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Lead sulfide concentrates — Determination of lead content — Back titration of EDTA after precipitation of lead sulfate

*Concentrés sulfurés de plomb — Dosage du plomb — Titration en retour
de l'EDTA après précipitation du sulfate de plomb*



Reference number
ISO 11441:1995(E)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11441 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

Lead sulfide concentrates — Determination of lead content — Back titration of EDTA after precipitation of lead sulfate

1 Scope

This International Standard specifies a lead sulfate precipitation 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 % (m/m) to 80 % (m/m).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*.

ISO 9599:1991, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*.

ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*.

3 Principle

The stages which comprise the determination are described in 3.1 to 3.4 inclusive.

3.1 Dissolution

Dissolution by fusion with sodium peroxide.

3.2 Precipitation

Double precipitation of lead as a sulfate, with removal of bismuth if applicable.

3.3 EDTA dissolution

Dissolution of lead sulfate in a known volume of an alkaline EDTA solution.

3.4 Titration

Titration of EDTA in excess by a titrated zinc solution.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

4.1 Lead metal, minimum 99,99 % purity.

The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in nitric acid solution (4.10) diluted 1 + 9 for 1 min, washed well with water followed by acetone and dried in an oven at 50 °C.

4.2 Zinc metal, minimum 99,99 % purity.

The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in nitric acid (4.10) diluted 1 + 9 for 1 min, washed well with water followed by acetone and dried in an oven at 50 °C.

4.3 Sodium peroxide.**4.4 Sodium fluoride.****4.5 Perchloric acid**, (ρ_{20} 1,70 g/ml).**4.6 Hydrogen peroxide**, at 100 volumes.**4.7 Ethanol**, at 95 % (V/V).**4.8 Hydrofluoric acid**, concentrated (ρ_{20} 1,15 g/ml)

WARNING — Even when diluted hydrofluoric acid is extremely dangerous and harmful to the eyes and skin; rubber gloves and goggles should be worn when using this acid. Hydrofluoric acid attacks glassware. Care should be taken to minimize the time of acid contact with glassware. Use only in a mechanically ventilated fume cupboard.

4.9 Sulfuric acid, concentrated (ρ_{20} 1,83 g/ml).**4.10 Nitric acid**, concentrated (ρ_{20} 1,42 g/ml).**4.11 Hydrochloric acid**, concentrated (ρ_{20} 1,16 g/ml to 1,19 g/ml).**4.12 Acid washing mixture**

Mix 50 ml of perchloric acid (4.5) with 100 ml of hot water and add 2 ml of hydrogen peroxide (4.6). (To be freshly prepared just before use.)

4.13 Sulfuric acid, diluted 1 + 1

Slowly add 500 ml of concentrated sulfuric acid (4.9) to 500 ml of water. Mix thoroughly.

4.14 Washing solution

Mix 200 ml of water with 100 ml of ethanol (4.7) and 50 ml of sulfuric acid (4.13). Mix thoroughly.

4.15 Potassium hydroxide solution

Dissolve 200 g of potassium hydroxide in water. Dilute to 1 litre and mix. Store in plastic bottles.

4.16 Nitric acid, diluted 1 + 1

Slowly add 500 ml of concentrated nitric acid (4.10) to 500 ml of water. Mix thoroughly.

4.17 Zirconium nitrate solution

Add 3,53 g of zirconium oxychloride ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$) to 20 ml of nitric acid (4.10). Boil for approximately 10 min. Cool to room temperature. Dilute to 1 litre with water and mix.

4.18 Basic washing solution

Dilute 50 ml of the potassium hydroxide solution (4.15) to 1 litre with water.

4.19 Ammonium acetate solution

Dissolve 500 g of ammonium acetate in water. Dilute to 1 litre and mix.

4.20 Aqueous hexamethylene tetramine solution, saturated at room temperature.**4.21 Solution of disodium salt of ethylenediaminetetraacetic acid**, $c(\text{EDTA}) = 0,1 \text{ mol/l}$. Dissolve 37,22 g of disodium versenate in water, dilute to 1 litre and mix.

NOTE 1 The procedure for standardizing the EDTA solution is given in 4.22.2.

