

POLAROGRAPHIC ANALYSIS OF ZINC ORE CONCENTRATES FOR
THE DETERMINATION OF LEAD, COPPER, CADMIUM & ZINC. (*)

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The present work was taken up to find out a rapid and reliable polarographic method for the analysis of a large number of samples of zinc ore concentrates required to be analysed during the beneficiation of zinc ores in this laboratory. The difficulties encountered for the determination of lead, copper, cadmium and zinc by existing wet chemical methods are well-known. It is often required to separate the metals from each other and the interfering elements also, making the processes time-consuming.

Solubility of lead sulphate in ammonium acetate has been utilised in the present work for the determination of lead. The sample was dissolved in hydrochloric and nitric acid mixture and lead was separated by fuming with sulphuric acid. The precipitate was filtered off. Lead sulphate in the precipitate was extracted with ammonium acetate solution and lead was estimated polarographically. From the filtrate copper, cadmium and zinc were determined polarographically making the solution ammonical in presence of ammonium chloride.

Preparation of Standard solutions and calibration graphs:-

For the preparation of standard solutions spectrographically pure copper, cadmium, lead & zinc metals were separately dissolved in nitric acid. Mixing different aliquot portions of the above solutions five standard solutions were prepared.

One gram each of spectrographically pure copper, cadmium, lead and zinc were weighed (-0.5 mg) and dissolved separately in nitric acid and made upto 100 ml in measuring flasks. Five synthetic solutions containing 0.0005 to 0.003 g/ml of the above metals were prepared

(*)Paper for presentation at the Symposium on "Recent Developments in Non-Ferrous Metals' Technology"- 4th to 7th December, 1968, Jamshedpur.

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by mixing the individual metal solutions. From these solutions lead was precipitated as lead sulphate by fuming with sulphuric acid. After filtering and washing, the precipitates were dissolved and diluted to 100 ml with 5% ammonium acetate solution (w/v). The solutions were polarographed and the wave heights of lead were measured and plotted against concentration/ml of lead to obtain the calibration graph for lead.

To the filtrate obtained after separation of lead 10g solid ammonium chloride was added and the solutions were made ammonical. The volumes were made up to 100 ml in measuring flasks. A suitable portion of the liquid was taken from each flask and after removal of dissolved oxygen polarograms were recorded. The step heights for copper (second wave), cadmium and Zinc were measured and plotted against their respective concentrations in g/ml to obtain the standard calibration graphs.

GENERAL PROCEDURE:

2.5 g of the finely ground sample was taken in 250 ml beaker and covered with a watch glass. A mixture of 20 ml concentrated HCl and 10 ml concentrated HNO_3 was slowly added to the sample and digested on a hot plate until a clear solution was obtained. 5 ml conc. H_2SO_4 was added to the cooled solution, fumed to almost dryness, again cooled, diluted with 20 ml water and the process of fuming repeated. After cooling 40 ml water was added to the solution and boiled. The precipitate was filtered off and washed with dil H_2SO_4 (1:99). The filtrate and washings were collected in a 100 ml measuring flask and were reserved for determination of copper, cadmium and zinc.

0.5 gm. of solid ammonium acetate was added to the precipitate on the filter paper and then a hot solution of 5% ammonium acetate was added. The filtrate with the filter paper was collected in a 100 ml measuring flask and shaken well in order to have the precipitate of PbSO_4 completely dissolved. The solution was cooled by placing under tap water. Ten ml. of 0.1% freshly prepared gelatine solution was added to it and the volume was made up to the mark with 5% ammonium acetate solution. A portion of the supernatant liquid was taken in the Polarographic cell. Dry hydrogen was passed for ten minutes through the cell to remove dissolved oxygen. The polarogram was recorded from -0.3 volt to -0.7 volt Vs. S.C.E. and step heights for lead was measured at -0.6 V Vs. S.C.E.

To the filtrate after removal of Pb, 10 gms of solid ammonium chloride were added and the solution was made ammonical. Iron was precipitated. The volume of the solution was made up to the mark after adding 10 ml. of

0.1% gelatin solution. Taking an aliquot portion of the supernatant solution into the cell, hydrogen was passed to remove oxygen for ten minutes & polarogram was recorded from -0.2 volt to - 1.5 volts Vs. S.C.E.

The step heights were measured at -0.5, -0.8 and -1.3 V Vs. S.C.E. respectively for Cu, Cd Zn and compared with those of standard solutions. Second wave of copper was taken for determination of copper.

Discussion: The concentration of cadmium of zinc ore concentrate is very low and more sample may be required for its determination. In that case for the determination of the other constituents an aliquot portion of the solution may be diluted to a suitable volume maintaining the concentration of ammonium chloride identical to prevent precipitation of zinc by hydrolysis.

Solubility of $PbSO_4$ in ammonium acetate is retarded by the presence of barium, silica, tin and antimony which can be eliminated by using sufficient amount of the reagent even when $BaSO_4$ is 100 times more of lead. Free sulphuric acid when present to the extent of 10% will prevent the extraction of $PbSO_4$ with ammonium acetate completely. But in the procedure described there is little chance of getting this concentration of sulphuric acid after washing. To separate lead from silica, the precipitate may be hydrofluorised by the usual procedure.

Gelatin was found to have a marked effect on the wave of lead 0.01% gelatin was found sufficient to suppress the lead maxima. Greater concentrations of gelatin may decrease the step height of the wave considerably.

Concentration of ammonium acetate was also found to reduce the step height of the lead wave when it was present in excess. Final concentration of the test solution was controlled to avoid any detrimental effect on the lead wave.

For determination of lead in some other alloys, few experiments were carried out with "Clad Metal" by the same procedure. It was found to be suitable for its accurate determination with far less expenditure of time required by usual chemical method.
