



Designation: E 508 – 03

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

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1. Scope

1.1 This test method covers the determination of calcium and magnesium in iron ores, concentrates, and agglomerates in the concentration range from 0.05 to 5 % of calcium and 0.05 to 3 % of magnesium.

1.2 *This standard does not purport to address all of the safety problems, 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

D 1193 Specification for Reagent Water

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores and Related Materials

E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials

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

2.2 *Other Documents*

E 663-86 (1991) Practice for Flame Atomic Absorption Analysis³

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 Available from Global Engineering Documents, 15 Inverness Way, East Englewood, CO 80112-5704

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 is dissolved in hydrochloric acid with the addition of a small amount of nitric acid. 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 spectrophotometry 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 percent amounts, interfere with the calcium determination. Also, lanthanum chloride 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 **E 882**.

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 Spectrophotometer*, 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, relative to the measurement of the highest calibration solution.

NOTE 2—Use of a strip chart recorder or digital readout device, 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, λ , Å	4227	2852
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 available⁴. 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 D 1193.

8.3 *Calcium, Standard Solution (1 mL = 25 μ g Ca)*—Dissolve 1.2487 g of anhydrous calcium carbonate in 100 mL of dilute hydrochloric acid (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, add 6 mL of HCl (sp gr 1.19), 60 mL of background solution, and 40 mL of lanthanum chloride 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, (sp gr 1.19) and oxidize by

the dropwise addition of nitric acid (HNO₃, sp gr 1.42). Evaporate to a syrupy consistency. Add 20 mL of HCl (sp gr 1.19) and dilute to 200 mL with water. Add 20 g of sodium chloride (NaCl) dissolved in 200 mL water and dilute to 1 L with water.

8.6 *Lanthanum Chloride Solution (50 g/L)*—Dissolve 50 g of lanthanum chloride in 50 mL of HCl (sp gr 1.19) and 300 mL of hot water. Cool and dilute to 1 L.

8.7 *Magnesium, Standard Solution (1 mL = 17.5 μ g 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 (sp gr 1.19), 40 mL of lanthanum chloride 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 Practice E 50.

10. Sample Preparation

10.1 The analytical sample shall be pulverized to pass a No. 100 (150- μ m) sieve (see Test Method E 276).

NOTE 5—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μ m) sieve. Table 1

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 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 weight loss. Transfer the sample to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two weights is the weight 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 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 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.

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