

RAPID DETERMINATION OF COPPER IN COPPER ORES(*)

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Low grade copper ores and copper ores in association with considerable quantities of other elements such as iron, lead, zinc and vanadium require a separational step wherein copper is generally separated as sulphide either with sodium thiosulfate or with thioacetamide. In these methods the precipitated copper sulfide is ignited to cupric oxide, dissolved in dilute nitric acid and titrated idometrically. In a recent method wet oxidation of precipitated copper sulfide is effected with nitric acid and perchloric acid and titrated with disodium ethylene diamine tetraacetate (EDTA). The authors' work on the beneficiation of low grade copper ores and extraction of copper from these ores has necessitated developing a rapid and accurate method for the determination of copper. A separational step wherein cupric ion could be precipitated as cuprous oxide has been introduced to avoid ignition of copper sulfide to cupric oxide, which is time consuming.

This paper presents a method for the rapid and accurate determination of copper in copper ores and concentrates. Copper is separated as cuprous oxide with ascorbic acid in dilute acetic acid medium. The precipitated cuprous oxide is dissolved in dilute nitric acid and titrated idometrically or alternatively with EDTA using murexide or pyrocatechol violet as indicator. This method is quite rapid and accurate and yields results which are comparable with those obtained by electro-gravimetric method.

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EXPERIMENTAL

Determination of copper

A known volume (containing 25-100 mg.) of copper solution was transferred to a 250 ml beaker, rendered ammoniacal with 1:4 ammonia and acidified with 1:4 acetic acid. Ascorbic acid (1 g) was added and the solution was boiled. The precipitated cuprous oxide was filtered through filter pulp and washed with hot water. Cuprous oxide was dissolved in warm 1:1 nitric acid and washed with 2% nitric acid. The solution and the washings were collected in a conical flask. Urea (2 g) was added and the solution was boiled. After cooling the solution, ammonia was added to remove free mineral acid. The solution was acidified with acetic acid and cooled to room temperature. 1 g Potassium iodide was added. The liberated iodine was titrated with standard sodium thiosulfate solution using 1% starch solution as indicator.

Alternatively, the solution and the washings were diluted to 100 ml; 2 to 3 drops of aqueous solution of murexide followed by small volumes of ammonium chloride-ammonia buffer (pH 10) to give yellowish green color were added. This solution was titrated with 0.05 M EDTA to a violet-blue end point.

Alternatively, the solution and the washings were diluted to 100 ml. 0.5 g ammonium nitrate, 4-5 drops of 0.1 per cent aqueous solution of pyrocatechol violet indicator were added. The acidity of the solution was adjusted with 0.5 M ammonia until the blue color of the copper pyrocatechol violet complex just appears. 2 g sodium acetate was added and the solution was titrated against 0.05 M EDTA solution to a sharp yellowish-green end point.

Determination of copper in ores and concentrates

2 g copper ore or 0.5 g, concentrate was weighed into a 500 ml conical beaker. 20 ml of mixed concentrated hydrochloric, sulfuric and nitric acids (1:1:1) was added and gently heated until the digestion was complete. 5 ml of concentrated sulfuric acid was added and the heating was continued till sulfur trioxide fumes were evolved. The contents were cooled to room temperature. 100-150 ml water was added and the solution was gently heated till all the salts formed were dissolved.

The solution was filtered and the residue was washed with 5% sulfuric acid and subsequently with hot water. The filtrate and the washings were collected into a 400 ml beaker. Citric acid or tartaric acid (5 g) was added to complex Fe (III) and neutralised with ammonia. The solution was rendered slightly acidic with 1:4 acetic acid and heated gently. 2 g. ascorbic acid was added and the solution was then boiled. The cuprous oxide thus precipitated was filtered through filter pulp and washed with 2% acetic acid and hot water. The washed precipitate was dissolved in minimum quantity of warm 1:1 nitric acid and washing was effected with hot 2% nitric acid. The solution was allowed to cool and was made upto 250 ml in a standard volumetric flask. Aliquots of the copper solution were taken and the copper was determined idometrically. Alternatively, copper solution was titrated with 0.05 M EDTA using pyrocatechol violet or murexide as indicator.

RESULTS

The results for the determination of copper in pure copper solutions by the above mentioned procedure are reproducible and accurate to within about 0.5 per cent. Various ore samples from Khetri, Kalihan and Agnigundala mines and ore concentrate from Mysore ore were analysed by the proposed method and the results are comparable with those obtained by electro-gravimetric method.

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