



Standard Test Method for the Determination of Boron in Ferroboron¹

This standard is issued under the fixed designation E 371; 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 These test methods cover the chemical analysis of ferroboron having chemical compositions within the following limits:

Element	Concentration, %
Aluminum	2.00 max
Boron	12.0–24.0
Carbon	1.50 max
Chromium	0.75 max
Copper	0.15 max
Lead	0.01 max
Manganese	1.00 max
Molybdenum	1.50 max
Nickel	0.10 max
Phosphorus	0.06 max
Silicon	4.00 max
Sulfur	0.02 max

1.2 The test methods in this standard are contained in the sections indicated below:

	Section
Boron by the Ion-exchange Titrimetric Method (12 to 24 %)	9

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 5 and in special “Warning” paragraphs throughout these test methods.

2. Referenced Documents

2.1 ASTM Standards:

A 323 Specification for Ferroboron²

D 1193 Specification for Reagent Water³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

E 32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition⁵

E 50 Practices for Apparatus, Reagents, and Safety Precau-

tions for Chemical Analysis of Metals⁵

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁶

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁵

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications, particularly those under the jurisdiction of ASTM Committees A1 on Stainless Steel and Related Alloys, and specifically Specification A 323. It is assumed that all who use these test methods 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 under appropriate quality control practices such as those described in Guide E 882.

4. Reagents

4.1 Reagents:

4.1.1 *Purity of Reagents*—Unless otherwise indicated, all reagents used in these test methods shall conform to the “Reagent Grade” Specifications of the American Chemical Society.⁷ Other chemicals may be used, provided it is first ascertained that they are of sufficiently high purity to permit their use without adversely affecting the expected performance of the determination, as indicated in the section on Precision and Bias.

4.1.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specifications D 1193.

5. Hazards

5.1 For hazards to be observed in the use of certain reagents and equipment in these test methods, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, and for

¹ These test methods are 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 Ferroalloys.

Current edition approved Nov. 10, 1999. Published January 2001. Originally published as E371 – 71 T. Last previous edition E 371–99.

² *Annual Book of ASTM Standards*, Vol 01.02.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.

⁶ Discontinued 1998; see *1997 Annual Book of ASTM Standards*, Vol 03.05.

⁷ *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 *Analar 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.

particle size of the sample for chemical analysis, refer to Practices E 32.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded off to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

8. Interlaboratory Studies

8.1 These test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision and bias section.

BORON BY THE ION-EXCHANGE TITRIMETRIC TEST METHOD

9. Scope

9.1 This test method covers the determination of boron in concentrations from 12.0 to 24.0 %.

10. Summary of Test Method

10.1 The sample is fused with sodium peroxide. The fusion is leached with acid under a reflux condenser, and the solution passed through a column of a strong acid-type ion-exchange resin to remove interfering cations. The effluent is nearly neutralized, refluxed to remove carbon dioxide, and cooled. After adjusting the pH, invert sugar or mannitol is added and the solution titrated potentiometrically to pH 6.9 with standard sodium hydroxide solution.

11. Apparatus

11.1 *Florence Flasks*, 250 and 500-mL capacity (low-boron glass).

11.2 *Reflux Condenser*, having an inner tube made of low-boron glass.

11.3 *Iron Crucible*, 30-mL—Crucibles made from No. 20 gage (0.97 mm, 0.038 in.) ingot iron are suitable.

11.4 *Ion-Exchange Apparatus*—Use a glass column (2 cm in diameter and 25 cm long) fitted with a stopcock and a reservoir cup (approx. 100-mL capacity). Lightly insert a glass wool plug above the stopcock.

12. Reagents

12.1 *Boron, Standard Solution* (1 mL = 0.00054 g B)—Pulverize 5 g of boric acid (H_3BO_3) to pass a No. 60 (250- μ m) sieve, and dry in vacuum over anhydrous magnesium perchlorate at room temperature to constant weight. Dissolve 3.092 g of the dry chemical in 500 mL of hot water, and cool. Transfer to a 1-L volumetric flask, dilute to volume with room temperature, recently boiled water, and mix.

12.2 *Ferrous Sulfate* ($FeSO_4$).

12.3 *Invert Sugar Solution*—Dissolve 1000 g of granulated sugar in 650 mL of previously boiled hot water and add 8 mL of 1 N HCl. Heat at 80 to 90° C for 2 h. Using a pH meter, adjust the pH to 6.9 with NaOH solution just before use.

12.4 *Ion-Exchange Resin*—Use a cation exchange resin of the 8 % cross linkage type, passing a No. 50 (300- μ m) sieve,

but retained on a No. 100 (150- μ m) sieve.⁸ To prepare the ion-exchange apparatus, place a 4-cm plug of glass wool in the bottom of the column, fill the tube with resin, wash with 100 mL of HCl (1 + 2), and then wash with water until neutral to litmus paper. During the washing process, adjust the flow rate to approximately 20 mL/minute.

