contents evaporated to dense white fumes. The residue was treated cautiously with about 50 ml of water, cooled, filtered and made up to 100 ml in a volumetric flask. The copper content in the ore solution was determined by the iodometric method. Different aliquots of the ore solution were analysed for their copper content with the procedure given above. The conventional procedure of precipitation employing sulphur dioxide is found to be more convenient since the other method involves the use of a complexing agent to mask the iron present. The present method showed the copper content to be 32·93% while the iodometric method showed it as 33·03%.

The method is more sensitive than the previous methods so far developed for the titrimetric determination of copper by precipitation as
cuprous thiocyanate. Moreover, the end point in this redox titration is more clearly discernable than in the precipitation titration employing silver nitrate reported earlier.8

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REFERENCES