



Designation: ~~D6722~~—~~11~~ D6722 – 19

Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Direct Combustion Analysis¹

This standard is issued under the fixed designation D6722; 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 procedures to determine the total mercury content in a sample of coal or coal combustion residue.

1.2 Units—The values stated in SI units are to be regarded as standard. ~~No other units of measurement are included in this~~ The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

1.4 *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~~ environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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](#)

[D2013/D2013M Practice for Preparing Coal Samples for Analysis](#)

[D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke](#)

[D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases](#) [1969/astm-d6722-19](#)

~~[D4621/D7448 Guide for Quality Management in an Organization That Samples or Tests](#)~~ [Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke](#) ~~(Withdrawn 2010)~~

[D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis](#)

~~[IEEE/ASTM-E691/SI-10 Standard for Use of the International System of Units \(SI\): The Modern Metric System](#)~~ [Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology [D121](#).

4. Summary of Test Method

4.1 Controlled heating of the analysis sample in oxygen is used to liberate mercury. The sample is heated to dryness in the instrument and then thermally and chemically decomposed. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace, where oxidation is completed and halogens as well as nitrogen and sulfur oxides are trapped. The remaining decomposition products are carried to a gold amalgamator that selectively traps mercury. After the system is flushed with

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.

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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.

oxygen to remove any remaining decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance peak height or peak area, as a function of mercury concentration, is measured at 253.7 nm.

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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 this procedure, the total mercury is representative of ~~concentrations~~ mass fractions in the sample.

6. Apparatus

6.1 There are several configurations of the instrumental components that can be used satisfactorily for this test method. Functionally, the instrument shall have the following components: drying compartment, decomposition tube, catalyst tube, gold amalgamator, amalgamator furnace, measuring cuvettes, mercury lamp, and detector. The following requirements are specified for all approved ~~instruments~~ instruments (Note 21).

NOTE 1—The approval of an instrument with respect to these functions is paramount to this test method, since such approval tacitly provides approval of both the materials and the procedures used with the system to provide these functions.

6.1.1 The instrument shall be capable of drying the sample once it is weighed and introduced.

6.1.2 The instrument shall have a decomposition tube which shall be operated at a temperature high enough to completely decompose the sample. The suggested operating temperature is ~~800°C~~ 800 °C.

6.1.3 The catalyst in the catalytic tube shall be capable of completing the oxidation of the sample and trapping halogens as well as nitrogen and sulfur oxides. The suggested operating temperature of the catalytic tube is ~~550°C~~ 550 °C.

6.1.4 The instrument shall contain a gold amalgamator fixed to an inert material and shall be capable of trapping all mercury.

6.1.5 The amalgamator shall contain a furnace capable of rapidly heating the amalgamator to release all trapped mercury.

6.1.6 The instrument shall have an absorption cell with measuring cuvettes through which the elemental mercury released from the gold amalgamator flows. The cell shall be heated to avoid any condensation of water or other decomposition products.

6.1.7 The light source for the atomic absorption process shall be a low pressure mercury lamp.

6.1.8 A narrow bandpass interference filter or monochromator, capable of isolating the 253.65 nm mercury line, shall be used.

6.1.9 The system may contain a computer for controlling the various operations of the apparatus, for recording data, and for reporting results.

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

6.3 *Sample Combustion Boats*, made of nickel and convenient size suitable for use in the instrument being used.

7. Sample

7.1 *Coal*—Prepare the analysis sample in accordance with Practice **D2013/D2013M** by pulverizing the material to pass a ~~250- μ m~~ 250 μ m (No. 60) sieve.

7.2 *Solid Combustion Residue*—Dry a representative portion of the solid residue to constant weight at ~~110°C~~ 110 °C to ~~115°C~~ 115 °C. Determine the moisture loss during this drying step if it is desirable to calculate results to an as-received basis. Crush the dried portion of the sample to pass a ~~No. 200 (75- μ m)~~ 75 μ m (No. 200) sieve. Use a mill that minimizes metal contamination. Use portions of the ~~200-mesh~~ passing material for analysis.

7.3 *Activated Carbon Sorbent Material*—Activated carbon sorbent material is used in tubes for the absorption of mercury from stack gases. The absorbed mercury is unevenly dispersed in the tubes. The activated carbon material in smaller absorption tubes (~~100–200 mg~~) (100 mg to 200 mg) may be analyzed directly without further preparation. The activated carbon and absorbed mercury in larger sorbent tubes must be homogenized before any sample subdivision. The procedure given in Appendix Annex A2 is recommended for the homogenization and subdivision of the activated carbon sorbent material.³

7.4 Analyze separate test portions for moisture content in accordance with Test Methods **D3173** or **D7582** so that calculation to other bases can be made.

