



# Standard Test Method for Determination of Gold in Activated Carbon by Fire Assay Gravimetry<sup>1</sup>

This standard is issued under the fixed designation E 1568; 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 determination of gold in activated carbon by fire assay collection and gravimetric measurement. It covers the range of 15 to 5000  $\mu\text{g/g}$  gold.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety problems, 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.* For specific hazards statements, see Section 9 and Notes 2-4, Note 6, and Note 7.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 2862 Test Method for Particle Size Distribution of Granular Activated Carbon<sup>2</sup>

D 2866 Test Method for Total Ash Content of Activated Carbon<sup>2</sup>

D 2867 Test Method for Moisture in Activated Carbon<sup>2</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>3</sup>

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>4</sup>

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>4</sup>

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals<sup>5</sup>

E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75 mm) Sieve and Finer for Metal-Bearing Ores and Related Materials<sup>4</sup>

E 300 Practice for Sampling Industrial Chemicals<sup>6</sup>

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory<sup>7</sup>

E 1601 Practice for Conducting an Interlaboratory Studies of Methods for Chemical Analysis of Metals<sup>4</sup>

### 2.2

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals<sup>8</sup>

## 3. Terminology

### 3.1 Definitions:

For definitions of terms used in this test method, refer to Terminology E 135.

## 4. Summary of Test Method

4.1 The weighed test sample is ignited and fused with fire assay flux in a clay crucible. The lead metal from the fusion is separated and the precious metals concentrated by oxidation and adsorption of the lead on a cupel, the silver is parted with nitric acid, and the gold is annealed and weighed on a microbalance.

## 5. Significance and Use

5.1 In the primary metallurgical processes used by the mineral processing industry for gold bearing ores, gold is extracted with alkaline cyanide solutions and adsorbed onto activated carbon for recovery of the metal. Metallurgical accounting, process control, and ore evaluation procedures for this type of mineral processing plant depend on accurate, precise, and prompt measurements of gold concentrations in the activated carbon.

5.2 This test method for gold in activated carbon is intended primarily as a referee method to test such materials for metal content. It is assumed that those who use these procedures will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>5</sup> Discontinued; see *Annual Book of ASTM Standards*, Vol 03.05.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>7</sup> *Annual Book of ASTM Standards*, Vol 03.06.

<sup>8</sup> Global Engineering Documents, 15 Inverness Way East, Englewood, Colorado 80112.

waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide E 882.

## 6. Interferences

6.1 Elements normally found in ore processing activated carbon do not interfere. When present, platinum group metals may be reported as gold in gravimetric fire assay determinations and must be less than 0.1 mg in the final gold bead.

## 7. Apparatus

7.1 *Analytical Balance*, capable of weighing to 0.1 g.

7.2 *Assay Mold*, 100-mL capacity.

7.3 *Cupel*, magnesite, 30-g lead capacity.

7.4 *Drying Oven*, having forced air circulation, with temperature control between 145 and 155°C.

7.5 *Fire Clay Crucible*, 30-g sample capacity.

7.6 *Hot Plate*, having variable temperature control, used with ventilation control for acid fumes.

7.7 *Jeweler's Rolls*, capable of flattening doré beads.

7.8 *Muffle Furnace*, having air circulation with draft controls, capable of temperatures to 1100°C, accurate to  $\pm 10^\circ\text{C}$ , used with ventilation controls for lead fumes.

7.9 *Semi-Microbalance*, capable of weighing to 0.01 mg.

7.10 *Roasting Dish*, 15-g sample capacity.

## 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 shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>9</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Ammonia Wash Solution*,  $\text{NH}_4\text{OH}$  (1 + 17)—Add 100 mL ammonium hydroxide to 1700 mL of  $\text{H}_2\text{O}$ .

8.3 *Borax*— $\text{Na}_2\text{B}_4\text{O}_7$ —Sodium borate powder, with gold content less than 0.001  $\mu\text{g/g}$ .

8.4 *Fire Assay Flux Mixture*—Mix 575 g of litharge (PbO) with 275 g of soda ash ( $\text{Na}_2\text{CO}_3$ ), 75 g of borax ( $\text{Na}_2\text{B}_4\text{O}_7$ ), 75 g of silica ( $\text{SiO}_2$ ), and 30 g of baking flour.