12.5 *Mannitol*, neutral, may be used in the procedure in place of invert sugar solution.

12.6 *Methyl Orange Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl orange in 100 mL of water. Filter if necessary.

12.7 *Sodium Hydroxide, Standard Solution* (1 mL equivalent to approx., 0.001 g B)—Transfer 4 g of carbonate-free sodium hydroxide (NaOH) to a polyethylene beaker and dissolve it in 100 mL of freshly boiled and cooled water. Transfer the solution to a 1-L polyethylene volumetric flask and dilute to the mark with more of the freshly boiled and cooled water. Mix. Transfer the solution to a polyethylene storage container fitted with an Ascarite-II U-tube to protect it from atmospheric carbon dioxide. Standardize against the boron solution as follows: Using a pipet, transfer 50 mL of the boron solution (1 mL = 0.00054 g B) to a 500-mL flask of low-boron glass, add 250 mL of water, and proceed as directed in 13.4 and 13.5, starting with the addition of the methyl orange solution. Calculate the boron equivalent of the NaOH solution as follows:

$$\text{Boron equivalent, g/mL} = A/B \quad (1)$$

where:

A = grams of boron taken, and

B = millilitres of NaOH solution required to titrate the boron.

12.8 *Sodium Peroxide* (Na_2O_2).

13. Procedure

13.1 Transfer a 1-g sample, weighed to the nearest 0.5 mg, to an iron crucible, add 10 g of Na_2O_2 , and mix well. Fuse the mixture (Note 1) carefully by first playing the flame of a laboratory burner cautiously on the surface of the mixture until fusion begins, and then revolving the crucible in the outer edge of the flame by gripping with a pair of tongs and rotating vigorously, gradually raising the temperature to about 900° C, until decomposition is complete.

NOTE 1—**Warning:** Use proper safety practices and equipment when performing sodium peroxide fusions.

13.2 Cover the crucible, cool to room temperature, and tap on a solid object to loosen the cake. Transfer the cake to a 250-mL Florence flask and connect the flask to the reflux condenser. Rinse the crucible with 50 mL of HCl (3 + 2), in small portions, and add the rinsings to the flask through the condenser. If the cake is not decomposed, add enough HCl to decompose it and make the solution just acidic to litmus, and boil gently for 5 to 10 min. Cool, disconnect the flask from the condenser, add 1 g of $FeSO_4$, agitate the flask, and blow a current of air on the surface of the solution to expel chlorine. Transfer to a 250-mL volumetric flask, dilute to volume, and

⁸ Dowex-50 resin has been found satisfactory for this purpose.

mix. Using a pipet, transfer 50 mL, equivalent to 0.2 g of the sample, to a 150-mL beaker and dilute to approximately 100 mL.

13.3 Pass the solution through the resin column, and wash the column with 200 mL of water, in small portions.

NOTE 2—Regenerate the resin before reuse by washing with HCl (1 + 2) until the iron is removed, and finally with water to remove the acid.

13.4 Transfer the effluent to a 500-mL flask of low-boron glass, add 5 drops of methyl orange indicator solution, and nearly neutralize by adding NaOH pellets. Boil the solution under a reflux condenser for 5 min to remove any carbon dioxide, cool to room temperature, and transfer to a 600-mL beaker.

13.5 Using a pH meter, adjust the pH to 6.9 by adding NaOH solution from a buret. Add sufficient invert sugar solution or mannitol to provide a concentration of 0.6 M (13 mL of invert sugar solution or 11 g of mannitol per 100 mL of solution). Titrate immediately to pH 6.9 with standard NaOH solution.

13.6 *Reagent Blank*—Carry a reagent blank through the entire procedure using the same amounts of all reagents but with the sample omitted.

14. Calculation

14.1 Calculate the percentage of boron as follows:

$$\text{Boron, \%} = [(A - B)C/D] \times 100 \quad (2)$$

where:

A = millilitres of NaOH solution required for titration of the sample,

B = millilitres of NaOH solution required for titration of the blank,

C = boron equivalent of the NaOH solution, and
D = grams of sample used.

15. Precision and Bias

15.1 *Precision*—Four laboratories cooperated in testing this method and obtained the data summarized in Table 1. Samples with boron concentrations near the upper limit of the scope were not available for testing. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this method is adequate for the contemplated use.

15.2 *Bias*—No information on the bias of this test method is known. The bias of this test method may be judged by comparing accepted reference values with the corresponding arithmetic average obtained by interlaboratory testing.

16. Keywords

16.1 chemical analysis; ferroboron

TABLE 1 Ion-Exchange Trimetric Method Statistical Information—Boron

Test Material	Boron Found, %			
	Lab A	Lab B	Lab C	Lab D
I. Ferroboron	13.54	13.73	13.60	13.70
	13.62	13.78	13.60	13.66
	13.54	13.76	13.65	13.66
	13.57			13.65
	13.54			13.68
	13.60			13.72
				13.73
Average	13.57	13.76	13.62	13.67
Grand Average		13.66		13.68

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