



Designation: ~~D7623~~—~~10~~ **D7623 – 10 (Reapproved 2015)**

## Standard Test Method for Total Mercury in Crude Oil Using Combustion-Gold Amalgamation and Cold Vapor Atomic Absorption Method<sup>1</sup>

This standard is issued under the fixed designation D7623; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This test method covers the procedures to determine the total mercury content in a sample of crude oil.
- 1.2 The test method may be applied to crude oil samples containing between 55 ng/mL to ~~400~~400 ng/mL of mercury. The results may be converted to mass basis, and reported as ng/g of mercury.
- 1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.4 **WARNING**—Mercury has been designated by many regulatory 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 and/or mercury containing products into your state or country may be prohibited by law.
- 1.5 *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:<sup>2</sup>

- [D1193 Specification for Reagent Water](#)
- [D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)
- [D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants](#)
- [D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)
- [D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)
- [D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants](#)
- [D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories](#)
- [D7482 Practice for Sampling, Storage, and Handling of Hydrocarbons for Mercury Analysis](#)
- [D7622 Test Method for Total Mercury in Crude Oil Using Combustion and Direct Cold Vapor Atomic Absorption Method with Zeeman Background Correction](#)

### 3. Terminology

- 3.1 For definitions of terms used in this standard, refer to Terminology [D4175](#).

### 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 (at about ~~700°C~~)700 °C) and chemically decomposed. The decomposition products are carried by flowing treated air to the catalytic section of the furnace (at about ~~850°C~~)850 °C), where oxidation is completed. The

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products—Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.03](#) on Elemental Analysis.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

decomposition products are carried to a gold amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining decomposition products other than mercury, the amalgamator is rapidly heated to about ~~600°C~~ 600 °C, releasing mercury vapor. Flowing oxygen carries the mercury vapor through absorbance cells positioned in the light path of single wavelength cold vapor atomic absorption spectrophotometer. Absorbance peak height or peak area, as a function of mercury concentration, is measured at ~~253.65 nm~~ 253.65 nm.

NOTE 1—Mercury and mercury salts can be volatilized at low temperatures. Precautions against inadvertent mercury loss should be taken when using this test method.

## 5. Significance and Use

5.1 The emission of mercury during crude oil refining is an environmental concern. The emission of mercury may also contaminate refined products and form amalgams with metals, such as aluminum.

5.2 When representative test portions are analyzed according to this procedure, the total mercury is representative of concentrations in the sample.

## 6. Apparatus

6.1 There are several configurations of the instrumental components that can be used satisfactorily for this test method.<sup>3</sup> Functionally, the instrument shall have the following components: sample heating furnace, decomposition furnace, gold amalgamator, amalgamator furnace, measuring cuvettes, mercury lamp, and detector. The following requirements are specified for all approved instruments.

NOTE 2—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 at least ~~700°C~~ 700 °C.

6.1.3 The catalyst 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 ~~850°C~~ 850 °C.

6.1.4 The instrument shall contain one or more 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 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~~ 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~~ 0.1 mg.

6.3 *Sample Combustion Boats*, porcelain, quartz, or other material as recommended and of convenient size suitable for use in the instrument being used.

6.4 *Crucibles*, porcelain, high-form, ~~40 mL~~ 40 mL capacity or suitable size for heating reagent in furnace.

6.5 *Micropipettes*, one or more units of variable volumes to cover a range of ~~10 µL~~ to ~~250 µL~~ 250 µL. Appropriately sized tips should also be available.

6.6 *Ultrasonic Homogenizer*—A bath type ultrasonic homogenizer is used to dissociate particulate mercury and thoroughly mix the sample.

6.7 *Electric Muffle Furnace*, capable of maintaining ~~750~~ 750 °C ± ~~25°C~~ 25 °C and sufficiently large to accommodate the sample boats and reagent containers.

6.8 *Glassware*, class A, volumetric flasks and pipettes of various capacities. All glassware must be thoroughly cleaned with freshly prepared 10 % nitric acid solution and rinsed with water. It is recommended that dedicated glassware be maintained to minimize cross contamination.

