Australian Standard®

Methods for the analysis of lead sulfide concentrates

Part 1: Determination of lead content—Acid dissolution solvent extraction EDTA titration method

This Australian Standard was prepared by Committee MN/5, Copper, Lead, Zinc, Gold and Silver Ores and Concentrates. It was approved on behalf of the Council of Standards Australia on 6 April 1992 and published on 13 July 1992.

The following interests are represented on Committee MN/5:

Australian Institute of Mining and Metallurgy

Australian Lead Development Association

Australian Mining Industry Council

CSIRO, Division of Mineral and Processing Engineering

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PREFACE

This Standard was prepared by Standards Australia's Committee on Copper, Lead, Zinc, Gold and Silver Ores and Concentrates as part of its program of standardizing methods for determination of elements of commercial interest in such materials.

This method was developed in conjunction with the International Standards Organization's Technical Committee (ISO/TC 183) for Copper, Lead and Zinc Ores and Concentrates and is technically identical to a draft International Standard which is in course of preparation. Throughout the formulation of this method and the draft International Standard Australia held the convenership of the ISO/TC 183 Working Group for the determination of lead content in lead concentrates. The precision data referred to in this method resulted from Australia's participation in two international test programs.

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1 SCOPE This Standard sets out a solvent extraction EDTA titrimetric method for the determination of lead content in lead sulfide concentrates.

The method is applicable to all lead sulfide concentrates with lead content in the range from 10 percent to 80 percent.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS
2134 Recommended practice for chemical analysis by atomic absorption spectrometry
2134.1 Part 1: Flame atomic absorption spectrometry

2162 Code of practice for the use of volumetric glassware

2164 One-mark volumetric flasks

2165 Burettes and bulb burettes

2816 Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method

2850 Chemical analysis — Interlaboratory test programs — For determining precision of analytical method(s) — Guide to the planning and conduct

3 PRINCIPLE The test portion is dissolved in nitric and hydrochloric acids.

Tartaric acid solution, ammonium hydroxide solution and sodium cyanide solution are added. The lead is extracted with sodium diethyldithiocarbamate in chloroform.

Nitric acid is added and the chloroform evaporated followed by the oxidation of residual organic matter.

Tartaric acid, ammonium chloride/ammonium hydroxide buffer solution, sodium cyanide solution, triethanolamine and ascorbic acid are added. The solution is titrated with EDTA using methyl thymol blue indicator.

4 REAGENTS

4.1 General requirements Unless otherwise specified, all reagents shall be of a recognized analytical grade and distilled water or deionized water shall be used throughout.

NOTE: A number of reagents used in this method (sodium cyanide), are toxic or dangerous. Advice on procedures for the handling and disposal of these reagents is contained in relevant Standards.

4.2 Lead metal, containing more than 99.99 percent lead The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in nitric acid (ϱ_{20} 1420 kg/m^3, diluted 1 + 9) for 1 min, washed well with water followed by acetone and dried in an oven at 50°C.

4.3 Ascorbic acid

4.4 Nitric acid (ϱ_{20} 1420 kg/m^3)

4.5 Hydrochloric acid (ϱ_{20} 1160 kg/m^3 to 1190 kg/m^3)

4.6 Hydrogen peroxide (100 vol.)

4.7 Tartaric acid solution (200 g/L). Dissolve 200 g of tartaric acid in 800 mL of water. Dilute to 1000 mL and mix.

4.8 Sodium cyanide solution (150 g/L). Dissolve three pellets of sodium hydroxide in 800 mL of water, add 150 g of sodium cyanide and mix to dissolve. Dilute to 1000 mL and mix.

4.9 Sodium diethyldithiocarbamate solution (100 g/L). Dissolve 10 g of sodium diethyldithiocarbamate in 80 mL of water containing five drops of ammonium hydroxide solution (4.12). Filter. Dilute to 100 mL and mix. Prepare fresh daily.

4.10 Chloroform

4.11 Triethanolamine (500 mL/L). Mix 50 mL of triethanolamine with 50 mL of water.

4.12 Ammonium hydroxide (ϱ_{20} 880 kg/m^3)