Designation: D 5671 – 95 (Reapproved 2001)

Standard Practice for Polishing and Etching Coal Samples for Microscopical Analysis by Reflected Light[1]

This standard is issued under the fixed designation D 5671; 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 preparing an etched, polished surface of granular and block samples of coal for examination with a microscope using reflected light illumination.

1.2 The values stated in SI units shall be considered as 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 121 Terminology of Coal and Coke[2]

D 2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light[2]

D 2798 Test Method for Microscopical Determination of the Reflectance of the Vitrinite in a Polished Specimen of Coal[2]

D 2799 Test Method for Microscopical Determination of Volume Percent of Physical Components of Coal[2]

D 4596 Practice for Collection of Channel Samples of Coal in a Mine[2]

D 5192 Practice for Collection of Coal Samples from Core[2]

3. Terminology

3.1 Terminology used in this standard can be found in Terminology D 121.

4. Summary of Practice

4.1 A subsplit of a representative sample obtained in accordance with Practice D 4596 and prepared in accordance with Practice D 2797 or a block of coal obtained in accordance with Practice D 5192 is polished to a flat, scratch-free surface, the reflectance of vitrinite is determined on a subsplit in accordance with Test Method D 2798, and then other subsplits are chemically etched using an acidified potassium permanganate solution.

5. Significance and Use

5.1 Components observable in surfaces of coal samples prepared in accordance with the laboratory procedures of this practice will have differential relief that will aid in their maceral identification by visual classification and enables identification of plant parts or tissues that formed the coal.

5.2 Samples prepared by this practice can be used for microscopical determination of the volume percent of physical components of coal in accordance with Test Method D 2799.

6. Apparatus

6.1 *Ultrasonic Cleaner*—large enough for sample holder and sample preparations to be immersed in cleaning solution.

6.2 *Beakers*—glass beakers, 50, 100, and 500 ml capacity, one each for each etching setup.

6.3 *Stirring Rods*—glass, approximately 20 cm long.

6.4 *Hot Plate*—electric or gas-heated with capability for temperature control and rotating stirring magnets.

6.5 *Watch Glasses*—glass, 100–200 mm in diameter depending on size of specimen blocks to be etched.

6.6 *Graduated Cylinders*—glass, 25 and 100 ml.

6.7 *Grinding and Polishing Equipment*—one or several laps on which the coal briquets or blocks can be ground and polished to a flat, scratch-free surface. Laps may be made of aluminum, iron, brass, or bronze.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used,

[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] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the quality of the etch.

7.2 *Potassium Permanganate* ($KMnO_4$), crystals.

7.3 *Sodium Sulfite* (Na_2SO_3), anhydrous, granular.

7.4 *Sulfuric Acid* (H_2SO_4), 47 % H_2SO_4.

7.5 *Sodium Hydroxide* solution, dissolve 10 g NaOH crystals in 90 g deionized water at room temperature.

8. Materials

8.1 *Grinding Abrasives*—Water-resistant, adhesive-backed silicon carbide papers of 45, 32, and 15 µm (240, 400, and 600 grit). Two or more of these can be used according to a plan such as one of those listed in Table 1.

8.2 *Polishing Abrasives*—Levigated aluminum oxide powders of 1.0 µm size (aqueous suspension) and colloidal silica of 0.06 µm size (in a prepared NaOH suspension).

8.3 *Lap Coverings*—Chemotextile material backed with water-resistant adhesive or similar quality lap coverings. Recommendations of the manufacturer of the polishing abrasive used should be followed for choice of lap covering.

8.4 *Diamond Impregnated Lap Wheel*—Impregnated with diamonds of 6 µm size.

8.5 *Detergent or Sonic Cleaning Solution*—Any nonoxidizing detergent may be used for cleaning sample surfaces after each grinding and polishing stage.

8.6 *Binder*—A potting epoxy resin and hardener or potting polyester resin and hardener that has a curing temperature less than 100°C.

8.7 *Sample Molds*—Prepared for block samples and is made from potting-type silicone rubber.

8.8 *Release Agent*—Spray silicon or any other preparation that does not damage the molds or adversely affect the coal or mounting medium may be used to coat the inside of the briquette mold and facilitate ejection of the briquet.

NOTE 1—Molds prepared from silicone rubber as described in Appendix X1 do not require release agent.

9. Sample Preparation

9.1 *Coal Briquets*:

9.1.1 Prepare granular samples as briquets in accordance with Practice D 2797.

9.2 *Coal Blocks*:

9.2.1 Obtain specimens from core or as blocks of coal from a mining face.

9.2.2 Trim specimens to about 0.5 mm smaller than the volume of the silicone rubber molds.

TABLE 1 Suggested Plans for Grinding and Polishing of Briquets and Blocks

Plan No.	Grinding with Silicon Carbide Paper			Polishing	
	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2
1	45µ m (240 grit)	22 µm (400 grit)	15 µm (600 grit)	1 µm Alumina	0.06 µm Colloidal Silica
2	22 µm (400 grit)	15 µm (600 grit)	...	1 µm Alumina	0.06 µm Colloidal Silica

9.2.3 Air dry the specimens to remove visible surface moisture.

NOTE 2—Overdrying specimens of low rank coals at any point in preparation can cause slaking or severe desiccation of specimen. In contrast, underdrying of specimens will prevent epoxy from setting properly.

9.2.4 Mix resin and hardener according to manufacturer's instructions.

9.2.5 Place specimens and labels into silicone rubber molds and pour resin mixture over the specimens and labels up to the level of the top of the molds. Allow to cure, then remove the specimens from the molds.