8.5 *Lead Foil*—99.9 % minimum, with gold content less than 0.001  $\mu\text{g/g}$ .

8.6 *Litharge*, PbO—Lead oxide powder, with gold content less than 0.001  $\mu\text{g/g}$ .

8.7 *Silica*,  $\text{SiO}_2$ —Silicon dioxide powder, with gold content less than 0.001  $\mu\text{g/g}$ .

8.8 *Silver Foil*—99.9 % minimum, with gold content less than 0.001  $\mu\text{g/g}$ .

8.9 *Soda Ash*,  $\text{Na}_2\text{CO}_3$ —Sodium carbonate powder, with gold content less than 0.001  $\mu\text{g/g}$ .

8.10 *Strong Nitric Acid Parting Solution*,  $\text{HNO}_3(1 + 2)$ —Add 330 mL nitric acid to 660 mL of  $\text{H}_2\text{O}$ .

8.11 *Weak Nitric Acid Parting Solution*,  $\text{HNO}_3(1 + 4)$ —Add 200 mL nitric acid to 800 mL  $\text{H}_2\text{O}$ .

## 9. Hazards

9.1 Refer to Practices E 50 for precautions to be observed in this test method.

9.2 Use care when handling hot crucibles and operating furnaces in order to avoid personal injury by either burn or electrical shock.

9.3 Lead and litharge (PbO) are toxic materials and are volatile at relatively low temperatures. Use accepted safety procedures to avoid inhalation, ingestion, or skin contact.

9.4 Refer to Notes 2-4, Note 6, and Note 7.

## 10. Sampling

10.1 Collect the sample in accordance with Practice E 300. Samples must be free of any extraneous materials such as sand, rocks, and wood.

10.2 *Sample Preparation*—Dry the sample, in accordance with the Oven-Drying Method of Test Method D 2867, to constant weight at 150°C. If the analysis of a particular particle size range is desired, separate fractions in accordance with Test Method D 2862.

10.3 *Test Sample*—Pulverize the gross sample so that at least 90 % passes a 150- $\mu\text{m}$  (100 mesh, Tyler) sieve, in accordance with Test Method E 276. Mix the gross sample. Weigh duplicate test samples of  $15.0 \pm 1.0$  g, and record the test sample weights.

## 11. Procedure

### 11.1 Ignition:

11.1.1 Spread 5 g of silica ( $\text{SiO}_2$ ) in the bottom of a preheated roasting dish.

11.1.2 Transfer the test sample to the top of the silica bed in the roasting dish.

11.1.3 Ash at 650°C in a muffle furnace in accordance with Test Method D 2866. Cool.

### 11.2 Fusion:

11.2.1 Transfer calcine and silica to a clay crucible containing 50 g of fire assay flux.

11.2.2 Cover with another 50 g portion of fire assay flux. Mix. Cover the mixture of 5 g of litharge (PbO).

11.2.3 Place the crucible in a muffle furnace at 1050°C for 1 h.

NOTE 1—Draft may be required for the first 15 min of fusion to prevent boil-over.

NOTE 2—**Warning:** Fire assay products produce toxic lead oxide fumes when heated. Use protective clothing, and work only in well-ventilated areas.

11.2.4 Remove the crucible from the muffle furnace and pour molten liquid into the assay mold carefully, making certain that all lead droplets are transferred to the mold.

NOTE 3—**Warning:** Rapidly cooling slag may eject flying glass. Use eye protection and cover the slag immediately after pouring.

<sup>9</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

11.2.5 After cooling, separate the lead metal from the slag. Form the lead metal into a cube using a hammer and metal plate.

NOTE 4—**Warning:** Lead slag is toxic; use protective equipment and dispose of waste properly.

### 11.3 Cupellation:

11.3.1 Preheat a cupel at 1000°C for 10 min in a muffle furnace. Reduce the muffle furnace temperature controller to 940°C.

11.3.2 Place the lead buttons on the hot cupels and melt, without draft, as the temperature drops to 940°C (approximately 5 min).

11.3.3 Open the draft when the lead is melted, and continue heating with the draft open until the lead has been removed, leaving the doré bead in the cupel.

