Designation: D8213 – 18

Standard Test Method for Determination of Boron in Coal[1]

This standard is issued under the fixed designation D8213; 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 describes a procedure for the analysis of total boron in coal.

1.2 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D121 Terminology of Coal and Coke
D1193 Specification for Reagent Water
D2013 Practice for Preparing Coal Samples for Analysis
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D5865 Test Method for Gross Calorific Value of Coal and Coke
D6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry
D6357 Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Furnace Atomic Ab
E144 Practice for Safe Use of Oxygen Combustion Vessels
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *SAI Standard:*[3]

AS 1038.10.3 Coal and Coke—Analysis and Testing, Part 10.3: Determination of Trace Elements—Coal and Coke—Determination of Boron Content—ICP-AES Method

3. Terminology

3.1 *Definitions*—Definitions applicable to these test methods are listed in Terminology D121.

4. Summary of Test Method

4.1 Total boron is determined following one of five test preparation procedures:

4.1.1 *Procedure A*—The coal or coke to be analyzed is combusted in a vessel with dilute nitric acid absorbing the boron vapors. The combustion vessel is rinsed into a vessel with dilute nitric acid.

4.1.2 *Procedure B*—The coal or coke to be analyzed is ashed under controlled conditions. The residue is digested by aqua-regia, and dissolved in 1 % nitric acid.

4.1.3 *Procedure C*—The coal or coke to be analyzed is ashed under controlled conditions. The residue is digested in a mixture of hydrofluoric, nitric, and hydrochloric acids.

4.1.4 *Procedure D*—Following AS 1038.10.3, the coal or coke to be analyzed is ignited at 800 °C with Eschka mixture. The residue is dissolved in dilute hydrochloric acid.

4.1.5 *Procedure E*—The coal or coke to be analyzed is digested using a microwave system following manufacturer's recommendation for temperatures, power, and time, using either the acid mixtures specified in Test Methods D6357 or Test Method D6349, depending upon instrument capabilities.

4.1.6 Combustion residues are digested on an as-received basis.

---

[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

Current edition approved Dec. 1, 2018. Published February 2019. DOI: 10.1520/D8213-18.

[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 SAI Global, Level 37, 680 George Street, Sydney NSW 2000, Australia, https://www.saiglobal.com/.

4.1.7 The concentration of boron is determined by either inductively coupled atomic emission spectrometry (ICPAES) or inductively coupled plasma mass spectrometry (ICPMS). Selected elements that occur at concentrations below the detection limits of ICPAES can be quantitatively analyzed by graphite furnace atomic absorption spectrometry (GFAAS).

5. Significance and Use

5.1 The possible emission of boron that may be found in coal from coal combustion is an environmental concern.

5.2 When test portions are burned according to this procedure, the total boron is quantitatively retained and is representative of concentrations in the whole coal.

6. Apparatus

6.1 *Balance*—capable of weighing the analysis sample to the nearest 0.1 mg.

6.2 *Procedure A:*

6.2.1 *Combustion Vessel,* constructed of materials that are not affected by the combustion process or products. The vessel must be designed so that all liquid combustion products can be quantitatively recovered by washing the inner surfaces. There must be no gas leakage during the test. The vessel must be capable of withstanding a hydrostatic-pressure test to approximately 20 MPa at room temperature without stressing any part beyond its elastic limit.

6.2.2 *Water Bath*—A container large enough to hold the combustion vessel and enough cooling water to dissipate the heat generated during the combustion process. The container shall be designed to allow a constant flow of water around the combustion vessel.

6.2.3 *Combustion Crucibles*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy.

6.2.4 *Firing Wire,* 100 mm, nickel-chromium alloy, No. 34B & S gauge, or platinum, No. 34 or No. 38B & S gauge.

6.2.5 *Firing Circuit*—A 6 V to 16 V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A step-down transformer connected to an alternating-current lighting circuit or batteries can be used. (**Warning—**The ignition circuit switch shall be of the momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the charge.)

6.2.6 *Spectrometer*—Refer to 6.1 (*Inductively Coupled Plasma Atomic Emission Spectrometer (ICPAES)*) or 6.2 (*Inductively Coupled Plasma Mass Spectrometer (ICPMS)*) of Test Methods D6357.

6.2.7 *Argon Gas Supply,* high purity (99.99 %).