<sup>3</sup> The sole source of supply of the apparatus known to the Committee at this time is Nippon SP3D model available from Nippon Instrument Corp., 14-8, Akaoji-cho, Takatsuki-shi, Osaka 569-1146, Japan. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

## 7. Sample

7.1 Obtain the analysis sample of crude oil in accordance with Practice [D4057](#) or [D4177](#). Crude oil should be collected in a manner that ensures a representative sample from the bulk container is obtained.

7.2 To prevent loss of mercury during storage and handling of samples, follow Practice [D7482](#). Sample should not be collected in metal containers. Precleaned, glass volatile organic analysis (VOA) vials have been found to be suitable for this purpose.

7.3 Samples should be analyzed as quickly as possible after collection. Sample containers should be kept tightly capped and stored in a cool location free from direct sunlight.

## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification [D1193](#). Water must be checked for potential mercury contamination before use.

8.3 *Air or Oxygen*—Filtered, purified air or high purity oxygen, as specified by the instrument manufacturer, shall be used. The air must be mercury-free. Some instruments process incoming combustion air with filters and driers before it is used for the analysis.

8.4 *Certified Reference Materials (CRMs)*—Use Certified Reference Material (CRM) crude oils with mercury values for which confidence limits are issued by a recognized certifying agency such as the National Institute of Standards and Technology (NIST).

8.4.1 All CRMs, reference crude oils, or calibrating agents must have precision values of less than or equal to method repeatability. Such CRMs, reference crude oils, or calibrating agents must be stable and must be mixed thoroughly before each use.

8.5 *L-cysteine*, 99+ % purity. Reagents must be evaluated for mercury content by checking blank solutions for response on the instrument.

8.6 *Nitric Acid*, concentrated, Trace Metal Grade or better.

8.7 *Mercuric Chloride*, 99.99+ % purity. A commercially prepared mercury stock standard may be used.

8.8 *Phosphate Buffer*, pH 7.2. Some phosphate buffers use mercury compounds as preservative. The buffer must be mercury-free. Check with the instrument manufacturer for sources of suitable buffer.

8.9 *Combustion Reagents*:

8.9.1 *Additive 1 or B (Aluminum Oxide)*, ground, available from instrument manufacturer.

8.9.2 *Additive 2 or M*, 40/60 m % mixture of calcium hydroxide and sodium carbonate available from instrument manufacturer.

8.9.3 *Calcium Hydroxide*, 99.5 % minimum purity.

8.9.4 *Sodium Carbonate*, 99.99 % minimum purity.

8.10 *Glassine Weighing Paper*.

## 9. Preparation of Mercury Standards

9.1 *L-cysteine Solution, 0.001 m %*—Weigh approximately ~~0.010 g~~ 0.010 g of L-cysteine to the nearest milligram onto a glassine weighing paper. Quantitatively transfer the contents of the paper to a ~~1-L~~ 1 L volumetric flask. Add approximately ~~800 mL~~ 800 mL of water. Add ~~2 mL~~ 2 mL of nitric acid into the flask with a pipette. Swirl to dissolve the solids. Fill to the mark with water. The L-cysteine solution stabilizes the ionic mercury and is used to prepare working standards.

9.2 *Mercuric Chloride Stock Solution*—Weigh ~~0.0677 g~~ 0.0677 g of mercuric chloride onto weighing paper. Quantitatively transfer the material to a ~~500 mL~~ 500 mL volumetric flask using the L-cysteine solution prepared in accordance with [9.1](#). Fill to the mark with L-cysteine solution and mix. This results in a mercury concentration of ~~100 mg/kg~~ 100 mg/kg.

9.3 *Working Standards Set*—Prepare a set of mercury standards that are appropriate to the range settings on the instrument in use. An example of such a set follows.

9.3.1 With a ~~5 mL~~ 5 mL pipette, transfer an aliquot of the mercuric chloride stock solution to a ~~100 mL~~ 100 mL volumetric flask. Fill the flask to the mark with L-cysteine solution and mix. This makes a ~~55 mg/kg~~ 55 mg/kg standard.

9.3.2 With a ~~1 mL~~ 1 mL pipette, transfer an aliquot of the mercuric chloride stock solution to a ~~100 mL~~ 100 mL volumetric flask and fill up to the mark with L-cysteine solution. Mix. This makes a ~~11 mg/kg~~ 11 mg/kg standard.

<sup>4</sup> *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.