



# Standard Practice for Polishing and Etching Coal Samples for Microscopical Analysis by Reflected Light<sup>1</sup>

This standard is issued under the fixed designation D5671; 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 ( $\epsilon$ ) 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 are to be regarded as 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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D121 Terminology of Coal and Coke

D2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light

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

D5192 Practice for Collection of Coal Samples from Core

## 3. Terminology

3.1 Terminology used in this standard can be found in Terminology D121.

<sup>1</sup> 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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<sup>2</sup> 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.

## 4. Summary of Practice

4.1 A subsplit of a representative sample obtained in accordance with Practice D4596 and prepared in accordance with Practice D2797 or a block of coal obtained in accordance with Practice D5192 is polished to a flat, scratch-free surface, the reflectance of vitrinite is determined on a subsplit in accordance with Test Method D2798, 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 D2799.

## 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.<sup>3</sup> 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 quality of the etch.

7.2 *Potassium Permanganate* (KMnO<sub>4</sub>), crystals.

7.3 *Sodium Sulfite* (Na<sub>2</sub>SO<sub>3</sub>), anhydrous, granular.

7.4 *Sulfuric Acid* (H<sub>2</sub>SO<sub>4</sub>), 47 % H<sub>2</sub>SO<sub>4</sub>.

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 **D2797**.

9.2 *Coal Blocks:*

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

<sup>3</sup> *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.

**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	45 μm (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
Colloidal					Silica

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

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**

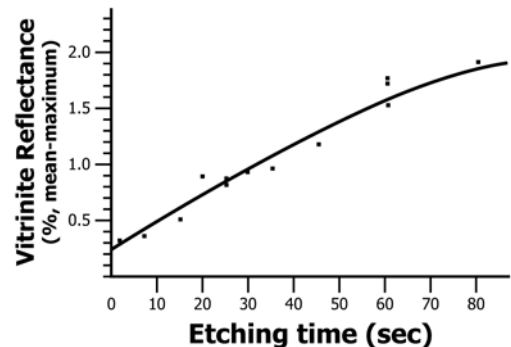
10.1 Grind and polish on the base surfaces of the briquet or block on a lap in a wet slurry to obtain a surface suitable for microscopical examination. Grinding and polishing should be done with automated equipment. Use a series of abrasives of decreasing particle size according to a plan such as one 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 **D2798**.

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.



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