Designation: E507 – 13

Standard Test Method for
Determination of Aluminum in Iron Ores by Flame Atomic Absorption Spectrometry[1]

This standard is issued under the fixed designation E507; 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 aluminum in iron ores, concentrates, and agglomerates in the mass fraction (%) range from 0.1 to 5.

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:*[2]

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
E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials
E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)[3]
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 dissolved in HCl with the addition of a small amount of HNO_3. After evaporation and dehydration, any insoluble residue is filtered, treated for the recovery of aluminum, and added to the main solution. The solution is aspirated into the flame of a nitrous oxide-acetylene burner. The absorbed energy at the resonance line of the spectrum of aluminum is measured by atomic absorption spectrometry and compared with that of the calibration solutions.

5. Significance and Use

5.1 This test method is intended as a referee method for compliance with compositional specifications for impurity content. It is assumed that all who use this procedure will be trained analysts capable of performing common laboratory practices 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. Follow appropriate quality control practices 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 *Atomic Absorption Spectrometer,* meeting the following criteria:

7.1.1 *Minimum Sensitivity*—The absorbance of the highest calibration solution (see 8.4) must be at least 0.3.

7.1.2 *Curve-Linearity*—The difference between the readings of the two highest calibration solutions must be more than 1.4 times the difference between the readings for the zero solution and the lowest calibration solution.

7.1.3 *Minimum Stability*—The coefficient of variation of a number of measurements of the highest calibration solution and of the zero calibration solution must be less than 1.5 % and 0.5 %, respectively.

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[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.

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

NOTE 1—Use of digital readout devices or printers, or both, is advisable.

NOTE 2—Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into a nitrous oxide-acetylene flame of a premix burner.

Hollow cathode lamp, mA	25
Wavelength, nm	396.15
Nitrous oxide flow rate, L/min	13.8
Acetylene flow rate, L/min	6.6

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 sufficiently 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 D1193.

8.3 *Aluminum, Standard Solution* (1 mL = 0.5 mg Al)—Dissolve 0.5000 g of high-purity aluminum in 25 mL of HCl, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

8.4 *Aluminum Calibration Solutions*—Transfer (5.0, 10.0, 20.0, 40.0, and 50.0)-mL portions of the standard aluminum solution to 200-mL volumetric flasks. Dilute to about 100 mL, add 6 mL of HCl and 60 mL of the background solution, dilute to volume, and mix. These calibration solutions contain (12.5, 25, 50, 100, and 125) µg/mL Al, respectively.

8.5 *Iron, Background Solution* (10 g/L)—Dissolve 10 g of high-purity iron in 50 mL of HCl and oxidize by the dropwise addition of HNO_3. Evaporate to a syrupy consistency. Add 20 mL of HCl and dilute to 200 mL with water. Add 20 g of NaCl dissolved in 200 mL water and dilute to 1 L with water.

8.6 *Zero Calibration Solution*—Transfer 60 mL of the background solution to a 200-mL volumetric flask, add 6 mL of HCl, dilute to volume, and mix.

9. Hazards

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

10. Sample Preparation

10.1 The analytical sample shall be pulverized to pass a 150-µm sieve (see Test Method E276).

NOTE 3—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a 75-µm sieve.

11. Procedure

11.1 Transfer approximately 1.0 g of the sample to a small weighing bottle previously dried at about 150 °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 sample to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two masses is the mass of the sample taken for analysis.

11.2 Carry a reagent blank through all steps of the procedure starting with 11.3.

11.3 *Decomposition of Sample*—Moisten the sample with a few millilitres of water, add 25 mL of HCl, cover and digest below the boiling point until no further attack is apparent. Add 2 mL of HNO_3 and digest for several minutes. Remove the cover and evaporate the solution to dryness. Heat the salts on a hot plate at 105 °C to 110 °C for 15 min. Add 5 mL of HCl, cover the beaker, and warm for several min. Add 50 mL of water and warm until soluble salts are in solution. Filter on a fine-textured paper, and collect the filtrate in a 250-mL beaker. Transfer the residue quantitatively to the filter paper. Wash alternatively with hot dilute HCl (1 + 9) and hot water until the yellow color of ferric chloride is no longer apparent and then with hot water six to eight times. Transfer the paper and residue to a platinum crucible. Reserve the filtrate.

