



Standard Test Method for Calcium and Magnesium in Iron Ores by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation E 508; 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 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:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

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 Materials²

E 663 Practice for Flame Atomic Absorption Analysis³

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

3. Summary of Test Method

3.1 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 spectroscopy 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

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² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

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.

4. Significance and Use

4.1 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.

5. Interferences

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

6. Apparatus

6.1 *Atomic Absorption Spectrometer*, meeting the following criteria:

6.1.1 *Minimum Sensitivity*—The absorbance of the highest calibration solution (see 7.3) must be at least 0.3.

6.1.2 *Curve-Linearity*—The difference between the readings of the highest calibration solutions (40 to 50 mL) must be more than 1.4 times the difference between the readings for the zero solutions and the lowest calibration solution (0 to 5 mL).

6.1.3 *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	...

 **E 508**

Air flow rate, L/min	...	22
Acetylene flow rate, L/min	6.6	4.2

7. Reagents

7.1 *Purity and Concentration of Reagents*—The purity and concentration of the common chemical reagents used shall conform to Practices E 50. Special apparatus and reagents required are located in separate sections preceding the procedure.

7.2 *Calcium, Standard Solution* (1 mL = 25 µg 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.

7.3 *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 100 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.

7.4 *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.

7.5 *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.

7.6 *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.

7.7 *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 6.1.

8. Sample Preparation

8.1 The analytical sample shall be pulverized to pass a No. 100 (150-µm) 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-µm) sieve.

9. Procedure

9.1 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.

9.2 Carry a reagent blank through all steps of the procedure starting with 9.3.

9.3 *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 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.

9.4 *Treatment of Residue*—Ignite the paper and residue in the platinum crucible. Cool, moisten with several drops of water, add 3 or 4 drops of dilute sulfuric acid (H₂SO₄, 1+1) and 10 mL of hydrofluoric acid (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 (Na₂CO₃) 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 9.3, 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 lanthanum chloride 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.

9.5 *Adjustment of Atomic Absorption Spectrometer*—In accordance with Practice E 663, set the initial instrument parameters to the values in Note 3 of 6.1.3. 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—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.

9.6 *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