Designation: ~~E372 – 13~~ E372 – 21

Standard Test Method for Determination of Calcium and Magnesium in Magnesium Ferrosilicon by EDTA ~~Titration~~Titrimetry[1]

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	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~~ safety, health, and ~~health~~environmental 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.

1.3 *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]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E32 Practices for Sampling Ferroalloys 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
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

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

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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.

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.

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~~to sample the material, refer to Practices E32.

7. Rounding Calculated Values

7.1 ~~Calculated values shall be rounded to the desired number of places~~ 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.

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 ~~(ETHYLENEDINITRILO)TETRAACETIC ACID (EDTA) TITRIMETRIC~~(ETHYLENEDINITRILO) TETRAACETIC ACID DISODIUM SALT (EDTA) TITRIMETRY METHOD

~~9. Scope~~

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

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10. Summary of Test Method

10.1 After dissolution of the sample in ~~nitric~~HNO_3 and ~~hydrofluoric acids,~~ HF, an ~~ammonium~~NH_4 ~~hydroxide~~ OH 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~~(ethylenedinitrilo) tetraacetic acid disodium salt (EDTA) at pH ~~12.~~12.5. Magnesium plus calcium is titrated with EDTA at pH ~~10.0~~10.0, and the magnesium 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.~~500 mL.

12.2 *pH Meter.*

13. Reagents

13.1 *Ammonium Chloride Buffer Solution* (pH 10.0)—Dissolve 60 g of ammonium chloride (NH_4Cl) in 200 mL of water, add 570 mL of NH_4OH, and dilute to 1 L.

13.2 *Calcium, Standard Solution* (1 mL = 0.2002 mg Ca)—Dissolve 0.5000 g of calcium carbonate ($CaCO_3$) (purity: 99.9 % min) in 100 mL of HCl (5 + 95). Boil 1 min, cool, transfer to a ~~1-L~~1 L volumetric flask, dilute to volume, and mix.

13.3 ~~*Disodium Ethylenedinitrilo-Tetraacetate Dihydrate*~~ *(Ethylenedinitrilo) Tetraacetic Acid, Disodium Salt (EDTA), Standard Solution:* (0.005 *M*)

NOTE 1—The complete chemical compound name, (ethylenedinitrilo) tetraacetic acid disodium salt, is commonly referred to as EDTA.

13.3.1 Dissolve 1.8613 g of EDTA in water; transfer to a ~~1-L~~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. Containers used for the storage of dilute solutions of EDTA should be pretreated with a hot alkaline EDTA solution (10 g/L) and rinsed with water.

13.3.2 Standardize the solution as follows: Using a pipet, ~~tranfer~~transfer 25 mL of the calcium solution (1 mL = 0.2002 mg Ca) to a ~~250-mL~~250 mL beaker, add 1 mL of $MgCl_2$ (13.6) solution and 100 mL of water, and proceed as directed in 14.4.

~~NOTE 1—Containers used for the storage of dilute solutions of EDTA should be pretreated with a hot alkaline EDTA solution (10 g/L), and rinsed with water.~~

13.3.3 Calculate the calcium equivalent of the EDTA solution as follows:

$$C\text{alcium equivalent, mg/m}L = A/B \quad (1)$$

where:

A = calcium, mg, and
B = EDTA solution required to titrate the calcium solution, mL.

13.3.4 Calculate the magnesium equivalent of the solution as follows:

$$M\text{agnesium equivalent, mg/m}L = C \times 0.6068 \quad (2)$$

where C = calcium equivalent (13.3.3).

13.4 *Eriochrome Black-T Indicator Solution* (6 g/L ~~of~~in methanol)—Dissolve 0.3 g of Eriochrome Black-T and 1 g of sodium borate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in 50 mL of methanol. Do not use a solution that has stood for more than ~~8 h.~~8 h.

13.5 *Hydroxy Naphthol Blue Mixture*—Add 1.0 g of hydroxy napthol blue indicator to 100 g NaCl and mix thoroughly.

13.6 *Magnesium Chloride* (2.5 g/L) —Dissolve 0.25 g of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) in 50 mL of water, and dilute to 100 mL.

13.7 *Potassium Cyanide Solution* (50 g/L)—Dissolve 2 g of ~~potassium hydroxide (KOH)~~ KOH in water, add 5 g of potassium cyanide (KCN) (**Warning;** see 13.7.1), dilute to 100 mL, and transfer to a plastic bottle.

13.7.1 **Warning:** The preparation, storage, and use of KCN ~~require~~requires care and attention. Avoid inhalation of fumes and exposure of the skin to the chemical and its solutions. Work in a well-ventilated hood. Refer to Section 8 of Practices E50.