



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 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.3 *This standard does not purport to address the safety concerns 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:*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 Maceral Composition of Coal
- D4596 Practice for Collection of Channel Samples of Coal in a Mine

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.

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) 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) sieve until all of it passes a 850- μm (No. 20) 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).

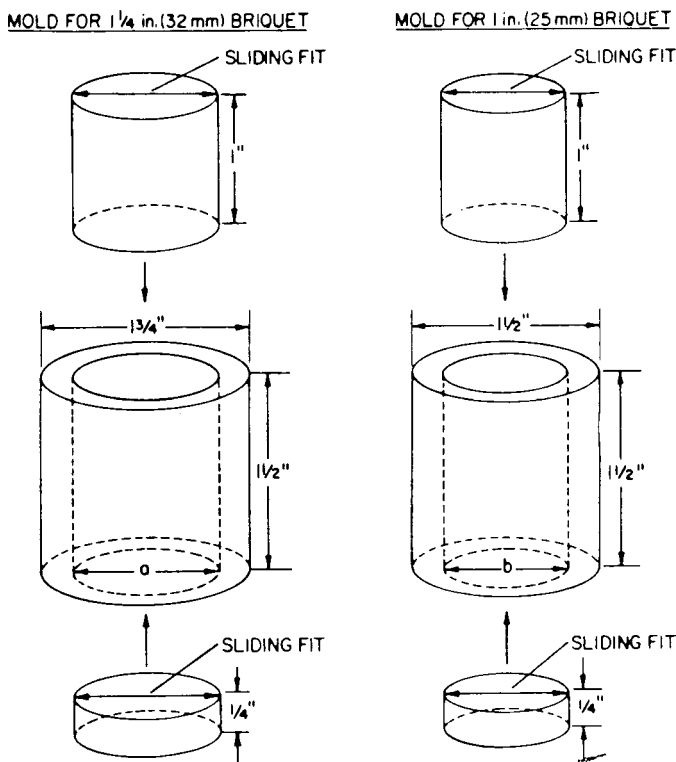
5.6 *Hydraulic Press*—a press that is capable of producing a pressure up to 35 MPa [5000 psi] on the briquet, and has an attachment to eject the briquet after solidification of the binder.

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. For notes on grinding and polishing practices, see Appendix X1.

NOTE 1—Equipment that has 203-mm [8-in.] diameter disk laps, gear-driven at 160 to 170 r/min, and that has an automatic sample holder attachment is recommended.

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.



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

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.

NOTE 2—Relief, or difference in level, particularly between the coal and the binder, is undesirable for reflectance determination. Although relief depends a great extent on the polishing technique, use of a binder with a hardness comparable to that of the coal is very helpful.

NOTE 3—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-, 1-, 0.3-, 0.1-, and 0.05- μ m 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) sieve size subsample (Note 4). Divide to a minimum weight of 500g (for cleaned coal) or 1000g (for unknown or all other coals.)

NOTE 4—The terms *gross sample* and *subsample* are defined, and the quality and amount of the subsample are specified in Practice D2013.

7.4 Stage crush or grind the 2.36 mm (No. 8) sieve size subsample to pass through a 850 μ m (No. 20) 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.

TABLE 1 Suggested Abrasive Sequences for Grinding and Polishing of Briquets

Plan No.	Grinding With Silicon Carbide Paper			Polishing with Alumina on Fabric Support		
	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3
1	240 grit	400 grit	600 grit	3 μm on chemotextile	0.05 μm on one or two layers of silk cloth over chemotextile	...
2	400 grit	600 grit	...	0.3 μm on chemotextile	0.05 μm on silk cloth alone or on silk cloth over cotton cloth	...
3	240 grit	400 grit	...	0.3 μm on chemotextile	0.05 μm on cotton cloth over chemotextile	...
4	320 grit	600 grit	...	3 μm on chemotextile	0.1 μm on silk cloth over chemotextile	...
5	400 grit	600 grit	...	1 μm on two layers of cotton cloth	0.3 μm on three layers of cotton cloth	0.05 μm on silk cloth over two layers of cotton cloth

7.5 Riffle the 850 μm (No. 20) sieve size 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) sieve size coal through the fine riffle sampler (5.3) to obtain the amount of coal needed for one or two briquets. Approximately 8 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.

8.1.2 Coat the internal surfaces of a mold (including plungers) with a release agent and insert the lower plunger. Fill the mold with the coal-resin mixture. Insert the upper plunger, place the mold in a hydraulic press, and apply a pressure of 28 to 35 MPa [4000 to 5000 psi] on the briquet for 3 to 5 s. Release and reapply the pressure; repeat this cycle about 15 times.

NOTE 5—The repeated application and release of pressure removes air bubbles introduced when the coal is mixed with the resin.