6.3 *Procedure B:*

6.3.1 *Spectrometer*—Refer to 6.1 (*Inductively Coupled Plasma Atomic Emission Spectrometer (ICPAES)*) or 6.2 (*Inductively Coupled Plasma Mass Spectrometer (ICPMS)*) of Test Methods D6357.

6.3.2 *Argon Gas Supply,* high purity (99.99 %).

6.3.3 *Muffle Furnace*—Refer to 6.4 (*Muffle Furnace*) of Test Methods D6357.

6.3.4 *Teflon Beakers*—Refer to 6.6 (*Teflon Beakers*) of Test Methods D6357.

6.3.5 *Hot Plate*—Refer to 6.7 (*Hot Plate*) of Test Methods D6357.

6.3.6 *Volumetric Flasks*—Refer to 6.8 (*Volumetric Flasks*) of Test Methods D6357.

6.3.7 *HDPE Bottles*—Refer to 6.9 (*HDPE Bottles*) of Test Methods D6357.

6.3.8 *Crucibles*—Refer to 6.10 (*Crucibles*) of Test Methods D6357.

6.4 *Procedure C:*

6.4.1 *Spectrometer*—Refer to 6.1 (*Inductively Coupled Plasma Atomic Emission Spectrometer (ICPAES)*) or 6.2 (*Inductively Coupled Plasma Mass Spectrometer (ICPMS)*) of Test Methods D6357.

6.4.2 *Argon Gas Supply,* high purity (99.99 %).

6.4.3 *Muffle Furnace*—Refer to 6.4 (*Muffle Furnace*) of Test Methods D6357.

6.4.4 *Crucibles*—Refer to 6.10 (*Crucibles*) of Test Methods D6357.

6.4.5 *Stirring Hotplate and Bars*—Refer to 7.5 (*Stirring Hotplate and Bars*) of Test Method D6349.

6.4.6 *Polycarbonate Bottles*—Refer to 7.6 (*Polycarbonate Bottles*) of Test Method D6349.

6.5 *Procedure D:*

6.5.1 Refer to Section 7 (*Apparatus*) of AS 1038.10.3.

6.6 *Procedure E:*

6.6.1 *Spectrometer*—Refer to 6.1 (*Inductively Coupled Plasma Atomic Emission Spectrometer (ICPAES)*) or 6.2 (*Inductively Coupled Plasma Mass Spectrometer (ICPMS)*) of Test Methods D6357.

6.6.2 *Argon Gas Supply,* high purity (99.99 %).

6.6.3 *Microwave Sample Preparation System*—Capable of rapid heating of the sample at high pressures.

6.6.4 *High Pressure Digestion Vessel*—Closed vessel, capable of withstanding the temperatures and pressures selected, and suitable for use with the acid mixture selected.

7. Reagents

7.1 *Purity of Reagents*—All acids used in these test methods must be trace metal purity grade or equivalent. Redistilled acids are acceptable.

7.2 *Purity of Water*—The purity of the water used in these test methods shall be equivalent to ASTM Type II reagent water of Specification D1193.

7.3 *Calibration Standards*—Aqueous single-element solutions made up in 10 % HNO_3 are used for calibration of ICPAES and ICPMS systems. The standards may be purchased or prepared from high-purity grade chemicals or metals.

7.4 *Boron Stock Standard Solution (1000 ppm)*—Single-element standards either purchased or prepared from high purity grade chemicals or metals.

7.5 *Nitric Acid (1+9)*—Dilute 100 mL of concentrated nitric acid (HNO_3, sp gr 1.42) to 1 L with water.

7.6 *Blank Solutions*—All of the test methods in this standard require two types of blank solutions. A calibration blank that is used to establish the analytical calibration curve and a method blank which is used to evaluate possible contamination and assess spectral background.

7.6.1 *Calibration Blank*—A 1 % nitric acid solution.

7.6.2 *Method Blank*—Consists of all the reagents in the same volumes as used in preparing the samples. The method blank shall be processed through the entire sample digestion scheme.

7.7 *Procedure A:*

7.7.1 *Oxygen*—Manufactured from liquid air, guaranteed to be greater than 99.5 % pure, and free of combustible matter. Oxygen made by the electrolytic process contains small amounts of hydrogen rendering it unfit unless purified by passage over copper oxide at 500 °C.

7.8 *Procedure B:*

7.8.1 *Aqua Regia Solution*—Refer to 7.3 (*Aqua Regia Solution*) of Test Methods D6357.

