



Designation: D2797/D2797M – 21

Standard Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light¹

This standard is issued under the fixed designation D2797/D2797M; 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 This practice covers laboratory procedures for the preparation of granular samples of bituminous coal and anthracite samples for examination with a microscope using reflected light illumination. It does not apply to the preparation of oriented blocks of coal.

1.2 *Units*—The values stated in either SI units or non-SI units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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

- D2013 Practice for Preparing Coal Samples for Analysis
- D2234/D2234M Practice for Collection of a Gross Sample of Coal
- D2798 Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal
- D2799 Test Method for Microscopical Determination of the

¹ This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.28 on Petrographic Analysis of Coal and Coke.

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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.

Maceral Composition of Coal

D4596 Practice for Collection of Channel Samples of Coal in a Mine

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Practice

3.1 A representative sample is crushed to a specified particle size, air-dried, mixed with a binder, and formed into a briquet. The briquet is then polished to a flat, scratch-free surface for examination by vertical illumination.

4. Significance and Use

4.1 Briquets of granular bituminous coals and anthracite prepared in accordance with the laboratory procedures of this practice will have flat, scratch-free surfaces suitable for examination with a microscope using reflected light illumination. The polished surfaces of briquettes prepared by this practice will contain particles representative of the original gross sample.

4.2 Samples prepared by this practice are used for microscopical determination of the reflectance of the organic components in a polished specimen of coal (Test Method D2798) and for microscopical determination of the volume percent of physical components of coal (Test Method D2799).

5. Apparatus

5.1 *Grinder, Pulverizer, or Mill*, for final crushing of the sample to pass a 850 μm [No. 20] U.S. Standard Sieve. Mortar and pestle or other equipment suitable for reducing the particle size of a 250 g sample that already passes a 4.75 mm [No. 4] U.S. Standard Sieve until all of it passes a 850 μm [No. 20] U.S. Standard Sieve (see 6.5).

5.2 *Coarse Riffle Sampler*—A riffle with at least twelve divisions of not less than 12.7 mm [$\frac{1}{2}$ in.] and not greater than 19.1 mm [$\frac{3}{4}$ in.].

5.3 *Fine Riffle Sampler*—A riffle with at least twelve divisions of not less than 3.2 mm [$\frac{1}{8}$ in.] and not greater than 6.4 mm [$\frac{1}{4}$ in.].

5.4 *Sieves*—A 4.75 mm [No. 4] and a 850 μm [No. 20] U.S. Standard Sieve (Specification E11).

5.5 *Molds*—Containers to hold the coal binder mixture while the binder hardens.

5.5.1 The mold shall be capable of withstanding pressures up to 70 MPa [10 000 psi], and shall be made of separable parts so that the briquet can be ejected after it has hardened.

5.5.2 The mold shall be large enough to provide a plane area of 4 cm² or more on one side of the briquet. (Designs of suitable 25 mm [1 in.] and 32 mm [1¼ in.] inside-diameter molds are shown in Fig. 1.)

5.6 *Mounting Press*—A manual or automated press that is capable of producing a pressure up to 35 MPa [5000 psi] on the briquet, and has an attachment to eject the briquet after solidification of the binder. A heat-setting automated press shall be used with an appropriate thermoset binder.

5.7 *Grinding and Polishing Equipment*—One or several laps on which the coal briquet can be ground and polished to a flat, scratch-free surface. Laps may be made of aluminum, iron, brass, bronze, lead, glass, wax, or wood. Equipment that has 203 mm [8 in.] diameter disk laps, gear-driven at 160 r/min to 170 r/min, and that has an automatic sample holder attachment is suitable. For notes on grinding and polishing practices, see Appendix X1.

5.8 *Sample Cleaner*—Some equipment is essential for cleaning coal briquets between the different grinding or polishing stages. This may be a simple stream of water or an air jet, but an ultrasonic cleaner is recommended.

5.9 *Containers*—If samples are to be transported or stored before briquets are prepared, glass jars, bottles, or metal cans having tight-fitting closures shall be used. Metal cans shall have plastic liners.

6. Materials

6.1 *Binder*—Any binding material fulfilling the following requirements may be used for preparing the coal briquet:

6.1.1 The binder shall hold all coal particles securely during grinding, polishing, and observing the sample.

6.1.2 The binder and the hardening process used shall not react with the coal or the atmosphere or expose the coal to thermal oxidation.

6.1.3 Under the microscope, the binder shall contrast markedly with constituents being measured when immersed in oil having an index of refraction of about 1.518.

6.1.4 The binder shall be such that a substantially flat and scratch-free surface can be obtained as a result of the grinding and polishing procedure.