10. Preparation of Sample Surface

10.1 Grind and polish on the base surfaces of the briquet or block on a lap in a wet slurry to obtain a surface suitable for microscopical examination. Grinding and polishing should be done with automated equipment. Use a series of abrasives of decreasing particle size according to a plan such as one of those described in Table 1.

11. Determination of Etching Time

11.1 In this procedure, the etching time is determined from the relationship between optimum etching time and measured reflectance of unetched polished vitrinite (Fig. 1).

11.1.1 Measure reflectance of vitrinite on a subsplit in accordance with Test Method D 2798.

11.1.2 Using the relationship shown in Fig. 1, determine the etching time required for obtaining an optimum etch of the polished sample surface.

12. Etching Procedure[4]

12.1 Preparation of etching solution: To 100 ml deionized water, add and mix 25 g $KMnO_4$, and 5 ml H_2SO_4(concentrated). **Caution:** always add acid to water.

12.2 Preparation of rinsing solution: To 100 ml deionized water, add 25 g Na_2SO_3 and 5 ml H_2SO_4. Stir solution until all Na_2SO_3 has dissolved.

[4] Modified from procedure outlined in Stach, Ernst, 1935, *Lehrbuch der Kohlenpetrographie*: Berlin, Borntraeger, 293 p.; Teichmüller, M. L., 1941, The fine structures of American coals in polished samples and thin sections: Reichsamt für das Jahr 1940, Band 61, p. 20–55.

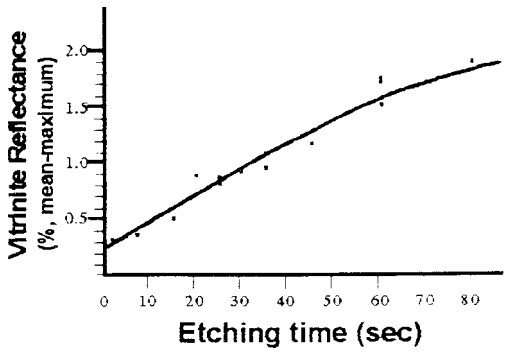

FIG. 1 Determination of Etching Time for Coal on the Basis of Measured Vitrinite Reflectance

12.3 Heat the etching solution in a water bath until most of the $KMnO_4$ has dissolved.

12.4 Pour part of the heated solution into a watch glass and submerse the polished coal surface in the etching solution for the time as determined from Fig. 1 and in accordance with 11.1.

12.5 Remove the coal surface from the solution and immediately rinse with flowing deionized water for 2–3 s to remove etching solution.

12.6 Submerse the coal surface into the rinsing solution for one min or until all purplish stain has been removed.

12.7 Clean the coal surface ultrasonically in deionized water for one minute.

12.8 Dry the surface with a stream of compressed air immediately after removing from ultrasonic bath.

NOTE 3—For some samples, a small area of the polished surface can be masked using cellophane tape smoothed so as to prevent any etching effects.[3] After etching, this tape is removed which produces a line that demarcates the etched and unetched areas.

NOTE 4—For block samples of low rank coal, primarily less than 0.5 % reflectance, blocks can be stored in a shallow bath of deionized water to prevent slaking or severe drying. Prior to examination, the sample surface can be dried with a stream of compressed air. Some low rank coals may also require using a diluted etching solution for the time shown in Fig. 1.

13. Keywords

13.1 coal microscopical analysis; etching; polishing

APPENDIX

(Nonmandatory Information)

X1. PREPARATION OF SILICONE RUBBER MOLDS FOR COAL BLOCKS[5]

X1.1 Construct a wooden block that will fit the block holder of the grinding and polishing equipment. This wooden block will be the size of the samples that will be prepared. Sand all sides of the wooden block to remove severe scratches (generally to a sanding fineness of about 90 µm (120 grit).

X1.2 Construct a mold holder using a smooth surfaced cardboard box that is at least 1 cm larger in all dimensions than the wooden block.

X1.3 Mix part of the silicone rubber according to manufacturer's directions and pour a layer of rubber that is about 1 cm thick into the bottom of the mold holder. Allow rubber to cure.

X1.4 Place the wooden block centered, on top of the layer of cured rubber, pour mixed silicone rubber around the outside of the mold up to level with the surface of thewooden block (Fig. X1.1). Allow rubber to cure.

X1.5 Eject the wooden block from the mold. Mold is ready to use for coal block sample preparation.

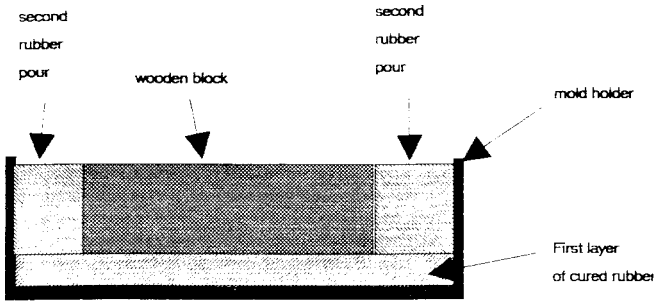

FIG. X1.1 Cross Section View of Materials Used to Prepare a Silicone Rubber Mold for Coal Blocks

[5] Adapted from Moore, T. A., 1991, "Using Silicone Rubber Molds," *The Society of Organic Petrology Newsletter*, vol 8, no. 2, p. 4–5.