Designation: E1568 − 13

Standard Test Method for
Determination of Gold in Activated Carbon by Fire Assay Gravimetry[1]

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 µg/g to 5000 µg/g gold.

1.2 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this 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.* For specific hazards statements, see Section 9 and 11.2.3-11.2.5, 11.3.4, and 11.3.4.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2862 Test Method for Particle Size Distribution of Granular Activated Carbon

D2866 Test Method for Total Ash Content of Activated Carbon

D2867 Test Methods for Moisture in Activated Carbon

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials

E300 Practice for Sampling Industrial Chemicals

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

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

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[1] 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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[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide E882.

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 °C 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 ± 10 °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.[4] 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,* NH_4OH (1 + 17)—Add 100 mL NH_4OH to 1700 mL of water.

8.3 *Borax*—$Na_2B_4O_7$—Sodium borate powder, with gold content less than 0.001 µg/g.

8.4 *Fire Assay Flux Mixture*—Mix 575 g of litharge (PbO) with 275 g of soda ash (Na_2CO_3), 75 g of borax ($Na_2B_4O_7$), 75 g of silica (SiO_2), and 30 g of baking flour.

8.5 *Lead Foil*—99.9 % minimum, with gold content less than 0.001 µg/g.

8.6 *PbO*—Lead oxide powder, with gold content less than 0.001 µg/g.

8.7 *SiO_2*—Silicon dioxide powder, with gold content less than 0.001 µg/g.

8.8 *Silver Foil*—99.9 % minimum, with gold content less than 0.001 µg/g.

8.9 *Na_2CO_3*—Sodium carbonate powder, with gold content less than 0.001 µg/g.

8.10 *Strong HNO_3 (1 + 2) Parting Solution*—Add 330 mL HNO_3 to 660 mL of water.

8.11 *Weak HNO_3 (1 + 4) Parting Solution*—Add 200 mL HNO_3 to 800 mL water.

9. Hazards

9.1 Refer to Practices E50 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 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 **WARNINGS** in 11.2.3-11.2.5, 11.3.4, and 11.3.4.

10. Sampling

10.1 Collect the sample in accordance with Practice E300. 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 Methods D2867, to constant weight at 150 °C. If the analysis of a particular particle size range is desired, separate fractions in accordance with Test Method D2862.

10.3 *Test Sample*—Pulverize the gross sample so that at least 90 % passes a 150-µm (No. 100 or 100 mesh, Tyler) sieve, in accordance with Test Method E276. Mix the gross sample. Weigh duplicate test samples of 15.0 g ± 1.0 g, and record the test sample weights.

11. Procedure

11.1 *Ignition:*

11.1.1 Spread 5 g of 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 D2866. 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 PbO.

11.2.3 Place the crucible in a muffle furnace at 1050 °C for 1 h. (**WARNING**—Fire assay products produce toxic lead oxide fumes when heated. Use protective clothing, and work only in well-ventilated areas.)

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[4] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

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

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. (**WARNING**—Rapidly cooling slag may eject flying glass. Use eye protection and cover the slag immediately after pouring.)

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. (**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 2—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. (**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. (**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 3—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_3 (1 + 4), preheated to 60 °C ± 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

TABLE 2 Interlaboratory Precision

Material	Mean Au, µg/g	R_1; Practice E173 Au, µg/g	R_2; Practice 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

11.5.5 Repeat the parting steps given in 11.5.3 and 11.5.4, using HNO_3 (1 + 2). Cool.

11.5.6 Rinse with 25 mL of NH_4OH (1 + 17) and five times with 25 mL water. 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.

12. Calculation

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

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

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 E29.

13. Precision and Bias[5]

13.1 *Precision*—An interlaboratory study was conducted in accordance with Practice E173 to establish the within-laboratory and between-laboratory precision. Three materials were tested by seven laboratories. The results are summarized in Table 2.

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 4—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 5—This test method has been evaluated in accordance with Practice E173. The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

14. Keywords

14.1 activated carbon; fire assay gravimetry; gold content

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[5] Support data have been filed at ASTM International Headquarters. Request RR:E01-1007.

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