6.1.4.1 Relief, or difference in level, particularly between the coal and the binder, is undesirable for reflectance determination. Although relief depends to a great extent on the polishing technique, use of a binder with a hardness comparable to that of the coal is very helpful.

6.1.4.2 Various types of epoxy resins and heat-setting powders are suitable as a binder.

6.2 *Release Agent*—Any preparation that does not damage the molds or adversely affect the coal or mounting medium may be used to coat the inside of the mold and facilitate ejection of the briquet.

6.3 *Grinding Abrasives*—Water-resistant, adhesive-backed silicon carbide papers of grit Nos. 240, 320, 400, and 600. Two or three of these are used according to a plan such as one of those listed in Table 1.

6.4 *Polishing Abrasives*—Aluminum oxide powders of 3 μm, 1 μm, 0.3 μm, and 0.05 μm sizes. Two or three of these are used according to a plan such as one of those listed in Table 1.

6.5 *Lap Coverings*—Nap-free cloths of cotton and silk, and chemotextile material backed with water-resistant adhesive.

6.6 *Detergent*—Any nonoxidizing detergent may be used for cleaning briquets after each grinding or polishing stage.

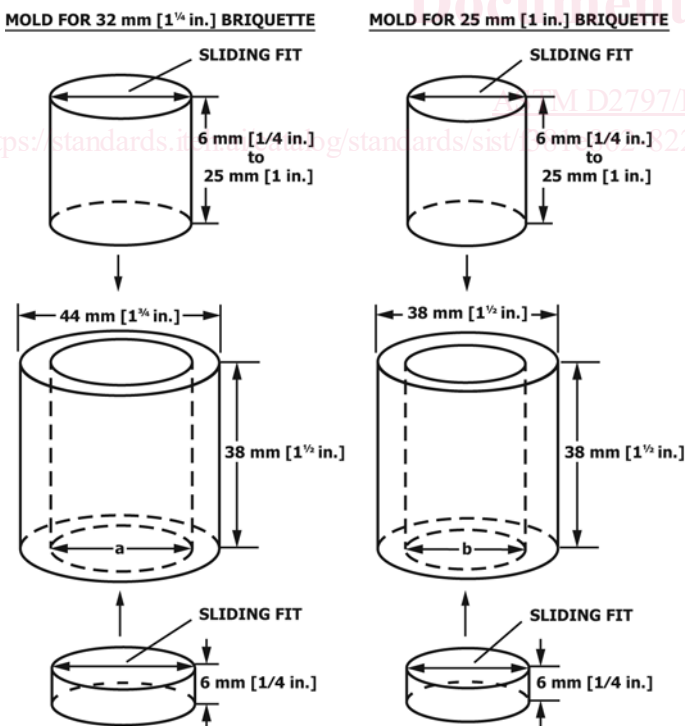
7. Sampling

7.1 Obtain samples of coal in accordance with Practice D2234/D2234M and Practice D4596, when appropriate.

7.2 If the coal sample has not previously been air dried, bring the sample to room conditions by exposure to the laboratory atmosphere.

7.3 Crush and divide the gross sample in accordance with Practice D2013 to obtain a representative 2.36 mm [No.8] U.S. Standard Sieve subsample (Note 1). Divide to a minimum mass of 500 g (for cleaned coal) or 1000 g (for unknown or all other coals.)

NOTE 1—The terms *gross sample* and *subsample* are defined, and the quality and amount of the subsample are specified, in Practice D2013.



NOTE 1—Material: cold rolled or stainless steel. Dimensions *a* and *b* (inside diameters) are nominally 32 mm [1¼ in.] and 25 mm [1 in.]. If an automatic polishing attachment is to be used, these dimensions should be specified to yield a briquet fitting snugly in the briquet holder.

FIG. 1 Molds Suitable for Briquetting Coal Samples

TABLE 1 Suggested Abrasive Sequences for Polishing of Briquets

Step	Grinding with Silicon Carbide Paper			Polishing with Alumina on Fabric Support		
	Coarse	Intermediate	Fine	Fine	Fine	Fine
1	53.5 μm [240 grit]	23.6 μm [400 grit]	16 μm [600 grit]	3 μm [118.11 * 10 ⁻⁶ in.] on non-woven napless textile	0.05 μm [1.968 x 10 ⁻⁶ in.] on one or two layers of silk cloth over chemotextile	-
2	23.6 μm [400 grit]	16 μm [600 grit]	-	3 μm [118.11 * 10 ⁻⁶ in.] on chemotextile	0.05 μm [1.968 x 10 ⁻⁶ in.] on silk cloth alone or over chemotextile	-
3	53.5 μm [240 grit]	23.6 μm [400 grit]	-	3 μm [118.11 * 10 ⁻⁶ in.] on chemotextile	0.05 μm [1.968 x 10 ⁻⁶ in.] on cotton cloth over chemotextile	-
4	23.6 μm [400 grit]	16 μm [600 grit]	-	1 μm [39.37 * 10 ⁻⁶ in.] on two layers of cotton cloth	0.3 μm [11.8 x 10 ⁻⁶ in.] on three layers of cotton cloth	0.05 μm [1.968 x 10 ⁻⁶ in.] on silk cloth over two layers of cotton cloth

