



Designation: D 4208 – 02

Standard Test Method for Total Chlorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method¹

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1. Scope

1.1 This test method covers the analysis of total chlorine in coal.

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke³

D 3180 Practice for Calculating Coal and Coke Analysis from As-Determined to Different Bases³

E 144 Practice for Safe Use of Oxygen Combustion Bombs⁴

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁵

3. Summary of Test Method

3.1 Total chlorine is determined in this method by combusting a weighed sample in an oxygen bomb with dilute base adsorbing the chlorine vapors. The bomb is rinsed into a beaker with water and following the addition of an ionic strength adjuster, the chloride is determined by ion-selective electrode.

4. Significance and Use

4.1 The purpose of this test method is to measure the total chlorine content of coal. The chlorine content of coals may be useful in the evaluation of slagging problems, corrosion in

engineering processes, and in the total analysis of coal and coke. When coal samples are combusted in accordance with this method, the chlorine is quantitatively retained and is representative of the total chlorine content of the whole coal.

5. Apparatus

5.1 *Combustion Bomb*, constructed of materials that are not affected by the combustion process or products. The bomb 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 bomb must be capable of withstanding a hydrostatic-pressure test to 3000 psig (approximately 20 MPa) at room temperature without stressing any part beyond its elastic limit.

5.2 *Water Bath*—A container large enough to hold the combustion bomb 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 bomb.

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

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

5.5 *Firing Circuit*—A 6 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.)

5.6 *Balance*, analytical, with a sensitivity of 0.1 mg.

5.7 *Specific-Ion Meter*—A pH meter with an expandable millivolt scale, specific-ion meter, sensitive to 0.1 mV, suitable for method of standard addition determinations.⁶

¹ This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.06.

⁴ *Annual Book of ASTM Standards*, Vol 14.04.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ Midgley, D., and Torrance, K., *Potentiometric Water Analysis*, John Wiley and Sons, 1978.