11.4 *Treatment of Residue*—Ignite the paper and residue in the platinum crucible. Cool, moisten with several drops of water, and add three or four drops of H_2SO_4 (1 + 1) and 10 mL of HF. Evaporate slowly to expel silica and then fume off the excess of H_2SO_4. Ignite to about 700 °C. Add 1 g of anhydrous Na_2CO_3 to the residue, cover the crucible, and fuse over a burner or in a muffle furnace until a clear melt is obtained. Dissolve the cool melt in the reserved filtrate from 11.3, remove and wash the crucible and cover. Transfer the solution to a 200-mL volumetric flask, dilute to volume, and mix. If the solution is cloudy at this stage, indicating the presence of titania, filter a portion of the solution through a dry paper into a dry beaker. Use this solution for the atomic absorption measurements, if the aluminum content is between 0.1 % and 2.5 %. For aluminum contents greater than 2.5 %, transfer a 40-mL aliquot to a 200-mL volumetric flask and add 50 mL of background solution and 4 mL of HCl. Dilute to volume and mix.

11.5 *Adjustment of Atomic Absorption Spectrometer*—In accordance with Practice E663, set the initial instrument parameters to the values in Note 2. Light the burner and aspirate water until the instrument comes to thermal equilibrium. Optimize instrument response by adjusting the wavelength, fuel, air, burner, and nebulizer while aspirating the highest calibration solution to obtain maximum absorption or absorbance. Aspirate water until a steady signal is obtained and adjust the instrument readout system to obtain zero absorption or absorbance.

NOTE 4—The manufacturer's instructions for igniting and extinguishing

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[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.

TABLE 1 Precision of Method Within and Between Laboratories

Average Mass Fraction (%)[A] of Al	Relative Standard Deviation[B] Within Laboratories, %	Relative Standard Deviation[B] Between Laboratories, %	Number of Laboratories	Number of Determinations
0.178	4.49	7.87	30	88
0.261	4.21	18.77	31	91
0.426	2.58	5.40	11	31
0.667	1.35	2.40	7	19
0.884	1.81	6.11	24	72
1.46	2.53	2.47	29	85
2.14	2.01	5.14	31	91

[A] Each mass fraction (%) of aluminum represents a different kind of iron ore.

[B] Relative standard deviation (RSD), in this test method is calculated as follows:

$$RSD = \left(100/\bar{X}\right)\sqrt{\left(\sum d^2\right)/(n-1)}$$

where:

$\bar{X}$ = average mass fraction (%),
d = difference of the determination from the mean, and
n = number of determinations.

the nitrous oxide-acetylene burner should be strictly followed to avoid possible explosion hazards. Tinted safety glasses should be worn by the operator whenever the flame is burning.

11.6 *Measurements*—Aspirate water until the initial reading is again obtained. Aspirate the calibration solutions in the order of increasing absorption or absorbance starting with the zero calibration solution. When a stable response is obtained for each solution, record the readings. Aspirate the test solutions at the proper points in the calibration series. Aspirate water between each calibration and test solution. Repeat all measurements at least two more times.

12. Calculation

12.1 If necessary, convert the average of the readings for each calibration solution to absorbance. Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the zero calibration solution. In a similar manner, obtain the net absorbance of the test solution by subtracting the absorbance of the reagent blank solution.

12.2 Prepare a calibration curve by plotting the net absorbance values of the calibration solutions against micrograms of aluminum per milliliter.

12.3 Convert the net absorbance values of the sample solution to micrograms of aluminum per milliliter, by means of the calibration curve.

12.4 Calculate the mass fraction (%) of aluminum as follows:

$$\text{Aluminum, \%} = (A \times 200)/(B \times 10\ 000) \quad (1)$$

where:

A = aluminum, µg/mL, and
B = sample represented in the 200-mL volume of the final test solution, g.

13. Precision and Bias

13.1 *Precision*—Table 1 indicates the precision of the method within and between laboratories when applied to a number of samples of iron ores.

13.2 *Bias*—No information on the accuracy of this test method is known. The accuracy of this test method may be judged by comparison of accepted values for certified reference materials, with the mean determined through interlaboratory testing.

14. Keywords

14.1 aluminum; atomic absorption spectrometry; iron ores