



Standard Test Method for Determination of Zinc in Zinc Ores and Concentrates by EDTA Complexometric Titrimetry¹

This standard is issued under the fixed designation E945; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of zinc in ores, concentrates, and related materials having chemical composition within the following limits:

Element	Application Range, %
Zinc	5.0 to 70.0
Lead	0.5 to 50.0
Copper	0.1 to 3.0
Iron	0.5 to 16.0
Sulfur	4.0 to 30.0
Calcium	0.1 to 20.0
Magnesium	0.1 to 10.0
Cadmium	0.1 to 8.0
Arsenic	0.01 to 1.0
Antimony	0.01 to 0.005
Bismuth	0.001 to 0.1
Cobalt	0.1 to 0.5
Nickel	0.3 to 3.0
Silver	0.00 to 150 oz/ton
Gold	0.00 to 1.0 oz/ton

NOTE 1—As used in this test method, *percent* or “%” refers to a mass fraction.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Consid-

¹ This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

erations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)³

E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)³

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 The sample, after appropriate acid decomposition, is evaporated to near dryness. The salts are dissolved in acid, interfering elements are removed, and the zinc is extracted as thiocyanate complex into MIBK. Zinc is determined in the extract by titrating with EDTA, using an internal indicator.

5. Significance and Use

5.1 This test method is primarily intended to test materials for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts working in properly equipped laboratories.

5.2 Appropriate quality control practices shall be followed such as those described in Guide E882.

6. Interferences

6.1 With the exception of cadmium and cobalt, elements do not interfere if their compositional ranges are under the maximum limits shown in 1.1.

³ The last approved version of this historical standard is referenced on www.astm.org.

6.2 When the cadmium content is above 5.0 % in the material to be analyzed, some of the cadmium is extracted and will titrate as zinc. The addition of potassium iodide before the titration serves to prevent the interference of cadmium. The amount of potassium iodide solution to add in order to prevent the interference of cadmium is listed in 13.10.3.

6.3 Cobalt is extracted and titrated with the zinc. If the cobalt content of the material to be analyzed is less than 0.05 %, the interference is negligible. For cobalt contents greater than 0.05 %, the cobalt must be extracted as outlined in 13.9.5.

7. Apparatus

7.1 *Magnetic Stirrer*, with TFE-fluorocarbon covered magnetic stirring bar. A magnetic stirrer provided with illumination is preferred.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample

8.3 *Ammonium Fluoride Solution* (250 g/L)—Dissolve 250 g of ammonium fluoride (NH₄F) in water and dilute to 1 L. Store in a polyethylene bottle.

8.4 *Ammonium Chloride Solution* (250 g/L)—Dissolve 250 g of ammonium chloride (NH₄Cl) in water and dilute to 1 L.

8.5 *Ammonium Thiocyanate* (500 g/L)—Dissolve 500 g of ammonium thiocyanate (NH₄SCN) in distilled water and dilute to 1 L.

8.6 *Buffer Solution* (pH 5.5)—Dissolve 250 g of hexamethylenetetramine (C₆H₁₂N₄) in 750 mL of water. Add 57 mL of acetic acid, dilute to 1 L, and mix.

8.7 *Chloroform* (CHCl₃).

8.8 *Disodium Ethylenedinitrilo Tetraacetate Dihydrate (EDTA) Standard Solution*—Prepare a solution as follows:

8.8.1 *Preparation*—Dissolve disodium ethylenedinitrilo tetraacetate (EDTA) dihydrate in water, transfer to a 1-L volumetric flask, dilute to volume, and mix. The solution is stable for several months when stored in plastic or borosilicate glass bottles.

Use the following table as a guide for the specific weight of EDTA · 2H₂O to use.

NOTE 2—The use of varying concentrations of EDTA solution allows the volume of the titrant to be between 30 mL and 50 mL.

If the expected zinc is:	EDTA · 2H ₂ O Mass, g/L	EDTA Concentration	Amount of Standard Zinc Solution to Use
5 % to 20 %	6.642	6 g/L	10 mL
20 % to 40 %	13.29	12 g/L	30 mL
40 % to 56 %	17.71	16 g/L	40 mL
56 % to 70 %	22.14	20 g/L	50 mL

8.8.2 Standardize the EDTA solution by pipetting the suggested amount of standard zinc solution into a 250-mL separatory funnel. Add 10 drops of ferric chloride solution, add distilled water to adjust volume to 50 mL, mix, and proceed as directed in 13.9.2. It is recommended that replicate standardizations be made to ensure better accuracy.

8.8.3 Calculate the zinc equivalent of the EDTA solution as follows:

$$\text{Zinc Equivalent (mg/mL)} = \frac{A}{B} \quad (1)$$

where:

A = zinc in the chosen aliquot, mg and

B = EDTA solution (8.8) used, mL

8.9 *Ethanol* (CH₃CH₂OH)—Certain denatured ethanols interfere with the color of the indicator during the titration. Pure ethanol is recommended for this reason.

8.10 *Ferric Chloride Solution* (140 g/L)—Dissolve 14 g of ferric chloride (FeCl₃·6H₂O) in water and dilute to 100 mL.

8.11 *4-Methyl-2-Pentanone* (MIBK) —CH₃COCH₂CH (CH₃)₂.

8.12 *2-Nitroso-1-Naphthol Solution* (10 g/L)—Dissolve 0.5 g of 2-nitroso-1-naphthol (NOC₁₀H₆OH) in 50 mL of acetic acid. Prepare fresh as needed.

8.13 *Potassium Iodide* (1000 g/L)—Dissolve 100 g of potassium iodide (KI) in distilled water and dilute to 100 mL.

8.14 *Sodium Fluoride Solution* (20 g/L)—Dissolve 10 g of sodium fluoride (NaF) in water and dilute to 500 mL. Store in a polyethylene bottle.

8.15 *Thiourea Solution* (100 g/L)—Dissolve 50 g of thiourea (NH₂CSNH₂) in water and dilute to 500 mL.

8.16 *Xylenol Orange Tetrasodium Salt Indicator Solution* (2 g/L)—Dissolve 100 mg of xylenol orange tetrasodium salt in water and dilute to 50 mL.

8.17 *Zinc, Standard Solution* (1 mL–3.50 mg)—Dissolve 3.50 g of zinc (minimum purity 99.99 %) in 10 mL of HNO₃ and 25 mL of water. Heat gently; when dissolution is complete, boil. Cool. Transfer to a 1-L flask. Dilute to the mark and mix thoroughly.

9. Hazards

9.1 For precautions to be observed in the use of certain reagents in this test method, refer to Practices E50.

10. Sampling and Sample Preparation

10.1 The gross sample shall be collected and prepared so as to be representative of the material to be analyzed.

⁴ *Reagent Chemicals, American Chemical Society Specifications, American Chemical Society*, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.