4.22 Standard solutions

NOTE 2 Standard solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.22.1 Zinc standard solution, $c(\text{Zn}^{2+}) = 0,1 \text{ mol/l}$ **4.22.1.1 Preparation**

Place 6,537 g of pure, freshly prepared, unoxidized zinc chips (4.2) in a covered beaker. Add, by small portions, a mixture of 25 ml of water and 20 ml of nitric acid (4.10). Heat gently. After dissolution is completed, boil to expel nitrous fumes. Allow to cool. Dilute to about 700 ml with water. Bring to pH 5,5 (using a pH-meter) by adding the hexamethylene tetramine solution (4.20). Transfer to a 1 000 ml volumetric flask, fill up with water nearly to the mark, mix and cool to room temperature, then fill up exactly to the mark and mix again.

NOTE 3 This solution is stable for several months.

4.22.1.2 Standardization

Place 25 ml of (0,1 mol/l) EDTA solution (4.21) in a 600-ml beaker. Add 25 ml of potassium hydroxide (4.15).

Dilute to 350 ml with water. Add 0,3 ml of xylenol orange indicator (4.23) and 5 ml of ammonium acetate solution (4.19). Carry out the procedure as specified in 7.5.

Determine the calibration factor t between the EDTA and zinc in triplicate and calculate the factors t_1 , t_2 and t_3 using the following equations

$$t_1 = \frac{V_{E_1}}{V_{Z_1}} \quad \dots (1)$$

$$t_2 = \frac{V_{E_2}}{V_{Z_2}} \quad \dots (2)$$

$$t_3 = \frac{V_{E_3}}{V_{Z_3}} \quad \dots (3)$$

where

V_E is the volume of (0,1 mol/l) EDTA solution, in millilitres, poured into the beaker (25 ml);

V_Z is the volume of zinc standard solution, in millilitres, used for titration.

Calculate, to four significant figures, the mean factor, t . If the range of the values of t_1 , t_2 , t_3 exceeds 0,001 repeat the standardization.

4.22.2 EDTA Standard solution.

NOTE 4 Standardization blank. A blank titration should be carried out in parallel with the standardization using all the reagents specified in the standardization but omitting the lead metal.

Weigh, to the nearest 0,1 mg, three portions of 0,1 g to 0,8 g of lead metal (4.1) and place in separate nickel crucibles (5.3). Record the masses as m_1 , m_2 and m_3 .

NOTE 5 The mass of lead metal should match the expected mass of lead metal in the test portion.

Carry out the procedure as specified in 7.3, 7.4 and 7.5.

Calculate the calibration factors f_1 , f_2 and f_3 between the EDTA solution and lead metal for each titration using the following equations:

$$f_1 = m_1/t(V_{B_1} - V_{Z_1}) \quad \dots (4)$$

$$f_2 = m_2/t(V_{B_2} - V_{Z_2}) \quad \dots (5)$$

$$f_3 = m_3/t(V_{B_3} - V_{Z_3}) \quad \dots (6)$$

where

m_1 , m_2 , m_3 are the masses, in grams, of each test portion of lead;

t is the mean factor of correspondence between EDTA and zinc solution determined in 4.22.1.2;

V_B is the volume of titrated zinc solution, in millilitres, used to titrate excess EDTA solution for the standardization blank;

V_Z is the volume of titrated zinc solution, in millilitres, used to titrate excess EDTA for lead metal.

Calculate to four significant figures, the mean factor, f , for the EDTA. If the range of values of f_1 , f_2 , f_3 exceeds 0,000 02 g/ml repeat the standardization.

4.23 Xylenol orange indicator.

Dissolve 0,5 g of xylenol orange in 100 ml of water.

5 Apparatus

5.1 Ordinary laboratory equipment.

5.2 Volumetric glassware, of class A complying with ISO 385-1, ISO 648 and ISO 1042 and used in accordance with ISO 4787.

5.3 Nickel crucibles, of capacity 30 ml to 35 ml with lids.

5.4 Zirconium crucibles, of capacity 30 ml to 35 ml with lids.

5.5 Membrane filter — cellulose nitrate, diameter 30 mm to 50 mm, mesh size 0,45 μm to 0,65 μm .

5.6 Membrane filter, diameter 30 mm to 50 mm, mesh size 0,45 μm . Membrane to be insoluble in sodium hydroxide EDTA solution.

NOTE 6 PVC is considered suitable.

5.7 A vacuum filtration apparatus, consisting of a 30 mm to 50 mm diameter funnel, and a one-litre evacuated Buchner flask.