



Designation: E 945 – 02

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

This standard is issued under the fixed designation E 945; 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	Concentration 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

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:

- D 1193 Specification for Reagent Water²
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores and Related Materials⁴
- E 173 Practice for Conducting Interlaboratory Studies of

Methods for Chemical Analysis of Metals⁵

E 663 Practice for Flame Atomic Absorption Analysis⁶

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁶

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry⁶

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method⁴

3. Terminology

3.1 **Definitions**—For definitions of terms used in this test method, refer to Terminology E 135.

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 E 882.

6. Interferences

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

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

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, Refractories, Cement, and Related Metallurgical Materials.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ Discontinued; see 1997 *Annual Book of ASTM Standards*, Vol 03.05.

⁶ *Annual Book of ASTM Standards*, Vol 03.06.



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 Atomic absorption spectrophotometers shall conform to Practice E 663 or Guide E 1024.

7.2 *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.⁷ 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 of Specification D 1193.

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 3.7225 g of disodium (ethylenedinitrilo) tetraacetate 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 salt to use.

NOTE 1—The use of varying concentrations of EDTA solution allows the volume of the titrant to be between 30 and 50 mL.

If the expected zinc is:	EDTA Concentration	Amount of Standard Zinc Solution to Use
5 to 20 %	6 g/L	10 mL
20 to 40 %	12 g/L	30 mL
40 to 56 %	16 g/L	40 mL
56 to 70 %	20 g/L	50 mL

⁷ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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 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 (NO-C₁₀H₆OH) 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 E 50.

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.

10.2 Pulverize the laboratory sample to pass a No. 100 (150-μm) sieve. The repeatability, R₁, of Practice E 173 corresponds to the repeatability index, r, of Practice E 1601.

11. Rounding Calculated Values

11.1 Calculated values shall be rounded to the desired number of places as directed in the Rounding-Off Procedure section of Practice E 29.

12. Interlaboratory Studies

12.1 This test method has been evaluated in accordance with Practice E 173. For those methods tested in accordance