



Designation: E508 – 21

Standard Test Method for Determination of Calcium and Magnesium in Iron Ores by Flame Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E508; 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 calcium and magnesium in iron ores, concentrates, and agglomerates in the mass fraction (%) range from 0.05 % to 5 % of calcium and 0.05 % to 3 % of magnesium.

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:*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 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

4.75 mm (No. 4) 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E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)³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 HNO₃. After evaporation and dehydration, any insoluble residue is filtered, treated for the recovery of calcium and magnesium, and added to the main solution. The solution is aspirated into the flame of the atomic absorption spectrometer. For calcium, a nitrous oxide-acetylene burner is used, for magnesium, an air-acetylene burner is preferred (Note 1). The absorbed energy at the resonance line of the spectrum of calcium or magnesium, or both, is measured by atomic absorption spectrometry and compared with that of the calibration solutions of the two elements.

NOTE 1—If a nitrous oxide burner is used for the determination of magnesium, the sensitivity is decreased by a factor of about three.

If an air-acetylene burner is used for the determination of calcium, several elements, if present in a mass fraction (%) of 1.0 or greater, interfere with the calcium determination. Also, lanthanum chloride (LaCl₃) must be employed and the sensitivity is decreased by a factor of about two.

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.

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

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.

6. Interferences

6.1 None of the elements normally found in iron ores interfere with this test method (Note 1).

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 highest calibration solutions must be more than 1.4 times the difference between the readings for the zero solutions 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.

NOTE 2—Use of digital readout devices or printers, or both, is advisable.

NOTE 3—Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. In the case of calcium, solutions were aspirated into a nitrous oxide-acetylene flame of a premix burner. For magnesium an air-acetylene burner was used.

| | Ca | Mg |
|--------------------------------|-------|-------|
| Hollow cathode lamp, mA | 15 | 15 |
| Wavelength, nm | 422.7 | 285.2 |
| Nitrous oxide flow rate, L/min | 13.8 | ... |
| Air flow rate, L/min | ... | 22 |
| Acetylene flow rate, L/min | 6.6 | 4.2 |

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 conforming to Type I or Type II of Specification D1193. Type III or Type IV may be used if they effect no measurable change in the blank or sample.

8.3 *Calcium, Standard Solution* (1 mL = 25 µg Ca)—Dissolve 1.2487 g of anhydrous calcium carbonate (CaCO₃) in 100 mL of dilute HCl (1 + 3). When solution is complete, cool and dilute to 1 L in a volumetric flask. Transfer 10 mL of this solution to a 200 mL volumetric flask. Dilute to volume and mix.

8.4 *Calcium and Magnesium Calibration Solutions*—Using pipets, transfer, (5.0, 10.0, 20.0, 40.0, and 50.0) mL portions of

the standard calcium and magnesium solutions to 200-mL volumetric flasks (Note 4). Dilute to 90 mL and add 6 mL of HCl, 60 mL of background solution, and 40 mL of LaCl₃ solution. Dilute to volume and mix.

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 HNO₃. Evaporate to a syrupy consistency. Add 20 mL of HCl and dilute to 200 mL with water. Add 20 g of sodium chloride dissolved in 200 mL water and dilute to 1 L with water.

8.6 *Lanthanum Chloride Solution* (50 g/L)—Dissolve 50 g of LaCl₃ in 50 mL of HCl and 300 mL of hot water. Cool and dilute to 1 L.

8.7 *Magnesium, Standard Solution* (1 mL = 17.5 µg Mg)—Dissolve 0.3500 g of high-purity magnesium by slowly adding 75 mL of HCl (1 + 3). When dissolved, dilute with water to 1 L in a volumetric flask. Transfer 10 mL of this solution to a 200 mL volumetric flask, dilute to volume, and mix.

8.8 *Zero Calibration Solution*—Transfer 60 mL of the background solution to a 200 mL volumetric flask, add 6 mL of HCl, 40 mL of LaCl₃ solution, dilute to volume, and mix.

NOTE 4—The range of calcium or magnesium that can be covered may vary from instrument to instrument. Attention should be paid to the minimum criteria given in 7.1.

9. Hazards

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

10. Sample Preparation

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

NOTE 5—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 milliliters of water, add 25 mL of HCl, cover, and digest below the boiling point until no further attack is apparent. Add 2 mL of HNO₃ 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 minutes. Add 50 mL of water and warm until soluble salts are in solution. Filter on 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.

fine-textured paper, and collect the filtrate in a 250 mL beaker. Transfer the residue quantitatively to the filter paper. Wash alternately three times with hot HCl (1 + 9) and hot water until the yellow color of ferric chloride is no longer apparent. Transfer the paper and residue to a platinum crucible. Reserve the filtrate.

12. Calculation

12.1 Only 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

TABLE 1 Dilution Guide for Test Solution

| Element | Expected Percentage of Element in Sample | Aliquot, mL | Equivalent Mass of Sample, g | Background Solution to be Added, mL | Hydrochloric Acid Added, mL |
|-----------|--|-------------|------------------------------|-------------------------------------|-----------------------------|
| Calcium | 0.05 to 0.5 | 50 | 0.25 | 45 | 4 |
| Calcium | 0.5 to 2.5 | 10 | 0.05 | 60 | 6 |
| Calcium | 2.5 to 10.0 | 2 | 0.01 | 60 | 6 |
| Magnesium | 0.05 to 0.25 | 40 | 0.20 | 50 | 4 |
| Magnesium | 0.25 to 1.25 | 10 | 0.05 | 60 | 6 |
| Magnesium | 1.25 to 5.00 | 2 | 0.01 | 60 | 6 |

11.4 *Treatment of Residue*—Ignite the paper and residue in the platinum crucible. Cool, moisten with several drops of water, add three or four drops of dilute H₂SO₄ (1 + 1) and 10 mL of HF. Evaporate slowly to expel silica and then fume off the excess of H₂SO₄. Finally, ignite at about 700 °C. Add 1 g of anhydrous sodium carbonate 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. Transfer a proper aliquot of this solution (see Table 1) to a 200 mL volumetric flask and add 40 mL of the LaCl₃ solution. Add the amount of background solution and HCl indicated in Table 1, dilute to volume, and mix.

NOTE 6—Dilutions shown in Table 1 will provide concentrations of calcium and magnesium falling within the range of the calibration solutions.

11.5 *Adjustment of Atomic Absorption Spectrometer*—In accordance with Practice E663, set the initial instrument parameters to the values in Note 3. 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 7—**Warning**—The manufacturer’s instructions for igniting and extinguishing 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.

manner, obtain the net absorbance of the test solution by subtracting the absorbance of the reagent blank solution.

12.2 Prepare calibration curves for calcium and magnesium by plotting the net absorbance values of the calibration solutions against micrograms of calcium or magnesium per milliliter.

12.3 Convert the net absorbance values of the sample solution to micrograms of calcium and magnesium per milliliter by means of the calibration curves.

12.4 Calculate the mass fraction (%) of calcium and magnesium as follows:

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

where:

A = measured concentration of calcium or magnesium per µg/mL, and

B = sample represented in the 200 mL volume of the final test solution, g.

12.5 Rounding of test results obtained using this test method shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

13. Precision and Bias⁵

13.1 *Precision*—Table 2 indicates the precision of the test method within and between laboratories when applied to a number of samples of iron ore.

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 atomic absorption spectrometry; calcium; iron ores; magnesium

⁵ Supporting data giving the results of cooperative testing have been filed at ASTM International Headquarters. Request RR:E16-1001. Contact ASTM Customer Service at service@astm.org.