



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

This standard is issued under the fixed designation D 2797; 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 SI units shall be considered as standard; and inch-pound units shall be considered as approximate equivalents.

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:

- D 2013 Method of Preparing Coal Samples for Analysis²
- D 2234 Test Methods for Collection of a Gross Sample of Coal²
- D 2798 Test Method for Microscopical Determination of the Reflectance of Vitrinite in a Polished Specimen of Coal²
- D 2799 Test Method for Microscopical Determination of Volume Percent of Physical Components of Coal²
- D 4596 Practice for Collection of Channel Samples of Coal in the Mine²
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

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.

¹ This practice is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.28 on Petrographic Analysis of Coal and Coke.

Current edition approved Aug. 30, 1985. Published October 1985. Originally published as D 2797 – 69 T. Last previous edition D 2797 – 85.

² *Annual Book of ASTM Standards*, Vol 05.06.

³ *Annual Book of ASTM Standards*, Vol 14.02.

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 D 2798) and for microscopical determination of the volume percent of physical components of coal (Test Method D 2799).

5. Apparatus

5.1 *Grinder, Pulverizer, or Mill*, for final crushing of the sample to pass a 850- μ m (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- μ m (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*⁴—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- μ m (No. 20) U.S. Standard Sieve (Specification E 11).

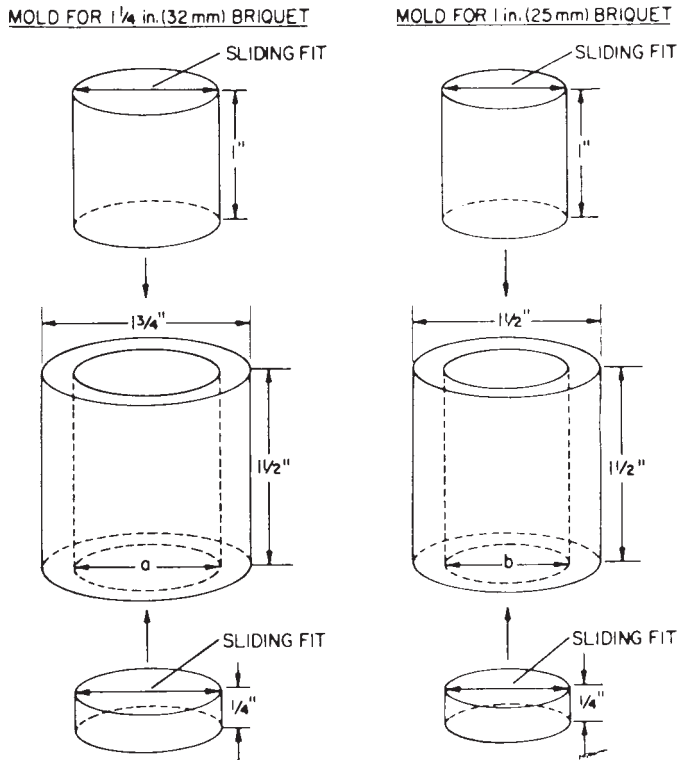
5.5 *Molds*—containers to hold the coal binder mixture while the binder hardens.

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 (1 $\frac{1}{4}$ -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.

⁴ A suitable riffle sampler is made by Sepor Laboratory, PO Box 736, Ottawa, IL.



NOTE 1—Material: cold rolled or stainless steel. Dimensions a and b (inside diameters) are nominally 32 mm (1/4 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

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

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 shall not react with the coal or the atmosphere or expose the coal sample to a temperature above 100°C.

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—An epoxy resin⁶ has been found suitable. The resin is composed of the diglycidyl ethers of bis-phenol-A and its homologs and is diluted with butyl glycidyl ether. The resin is used with an activator composed of aliphatic polyamines or their adducts or both. The amount of activator is calculated from the equation:

$$A = 100 M_A/EH \quad (1)$$

where:

A = amount of activator, parts per 100 (by weight) of resin diluent mixture;

M_A = molecular weight of activator;

E = epoxide equivalent weight of epoxy-diluent mixture; and

H = number of active hydrogens per molecular of activator.

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.⁷

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.⁸

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

7. Sampling

7.1 Obtain samples (Note 4) of coal in accordance with Test Methods D 2234 and Practice D 4596, 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.

⁵ Buehler, Lt., 2120 Greenwood St., Evanston, IL, and Geoscience Instruments Corp., 110 Beckman St., New York, NY, supply grinding and polishing machines with automatic attachments.

