



Designation: E738 – 20

Standard Test Method for Determination of Aluminum in Iron Ores and Related Materials by EDTA Complexometric Titrimetry¹

This standard is issued under the fixed designation E738; 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 method² covers the determination of aluminum in iron ores, concentrates, and agglomerates in the compositional range from 0.25 % to 5 % aluminum.

NOTE 1—As used in this test method (except as related to the term *relative standard deviation*), “percent” or “%” refers to mass fraction (mass/mass) of the form g/100 g.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*³

[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 Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

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 Bhargava, O. P., “Complexometric Determination of Aluminum in Iron Ore, Sinter, Concentrates, and Agglomerates,” *Talanta*, Vol 26, 1979, pp. 146 to 148.

³ 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.

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties](#)

[E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory](#)

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 is fused in a zirconium crucible with a mixed flux of sodium carbonate and sodium peroxide. The fused mass is dissolved in dilute HCl. The R_2O_3 ([Note 2](#)) hydroxides are precipitated with NH_4OH and redissolved in HCl. Iron, titanium, and so forth, are removed with cupferron and chloroform. The aqueous phase is treated with HNO_3 and $HClO_4$ and evaporated to dryness. After dissolving in dilute HCl, the solution is filtered, and the filtrate is treated with an excess of ethylenediaminetetraacetic acid (EDTA). The excess EDTA is titrated with a standard zinc solution using xylenol orange indicator. Ammonium fluoride is added to release the EDTA bound to aluminum. This EDTA is then titrated with standard zinc solution, and the percent aluminum is calculated.

NOTE 2— R_2O_3 hydroxides are rare earth sesquioxides such as La_2O_3 , Y_2O_3 , Ce_2O_3 , Gd_2O_3 , and Eu_2O_3 .

5. Significance and Use

5.1 This test method for the analysis of iron ore concentrates and agglomerates is primarily intended as a referee method to test for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices shall be followed, such as those described in [Guide E882](#).

6. Interferences

6.1 None of the elements normally found in iron ores interfere with this test method.

7. Apparatus

7.1 *Zirconium Crucible*, 50-mL.

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 conforming to 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 Chloride Solution* (10 g/L)—Dissolve 10 g of ammonium chloride (NH_4Cl) in water and dilute to 1 L. Add 2 drops (0.1 mL) of NH_4OH per 100 mL of solution.

8.4 *Ammonium Fluoride Solution*—Dissolve 10 g of ammonium fluoride (NH_4F) in 100 mL of water in a polyethylene beaker. Prepare fresh each day.

8.5 *Chloroform* (CHCl_3).

8.6 *Cupferron Solution* (60 g/L)—Dissolve 6 g of cupferron in 80 mL of cold water, dilute to 100 mL, and filter. Prepare fresh as needed.

8.7 *Ethylenediaminetetraacetic Acid (EDTA) Solution* (0.01 mol/L).

8.8 *Sodium Acetate Buffer Solution*—Dissolve 136 g of sodium acetate trihydrate ($\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$) in about 600 mL of water. Add 7 mL of glacial acetic acid and dilute to 1 L. Store in a polyethylene bottle.

8.9 *Sodium Carbonate* (Na_2CO_3).

8.10 *NaOH Solution A*—Dissolve 100 g of NaOH in about 100 mL of water. When dissolution is complete, cool, and dilute to 1 L. Store in a plastic bottle.

8.11 *NaOH Solution B*—Dilute 100 mL of NaOH solution A to 1 L with water.

8.12 *Sodium Peroxide* (H_2O_2).

8.13 *Xylenol Orange Indicator*—Thoroughly grind 0.1 g xylenol orange with a little water to make a fine paste. Dilute to 100 mL with water, filter, and store in an amber-colored glass stoppered bottle. The solution is stable for 1 week.

8.14 *Zinc, Standard Solution* (0.01 M)—Weigh 0.6538 g of high purity (99.99 %) zinc metal into a 125-mL Erlenmeyer flask. Add 5 mL of water followed by 2 mL to 3 mL of HNO_3 . Cover the flask and simmer. If the reaction subsides leaving

undissolved metal, add 2-mL increments of water followed by simmering until complete solution is obtained. Add 50 mL of water and gently boil for 2 min. Cool. By dropwise addition of NaOH solution B, adjust the pH to 4. Dilute to 1 L with water in a volumetric flask.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practices **E50**.

10. Sampling and Sample Preparation

10.1 Sample the material in accordance with Practice **E877**.

10.2 Pulverize the laboratory sample to pass a 150- μm (No. 100).

NOTE 3—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a 75- μm (No. 200) sieve.

11. Procedure

11.1 Select and weigh a test sample as follows:

Aluminum, %	Test Sample Mass, g	Tolerance in Test Sample mass, mg
0.25 to 1	0.3	5
1 to 2	0.2	5
2 to 5	0.1	5

Transfer the prepared sample to a small weighing bottle previously dried at about 105 °C. Dry the bottle and contents for 1 h at 105 °C to 110 °C. Cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize the pressure and weigh the capped bottle and sample to the nearest 0.1 mg. Repeat the drying and weighing until there is no further loss of mass. Transfer the test sample to a dry, zirconium crucible containing 0.5 g of Na_2CO_3 and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two masses is the mass of the test sample taken for analysis. Add 2 g of Na_2O_2 to the crucible and mix with a dry, stainless steel spatula.

11.2 Fuse over a burner, swirling the crucible until the melt is cherry red and clear. Remove from the heat and swirl until the melt solidifies on the wall of the crucible.

11.3 Place the crucible in a 250-mL dry beaker and cool. Cover with a watchglass and add about 10 mL of water to the crucible. After the reaction ceases, transfer the crucible contents into the beaker and wash the crucible with water. Add 20 mL of HCl (1 + 1) by way of the crucible into the beaker. Rinse the crucible with water and add rinsings to the beaker. Dilute to about 70 mL with water and heat to boiling.

11.4 Remove from heat and add NH_4OH (1 + 1) dropwise until the precipitation of the hydroxides is complete (pH 6 to pH 7). Boil for 1 min and immediately filter through a medium-textured paper. Wash five times with hot ammonium chloride solution (10 g/L). Then wash five times with hot water. Discard the filtrate. Place the unfolded filter paper on the inner wall of the original beaker and remove the precipitate from the paper with a fine jet of hot water. Then wash the filter paper with 25 mL of hot HCl (1 + 1) and finally with a jet of hot water. Discard the filter paper, cover the beaker with a

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD, <http://www.uspc.org>.