NOTE 5—A row of empty cupels should be placed in the front of the muffle furnace for temperature control of the draft.

11.3.4 Vent the muffle furnace and remove the cupel from the furnace. Cool.

NOTE 6—**Warning:** Hot cupels emit lead fumes. Use proper ventilation.

### 11.4 Inquartation:

11.4.1 Carefully remove the doré bead from the cupel and weigh on the semimicrobalance to the nearest 0.01 mg. Record the doré weight.

NOTE 7—**Warning:** Used cupels are toxic; use protective equipment and dispose of waste properly.

11.4.2 Weigh the proper amount of silver foil for the doré weight, according to Table 1, to the nearest 1 mg.

11.4.3 Wrap the doré bead and silver foil in 10 g of lead foil.

11.4.4 Repeat the cupellation procedure (11.3) with the foil packet.

### 11.5 Parting:

11.5.1 Flatten the doré bead and place it in a 50-mL porcelain crucible.

NOTE 8—The use of jeweler’s rolls is recommended for flattening large doré beads.

11.5.2 Anneal the bead in the crucible at 650°C in a muffler furnace or over a bunsen burner.

11.5.3 Place the crucible on a hot plate at 95°C and add 25 mL of HNO<sub>3</sub>(1 + 4), preheated to 60 ± 10°C. Heat for 30 min.

11.5.4 Carefully decant spent parting solution and discard.

**TABLE 1 Silver Additions for Inquartation**

Doré Weight Range, mg	Silver Foil Weight Factor Applied to the Doré Weight
0.00 to 0.1	30
0.1 to 0.2	10
0.2 to 1.0	6
0.1 to 10.0	4
>10.0	2.25

11.5.5 Repeat the parting steps given in 11.5.3 and 11.5.4, using HNO<sub>3</sub>(1 + 2). Cool.

11.5.6 Rinse with 25 mL of NH<sub>4</sub>OH (1 + 17) and five times with 25 mL H<sub>2</sub>O. Dry on a hot plate.

11.5.7 Repeat the annealing step given in 11.5.2. Cool.

11.6 *Gravimetry*—Weigh the gold bead to the nearest 0.01 mg. Record the weight. If silver is detected visually in the gold bead, repeat the steps given in 11.4.3-11.5.7, reweigh, and record the weight.

**TABLE 2 Interlaboratory Precision**

Material	Mean Au, µg/g	R <sub>1</sub> ; E173 Au, µg/g	R <sub>2</sub> ; E173 Au, µg/g
Stripped carbon	108.2	5.4	7.6
Fine carbon	347.8	17.6	17.6
Loaded carbon	3983	99.5	243

## 12. Calculation

12.1 Calculate the µg/g gold in the sample as follows:

$$\text{Gold, } \mu\text{g/g} = 1000 A/B \quad (1)$$

where:

A = gold measured, mg, and

B = test sample weight, g.

12.2 Average the results from duplicate determinations, and round them to the nearest 1 µg/g in accordance with Practice E 29.

## 13. Precision and Bias

13.1 *Precision*—An interlaboratory study was conducted in accordance with Practice E 173 to establish the within-laboratory and between-laboratory precision. Three materials were tested by seven laboratories. The results are summarized in Table 2.<sup>10</sup>

13.2 *Bias*—No information concerning the bias of this test method is available. However, the bias of the test method in a single laboratory, or between laboratories, may be judged by the deviation of the mean value obtained using the test method from the accepted value for a suitable reference material.

NOTE 9—The within-laboratory precision measures the maximum difference expected in testing the material in a single laboratory at 95 % confidence. The between-laboratory precision measures the maximum difference expected in testing the material in more than one laboratory at 95 % confidence.

NOTE 10—This test method has been evaluated in accordance with Practice E 173. The Reproducibility R<sub>2</sub> of Practice E 173 corresponds to the Reproducibility index R of Practice E 1601. The Repeatability R<sub>1</sub> of Practice E 173 corresponds to the Repeatability Index r of Practice E 1601.

## 14. Keywords

14.1 activated carbon; gold content

<sup>10</sup> Support data have been filed at ASTM International Headquarters. Request RR:E01-1007.

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