³ This procedure was developed by the LECO Corporation in St. Joseph, MI. The procedure and results from an 11-laboratory interlaboratory study of four analytical methods for the analysis of mercury in Appendix K sorbent tubes can be found in the publication: Evaluation of Methods for Mercury Analysis of Appendix K Sorbent Tubes, EPRI, Palo Alto, CA, Tennessee Valley Authority (TVA), Chattanooga, TN, AEP, Columbus, OH, Consumer's Energy, Jackson, MI, First Energy, Cleveland, OH, LECO Corporation, St. Joseph, MI, Eon U.S., Lexington, KY, Southern Company, Birmingham, AL and TXU Power, Dallas, TX: 2007, 1014565, 110 pp.

8. Reagents

8.1 *Oxygen*—High purity oxygen, as specified by the instrument manufacturer, shall be used.

8.2 *Certified Reference Materials (CRMs)*—Use Certified Reference Material (CRM) coals with dry-basis mercury values for which confidence limits are issued by a recognized certifying agency such as the National Institute of Standards and Technology (NIST). It is recommended that the user verify the value with the certifying agency before using the CRM coal for quality control purposes.

8.3 All CRMs, reference coals, or calibrating agents must have precision values of less than or equal to method repeatability. Such ~~CRMS, CRMs~~, reference coals, or calibrating agents must be stable with respect to moisture and be pulverized to pass 100 % through a 250 μm (No. 60) USA Standard Sieve. CRMs, reference coals, or calibrating agents must be mixed thoroughly before each use.

9. Instrument Preparation

9.1 Assemble the instrumental system in accordance with the manufacturer’s instructions. Follow the instrument manufacturer’s recommended procedure to optimize the performance of the instrument.

9.2 *Adjustment of Response of Measurement System*—Weigh an appropriate test portion of certified reference material (CRM), calibrating agent, or reference coal. Analyze the test portion (see 9.1). Repeat this procedure. Adjust instrument response, as recommended by the manufacturer, until the absence of drift is indicated.

9.3 *Calibration*—Select coal CRMs or other calibrating agents and materials specified by the manufacturer that have certified mercury values in the range of samples to be analyzed. Three such CRMs or calibrating agents are recommended for each range of mercury values to be tested. When possible, two of the CRMs or calibrating agents shall bracket the range of mercury to be tested, with the third falling within the range.

9.3.1 All coal CRMs should be in accordance with 8.2 and shall be supplied by or have traceability to an internationally recognized certifying organization. ~~Warning—An indicated problem with linearity of the instrument during calibration can result from contamination of the CRM or calibrating agent as the container becomes depleted. It is therefore recommended that the CRM or calibrating agent be discarded when less than five grams remain in the container. An indicated problem with linearity of the instrument during calibration can result from contamination of the CRM or calibrating agent as the container becomes depleted. It is therefore recommended that the CRM or calibrating agent be discarded when less than five grams remain in the container.~~

9.3.2 *Calibration Procedure*—Analyze, as samples, portions of a CRM, reference coal, or calibrating agent chosen to represent the level of mercury in the samples to be tested. Use the “as-determined” mercury values for calibration. These values must have been calculated previously from the certified “dry basis” mercury values and residual moisture determined using either Test Methods [D3173](#) or [D7582](#). Continue analyzing until the results from five consecutive determinations fall within the repeatability interval of these test methods. Calibrate the instrument according to the manufacturer’s instructions using these values. Analyze, as samples, two CRM reference coals or calibrating agents that bracket the range of values to be tested. The results obtained for these samples must be within the stated precision limits of the CRM, reference coal, or calibrating agent, or the calibration procedure must be repeated. ~~Records for all calibrations must be in accordance with Guide [D4621](#).~~

9.3.3 *Periodic Calibration Verification and Recalibration*—In accordance with ~~Guide~~[Practice \[D4621\]\(#\)+\[D7448\]\(#\)](#), analyze a control sample on a periodic basis. Results obtained for the control sample must be within established limits, or all results obtained since the last successful control check must be rejected and the calibration procedure repeated.

10. Procedure

10.1 Analyze a test specimen of the analysis sample in accordance with the manufacturer’s instructions.

11. Calculation

11.1 Calculate the ~~concentration~~ mass fraction of mercury, on the appropriate sample basis, as follows:

$$A = \frac{(B \times C)}{D} \times 100 \quad (1)$$

$$A = \frac{(B \times C)}{D} \quad (1)$$

where:

A = mg/Kg of the analyte,

A = mass fraction of mercury, μg/g,

B = detector response for that analyte,

B = detector response for mercury, counts,

C = ~~unit mass per detector response established for the analyte during calibration, and~~

C = unit mass per detector response established for mercury during calibration, μg/counts, and