7.4 Stage crush or grind the 2.36 mm [No. 8] U.S. Standard Sieve subsample to pass through a 850 μm [No. 20] U.S. Standard Sieve in a manner that minimizes the production of excessive fines. This is accomplished by successive sieving with intermittent grinding of the oversize coal for short periods until all coal passes the specified size. A mortar and pestle, adjustable plate mill, or other apparatus and procedures are acceptable if production of fines is minimized.

7.5 Riffle the 850 μm [No. 20] U.S. Standard Sieve coal to 250 g and store in a container that meets the requirements in 5.9. No additional crushing or grinding is required.

7.6 Pass the 850 μm [No. 20] U.S. Standard Sieve coal through the fine riffle sampler (5.3) to obtain the amount of coal needed for one or two briquets. Approximately 8 g to 16 g of coal is adequate for one 25 mm [1 in.] diameter briquet.

7.7 Place the coal in a container (5.9) or proceed with preparing the coal briquet.

8. Preparation of Coal Briquet

8.1 Prepare a coal briquet using a binder that meets the requirements of 6.1 in such a manner that, when polished, at least 60 % of the cross-sectional area will be coal.

8.1.1 Add activator or hardener, in the amount recommended by the supplier, to about 4 g of the epoxy resin for each 10 g of coal. Mix thoroughly with a spatula or disposable wooden stirring stick. Place the riffled coal sample in a suitable small container. Thoroughly stir a few drops of resin into the coal sample. Continue to add resin, a few drops at a time, and stir until all coal particles are wetted and the coal-resin mixture coheres when pressed to the side of the container with the spatula. For thermoset briquettes, mix coal and binder together in a small container or disposable paper cup in appropriate proportions so that at least 60 % of the cross sectional base of the briquette will be coal.

8.1.2 Coat the internal surfaces of a mold or interior of the automated press mold and fittings (including plungers) with a release agent and insert the lower plunger. Fill the mold with the coal-resin mixture. For manual presses, insert the upper plunger, place the mold in a hydraulic press, and apply a pressure of 28 MPa to 35 MPa [4000 psi to 5000 psi] on the briquet for 3 s to 5 s. Release and reapply the pressure; repeat this cycle about 5 times. The repeated application and release of pressure removes air bubbles introduced when the coal is mixed with the resin.

8.1.3 For automated presses, program pressures and temperatures appropriate to the size and number of briquettes being prepared in the mold and type of thermoset binder used.

8.1.4 For manual presses, allow the briquet in the mold to harden adequately for polishing. Overnight at room temperature is acceptable. Heating the briquet at elevated temperatures in an oven is acceptable if the procedure meets requirements in 6.1.2. Briquettes made with thermoset powders require elevated temperatures as specified by the manufacturer for the mounting medium to set.

8.1.5 For manual presses, eject the coal briquet from the mold using the special attachment on the press and label the briquet. For automated presses, eject coal briquette when it has cooled.

9. Preparation of Briquet Surface

9.1 Grind and polish one of the base surfaces of the briquet on a lap to obtain a surface suitable for microscopical examination. This method describes the use of mechanical polishing methods only; the use of Ion Milling has been shown to affect vitrinite reflectance and is not allowed. Grinding and polishing may be done either by hand manipulation or with an automatic attachment. Use a series of abrasives of decreasing particle size, such as those described in Table 1. The surface so obtained shall meet the following requirements:

9.1.1 Enough material shall be removed from the briquet to produce a flat surface over the entire area.

9.1.2 The surface shall be free of pits caused by loss of grains of coal constituents or mineral matter.

9.1.3 The surface shall be substantially free of scratches when examined at a magnification of 600 \times or more under oil immersion.

9.1.4 The surface shall be substantially free of relief.

9.1.5 The coal macerals shall be free of charring and smearing.

9.1.6 The surface shall be free of grinding and polishing compounds.

9.2 After each grinding or polishing step, clean the briquet or briquets to remove all abrasive and dislodged mineral particles. Use of an ultrasonic cleaner filled with water and detergent is recommended.

10. Moisture Control

10.1 An experimental test program has determined that polished coal briquet surfaces for most coals in the vitrinite