Designation: E738 – 11

Standard Test Method for Determination of Aluminum in Iron Ores and Related Materials by EDTA Complexometric Titrimetry[1]

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 method[2] 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 (wt/wt) of the form g/100g.

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

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

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 ammonia and redissolved in HCL. Iron, titanium, and so forth, are removed with cupferron and chloroform. The aqueous phase is treated with H_2NO_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, $Ce_2 O_3$, 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.

---

[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.

Current edition approved Sept. 1, 2011. Published September 2011. Originally approved in 1980. Last previous edition approved in 2005 as E738 – 00(2005). DOI: 10.1520/E0738-11.

[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.

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 in water and dilute to 1 L. Add 2 drops (0.1 mL) of ammonium hydroxide 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 *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.

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

8.7 *Sodium Acetate Buffer Solution*—Dissolve 136 g of sodium acetate trihydrate ($CH_3\ COONa\cdot 3H_2O$) in about 600 mL of water. Add 7 mL of glacial acetic acid and dilute to 1 L. Store in a polyethylene bottle.

8.8 *Sodium Carbonate.*

8.9 *Sodium Hydroxide Solution A.*

8.9.1 Dissolve 100 g of sodium hydroxide (NaOH) in about 100 mL of water. When dissolution is complete, cool, and dilute to 1 L. Store in a plastic bottle.

8.10 *Sodium Hydroxide Solution B*—Dilute 100 mL of sodium hydroxide solution A to 1 L with water.

8.11 *Sodium Peroxide.*

8.12 *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.

8.13 *Zinc, Standard Solution* (0.01 *M*)—Weigh 0.6538 g of high purity (99.99 % zinc) zinc metal into a 125-mL Erlenmeyer flask. Add 5 mL of water followed by 2 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 sodium hydroxide 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 No. 100 (150-µm sieve).

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

11. Procedure

11.1 Select and weigh a test sample in accordance with the following:

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 sodium carbonate 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 sodium peroxide 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 watchglass, and bring the contents to a boil. Cool and adjust the volume to 50 mL using water. Cool to about 10 °C.

---

[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 *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.

11.5 Transfer the test solution to a cold 250-mL separatory funnel. Rinse the beaker with 25 mL of cold water and add to the separatory funnel. Add 25 mL of cold cupferron solution and mix. Add 30 mL of chloroform and shake vigorously for 1 min. Let the layers separate and draw off the organic phase and discard. Wash the aqueous layer with two successive additions of 20 mL of chloroform, again discarding the organic phase. Wipe the stem of the separatory funnel with a filter paper wick.

11.5.1 Drain the aqueous phase into a 250-mL beaker, rinsing with 5 mL of HCl (1 + 5). Boil for a few minutes and remove from the heat. Add 5 mL of HNO_3 and 10 mL of $HClO_4$. Cover with a ribbed cover glass and evaporate nearly to dryness. Remove from the heat and add 10 mL of HCl (1 + 1). Heat to dissolve the salts, add 50 mL of water, and bring to a boil. Filter through a medium-textured filter paper and wash several times with hot water.

11.6 Cool and add 25.0 mL of EDTA solution. Using a pH meter, adjust the pH to 2.5 by the dropwise addition of NaOH solution A then to pH 4.0 by the dropwise addition of NaOH solution B. Dilute to 100 mL with water, cover the beaker, and boil gently for 10 min. Cool and add 15 mL of sodium acetate buffer solution and 7 drops of xylenol orange indicator solution. Stir using a magnetic stirrer and titrate with zinc standard solution to a persistent (30 s) pink end point.

11.7 Add 10 mL of ammonium fluoride solution and boil gently for 10 min. Cool, add 2 drops to 3 drops of xylenol orange indicator solution, and titrate the liberated EDTA with zinc standard solution to a persistent (30 s) pink end point.

12. Calculation

12.1 Calculate the aluminum content as follows:

$$\text{Aluminum (dry basis), \%} = \frac{0.02698A}{B} \quad (1)$$

where:

A = millilitres of zinc standard solution used in 11.7, and
B = grams of sample used.

TABLE 1 Statistical Information

Iron Ore	Average Aluminum Content, %	Relative Standard Deviation, %	Number of Participating Laboratories	Number of Determinations
Marcona	0.362	3.04	23	67
Krivoj	0.661	2.72	23	67
BCS 377	1.76	1.82	23	67
Monette	2.34	1.79	23	66
BCS 303	3.51	1.25	23	69
Phillipine Iron Sand	1.44	1.25	23	68

13. Precision and Bias[5]

13.1 *Precision*—Table 1 indicates the precision of the test method between laboratories.

Bias—No information on the bias of this test method is known. Accepted reference materials may have not been included in the materials used in the interlaboratory study. Users of this test method are encouraged to employ accepted reference materials, if available, and to judge the bias of the method from the difference between the accepted value for the aluminum content and the mean value from interlaboratory testing of the reference material.

14. Keywords

14.1 agglomerates and related materials; aluminum content; concentrates; iron ores

---

[5] Supporting data giving the results of cooperative testing are available from ASTM International Headquarters. Request RR:E16-1002.