



Standard Test Methods for Total Mercury in Coal and Coal Combustion Residues by Acid Extraction or Wet Oxidation/Cold Vapor Atomic Absorption¹

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1. Scope

1.1 These test methods cover procedures to determine the total mercury content in a sample of coal or coal combustion residue.

1.2 The values stated in SI units are regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 121 Terminology of Coal and Coke²

D 1193 Specification for Reagent Water³

D 2013 Method of Preparing Coal Samples for Analyses²

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²

D 5142 Test Methods for the Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures²

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases²

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System⁴

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology D 121.

4. Summary of Test Method

4.1 *Test Method A*—Mercury in the analysis sample is solubilized by heating the test sample at a specified temperature in a mixture of nitric and hydrochloric acids. The acid solutions produced are transferred into a vessel in which the

mercury is reduced to elemental mercury. The mercury vapor is determined by flameless cold-vapor atomic absorption spectroscopy.

4.2 *Test Method B*—Mercury in the analysis sample is solubilized by heating the test sample in a mixture of nitric and sulfuric acids with vanadium pentoxide.⁵ The acid solutions produced are transferred into a vessel in which the mercury is reduced to elemental mercury. The mercury vapor is determined by flameless cold-vapor atomic absorption spectroscopy.

5. Significance and Use

5.1 The emission of mercury during coal combustion can be an environmental concern.

5.2 When representative test portions are analyzed according to one of these procedures, the total mercury is representative of concentrations in the sample.

6. Apparatus

6.1 Apparatus for Test Method A:

6.1.1 *Analytical Balance*, with a sensitivity of 0.1 mg.

6.1.2 *Atomic Absorption Spectrophotometer*, with a flameless cold-vapor mercury analysis system.

6.1.3 *Digestion Vessels*, 100- to 250-mL bottles with an O-ring seal and screw cap. Bottle must be compatible for use with aqua regia. Polycarbonate and HDPE are acceptable. Bottles and cap assemblies shall be washed in 1-to-1 HCl then dried before each use.

NOTE 1—Other bottle and cap assemblies may be used provided they are compatible for use with aqua regia at a temperature of 80°C.

6.1.4 *Heat Source*, a water bath capable of maintaining a temperature of 80°C.

6.1.5 *Syringe and Filter*, a 20-cm³ syringe and a 1-μm PTFE filter to fit syringe.

6.2 Apparatus for Test Method B:

6.2.1 *Analytical Balance*, with a sensitivity of 0.1 mg.

6.2.2 *Atomic Absorption Spectrophotometer*, with a flameless cold-vapor mercury analysis system.

6.2.3 *Digestion Vessels*, 16- by 150-mm disposable glass test tubes.

¹ These Test Methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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² *Annual Book of ASTM Standards*, Vol 05.05.

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

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

⁵ Crock, J.G., *Open-File Report*, U.S. Geological Survey, 87–84, p.19.