



Designation: **E372 – 01 (Reapproved 2006) E372 – 13**

Standard Test Method for Determination of Calcium and Magnesium in Magnesium Ferrosilicon by EDTA Titration¹

This standard is issued under the fixed designation E372; 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 chemical analysis of magnesium ferrosilicon having chemical compositions within the following limits:

Element	Concentration Range, %
Element	Composition Range, %
Aluminum	2.0 max
Calcium	0.25 to 3.00
Carbon	0.50 max
Cerium	1.0 max
Chromium	0.50 max
Magnesium	2.00 to 12.00
Manganese	1.0 max
Silicon	40.00 to 55.00
Sulfur	0.025 max
Titanium	0.2 max

1.2 *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.* For general precautions to be observed in this test method, refer to Practices **E50**.

2. Referenced Documents

2.1 *ASTM Standards:*²

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E32 Practices for Sampling Ferrous Alloys and Steel Additives for Determination of Chemical Composition

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

E60**E135** Practice for Analysis of Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials by Spectrophotometry

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)³

3. Terminology

3.1 For definitions of terms used in this test method refer to Terminology **E135**.

4. Significance and Use

4.1 This test method for the chemical analysis of metals and alloys is primarily intended to test such materials 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.

¹ This test method is under the jurisdiction of ASTM Committee **E01** on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee **E01.01** on Iron, Steel, and Ferrous Alloys.

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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.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E50, except that photometers shall conform to the requirements prescribed in Practice E60.

4.2 Photometric practice prescribed in this test method shall conform to Practice E60.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents and equipment in this test method, refer to Practices E50.

5.2 Specific hazard statements are given in 13.7.1 and 14.1.

6. Sampling

6.1 For procedures for sampling the material, refer to MethodsPractices E32.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places as directed in 3.4 to 3.6 of Practice E29.

8. Interlaboratory Studies

8.1 This test method has been evaluated in accordance with Practice E173, unless otherwise noted in the precision and bias section.

CALCIUM AND MAGNESIUM BY THE (ETHYLENEDINITRIL)TETRAACETIC ACID (EDTA) TITRIMETRIC METHOD

9. Scope

9.1 This test method covers the determination of magnesium in concentrations/compositions from 2 % to 12 % and calcium in concentrations/compositions from 0.25 % to 3.0 %.

10. Summary of Test Method

10.1 After dissolution of the sample in nitric and hydrofluoric acids, an ammonium hydroxide precipitation is made to separate other elements from calcium and magnesium. Calcium, and magnesium plus calcium are titrated in separate aliquot portions after adding triethanolamine and potassium cyanide to mask residual traces of iron, copper, nickel, manganese, and aluminum that may be present. Calcium is titrated with disodium (ethylenedinitrilo)tetraacetate (EDTA) at pH 12. Magnesium plus calcium is titrated with EDTA at pH 10.0 and the magnesium concentration/content is calculated by correcting for the volume of EDTA required to titrate the calcium.

11. Interferences

11.1 Provision is made for the removal or masking of interfering elements ordinarily present in magnesium ferrosilicon.

12. Apparatus

12.1 *Beakers*, TFE-fluorocarbon 500-mL.

12.2 *pH Meter*—Meter. Apparatus No. 3A.

13. Reagents

13.1 *Ammonium Chloride Buffer Solution* (pH 10.0)—Dissolve 60 g of ammonium chloride (NH₄Cl) in 200 mL of water, add 570 mL of NH₄OH, and dilute to 1 L.

13.2 *Calcium, Standard Solution* (1 mL = 0.2002 mg Ca)—Dissolve 0.5000 g of calcium carbonate (CaCO₃) (purity: 99.9 % min) in 100 mL of HCl (5+95)-(5 + 95). Boil 1 min, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

13.3 *Disodium Ethylenedinitrilo-Tetraacetate Dihydrate (EDTA), Standard Solution*Solution: (0.005 M)—Prepare a solution as directed for Reagent No. 22, using 1.8613 g instead of the specified weight.)

13.3.1 Dissolve 1.8613 g of EDTA in water; transfer to a 1-L volumetric flask; dilute to volume; and mix. The solution will remain stable for several months when stored in plastic or borosilicate glass bottles.

13.3.2 Standardize the solution as follows: Using a pipet, transfer 25 mL of the calcium solution (1 mL = 0.2002 mg Ca) to a 250-mL beaker, add 1 mL of MgCl₂ (13.6) solution and 100 mL of water, and proceed as directed in 13.414.4.