⁶ Armstrong epoxy resin C-4 with activator "D," supplied by Armstrong Products Co., Inc., Warsaw, IN, and Helix Bonding Agent R-283 with hardner B, supplied by Carl H. Biggs Co., Inc., 1547 14th St., Santa Monica, CA, are suitable.

⁷ Orthofire Separator, produced by Vernon-Benshoff Co., Pittsburgh, PA, and S-122 Fluorocarbon release agent, produced by Miller-Stephenson Chemical Co., Inc., Chicago, IL have been found suitable for this purpose. The precautions listed on the label should be followed when using the first material.

⁸ Texmet, supplied by Buehler, Ltd., 2120 Greenwood St., Evanston, IL, and Politex, supplied by Geoscience Instruments Corp., 110 Beckman St., New York, NY, have been found suitable for this purpose.

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

7.3 Crush and divide the gross sample in accordance with Method D 2013 to obtain a subsample (Note 4) of either 4.75-mm (No. 4) or 2.36-mm (No. 8) sieve size.

NOTE 4—The terms *gross sample* and *subsample* are defined, and the quality and amount of the subsample are specified in Method D 2013.

7.4 Crush oversize particles (if present) of the subsample until all pass a 4.75-mm (No. 4) sieve. Reduce this subsample by riffing with the coarse riffle sampler to not less than 250 g. Except for the final weight, the procedure shall be in accordance with Method D 2013.

7.5 Crush this 250-g subsample until 100 % passes a 850-μm (No. 20) sieve in a manner such that production of fines is minimized. This can be accomplished by stage grinding (successive sieving with intermittent grinding of the oversize coal for short periods until all coal passes the specified size) with a mortar and pestle. Other apparatus or procedures that do not produce appreciably more fines may be used.

7.6 Pass the sieved coal sample through the fine riffle sampler (5.3) to obtain a sample of 8 to 16 g for a briquet about 25 mm (1 in.) in diameter or 14 to 20 g for a briquet about 32 mm (1¼ in.) in diameter. If the coal sample has not previously been air dried, air dry this sample at room conditions.

7.7 Place portions of the coal sample to be shipped or stored in a container that meets the requirements of 5.9.

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. A suitable procedure for use with the epoxy resin described in Note 3 is as follows:

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 to harden adequately for polishing. Overnight at room temperature or 4 to 8 h at 35 to 40°C is sufficient.

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 examination. 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× 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 Unless it has been established that the reflectance of the coal is not significantly affected by moisture content, place the briquet in a desiccator for at least 15 h before measurement of reflectance.

NOTE 6—During the wet polishing process, some coals take up moisture that can significantly affect the amount of light reflected from the coal. In general, the greater the amount of water absorbed the less the reflectance. The amount of moisture taken up by a coal is affected by

factors other than rank. 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.

APPENDIX

(Nonmandatory Information)

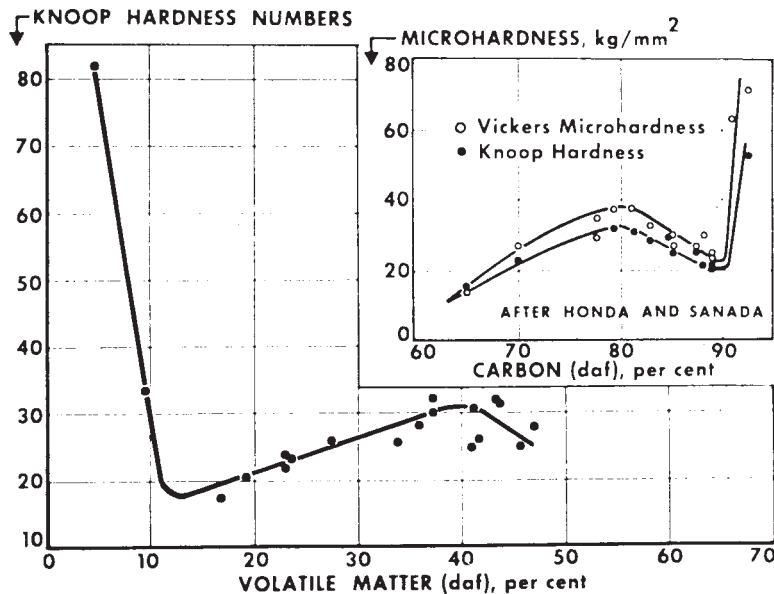
X1. NOTES ON GRINDING AND POLISHING PRACTICES

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

 **D 2797 – 85 (1999)**

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