7.9 *Procedure C:*

7.9.1 *Mixed Acid Solution, 70/30 HCl/HF*—Refer to 8.5.5 (*Mixed Acid Solution, 70/30 HCl/HF*) of Test Method D6349.

7.10 *Procedure D:*

7.10.1 Refer to Section 6 (*Reagents*) of AS 1038.10.3.

7.11 *Procedure E:*

7.11.1 *Hydrochloric Acid*

7.11.2 *Hydrofluoric Acid*

7.11.3 *Nitric Acid*

8. Hazards

8.1 *Procedure A:* Refer to Section 8 (*Hazards*) of Test Method D5865.

9. Sample

9.1 The analysis sample is the material pulverized to pass 250 µm (No. 60) sieve, prepared in accordance with Practice D2013.

10. Test Solution Preparation

10.1 *Procedure A: Vessel Combustion*

10.1.1 Thoroughly mix the analysis sample of coal. Carefully weigh approximately 1 g ± 0.2 g into a previously ignited crucible in which it is to be combusted.

10.1.2 For samples in excess of 5 % sulfur, the mass of coal must be reduced to 0.5 g ± 0.1 g to ensure that all the acidic vapors produced in the combustion process are quantitatively retained in solution.

10.1.3 Transfer 10 mL of HNO_3 (1+9) to the combustion vessel. Attach the fuse wire to the vessel electrodes. Place the crucible with the sample into the electrode support of the vessel, and insert the fuse wire so that it just touches the surface of the sample.

10.1.4 Assemble the vessel in the normal manner and charge it with oxygen to a pressure between 2.0 MPa to 3.0 MPa. If the oxygen should exceed the specified pressure, do not proceed with the combustion. In this case, detach the filling connection, exhaust the vessel in the usual manner, and discard the sample. (**Warning—**The following precautions are recommended for safe operations in the use of the oxygen combustion vessel. Additional precautions are given in Recommended Practice E144, for use of oxygen combustion vessels.)

10.1.4.1 The mass of coal sample and the pressure of the oxygen admitted to the vessel must not exceed the vessel manufacturer's recommendation.

10.1.4.2 Inspect the vessel parts carefully after each use. Frequently check the threads on the main closure for wear. Replace the cracked or significantly worn parts. Return the vessel to the manufacturer occasionally for inspection and possibly proof testing.

10.1.4.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gauge used in regulating the oxygen feed to the vessel. Valves, gauges, and gaskets must meet industry safety code. Suitable reducing valves and adaptors for approximately 3.0 MPa to 5.0 MPa discharge pressure are obtainable from commercial sources of compressed-gas equipment. Check the pressure gauge periodically for accuracy.

10.1.4.4 During ignition of a sample, the operator must not permit any portion of his body to extend over the combustion vessel or its container.

10.1.4.5 Exercise extreme caution when combustion aids are employed so as not to exceed the vessel manufacturer's recommendations and to avoid damage to the vessel.

10.1.4.6 Admit oxygen slowly into the vessel to avoid blowing powdered material from the crucible.

10.1.4.7 Do not fire the vessel if it has been filled to greater 3.0 MPa pressure with oxygen, if the vessel has been dropped or turned over after loading, or if there is evidence of a gas leak when the vessel is submerged in the water bath.

10.1.5 Place the vessel in a cooling water bath, with water moving. Attach the ignition wires from the firing circuits, and ignite the sample. Allow the vessel to remain in the cooling water for 15 min to allow cooling and absorption of soluble vapors within the vessel.

10.1.6 Remove the vessel and release the pressure at a uniform rate, such that the operation will require not less than 2 min. Examine the vessel interior and discard the test if unburned or sooty deposits are found.

10.1.7 Quantitatively rinse the vessel, electrodes, and crucible into the vessel with several small portions of HNO_3 (1+9). Dilute the contents of the vessel with HNO_3 (1+9) to a total volume of 50 mL or 100 mL.

10.1.8 Prepare a method blank with each batch of samples to be analyzed.

10.2 *Procedure B: D6357 (Ashing) and Modified D6357 (Dissolution, Omitting HF)*

10.2.1 Refer to Test Methods D6357, subsection 9.1 (*Ashing*). Follow the steps as outlined for ashing.

10.2.2 Refer to Test Methods D6357, subsection 9.2 (*Dissolution*). Follow the steps as outlined for acid dissolution, omitting concentrated hydrofluoric acid (HF).

10.3 *Procedure C: D6357 (Ashing) and Modified D6349 (Mixed Acid Dissolution, Omitting H_3BO_4)*