8.1.3 Allow the briquet in the mold to harden adequately for polishing. Overnight at room temperature acceptable. Heating the briquet at elevated temperatures in an oven is acceptable if the procedure meets requirements in 6.1.2.

8.1.4 Eject the coal briquet from the mold using the special attachment on the press and label the briquet.

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 exami-

nation. Grinding and polishing may be done either by hand manipulation or with an automatic attachment. Use a series of abrasives of decreasing particle size according to a plan such as one of 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 reflectance range of 0.6 % to 1.70 % (mean maximum reflectance) do not require desiccation prior to measuring reflectance³

10.2 If the vitrinite reflectance is outside the range specified in 10.1 or it has been established that a particular coal is affected by water absorption on the polished coal surface causing lower reflectance, then desiccate the briquet for 15 h before measuring reflectance.

NOTE 6—During the wet polishing process, some coals absorb moisture that can affect the amount of light reflected from the coal. In general, the greater the amount of water absorbed the less the reflectance. The reflectance of high-volatile C bituminous coal, some high-volatile B bituminous coals, and anthracite can be affected to varying degrees by absorption of moisture.

³ DeVanney, N., "Impact of Sample Desiccation on the Mean Maximum Vitrinite Reflectance for Various Ranks of Coal", *The Society for Organic Petrology Newsletter*, Vol 18, No. 3, September 2001, pp. 15–20.

APPENDIX

(Nonmandatory Information)

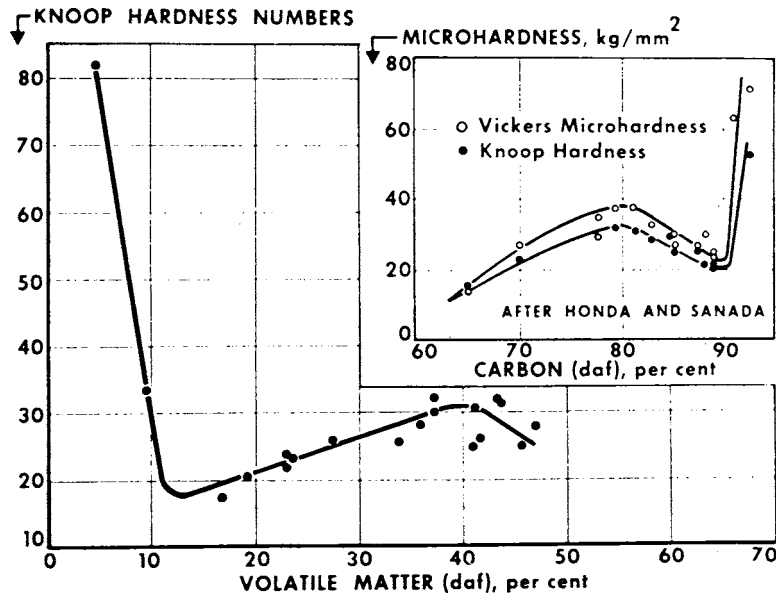
X1. NOTES ON GRINDING AND POLISHING PRACTICES

X1.1 A stream of water is recommended to carry away the cuttings and cool the sample when grinding with the silicon carbide papers. The objective of the first (coarsest) grinding step is to obtain a coplanar surface on all briquets and penetrate below the surface layer of particles. In subsequent grinding steps, the period should be long enough to remove scratches of the preceding step. A grinding time of 15 to 30 s is usually, but not always, sufficient. (Use of a rotating lap with automatic attachment is assumed here.)

X1.2 Room air cleanliness is important in polishing. Air pollutants, if hard (for example, quartz), can scratch the surface or, if soft enough (for example, soot), can smear it.

X1.3 Polishing abrasives are usually applied as a slurry in distilled water, diluted so that, on standing, the abrasive settles

to a level constituting 5 to 10 % of the column of mixture. Polishing cloths must be free of all knots, snags, holes, or other imperfections and should be stretched tightly over the wheel. The amount of load (pressure) applied in polishing is very important. Variation of hardness of vitrinite with coal rank can be used as a guide for choice of load (see Fig. X1.1). A load of about 14 kg [30 lbs] on six 25-mm [1-in.] briquets (with use of a rotating lap with automatic attachment) is commonly used on high-volatile bituminous coals. Considerably lower pressure should be used on low-volatile bituminous coal. Use of multilayers of cloth or of cloth with high nap tends to produce relief in the polished surface of the sample, although this can be reduced to some extent by decreasing the load and shortening the polishing period.



NOTE 1—From Shapiro, N., and Gray, R., "Petrographic Classification Applicable to Coals of All Ranks," *Proceedings of the Illinois Mining Institute, PIMIA*, Vol 68, 1960, pp. 83-97.

FIG. X1.1 Relationship Between Hardness of Vitrinite and Rank in Coals



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