



Designation: D 3997 – 97

## Standard Practice for Preparing Coke Samples for Microscopical Analysis by Reflected Light<sup>1</sup>

This standard is issued under the fixed designation D 3997; 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 the preparation of granular samples of coke for examination with a reflected light microscope. The samples prepared are used for identifying and quantifying the textural components in coke. This practice does not apply to the preparation of oriented lump specimens of coke for structural analysis.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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<sup>2</sup>

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis<sup>2</sup>

D 5061 Test Method for Microscopical Determination of Volume Percent of Textural Components in Metallurgical Coke<sup>2</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>2</sup>

### 3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this practice, refer to Terminology D 121.

3.1.1 *briquette, n*—a cylindrical block composed of granulated coal or coke particles compressed and embedded with an epoxy binder.

### 4. Summary of Practice

4.1 A representative sample is crushed to a specified particle size, oven-dried, mixed with a binder, and formed into a block

specimen referred to as a briquette. The briquette is then polished to a flat, scratch-free surface for microscopical examination under reflected light.

### 5. Significance and Use

5.1 Briquettes of granular coke 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 surface of briquettes prepared using this practice will contain particles representative of the original gross sample.

5.2 Samples prepared by this practice are used for microscopical determination of the textural components in coke (see Test Method D 5061).

### 6. Apparatus

6.1 *Grinder, Pulverizer, Mill, or Jaw Crusher*, or other suitable equipment for final crushing of the sample to pass a 2.36-mm (No. 8) sieve.

6.2 *Coarse Riffle Sampler*, 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.).

6.3 *Medium Riffle Sampler*, with at least twelve divisions of not less than 6.4 mm ( $\frac{1}{4}$  in.) and not greater than 12.7 mm ( $\frac{1}{2}$  in.).

6.4 *Sieves*—A 6.4-mm ( $\frac{1}{4}$ -in.) and 2.36-mm (No. 8) U.S.A. Standard Sieve (see Specification E 11).

6.5 *Molds*—Containers to hold the coke/binder mixture while the binder hardens. Generally, steel cylindrical molds are used (see Fig. 1). However, it is acceptable to use other mold materials that successfully yield the same type of briquette.

6.5.1 The mold shall be made of separable parts or some other design so that the briquette can be ejected after the briquette has hardened.

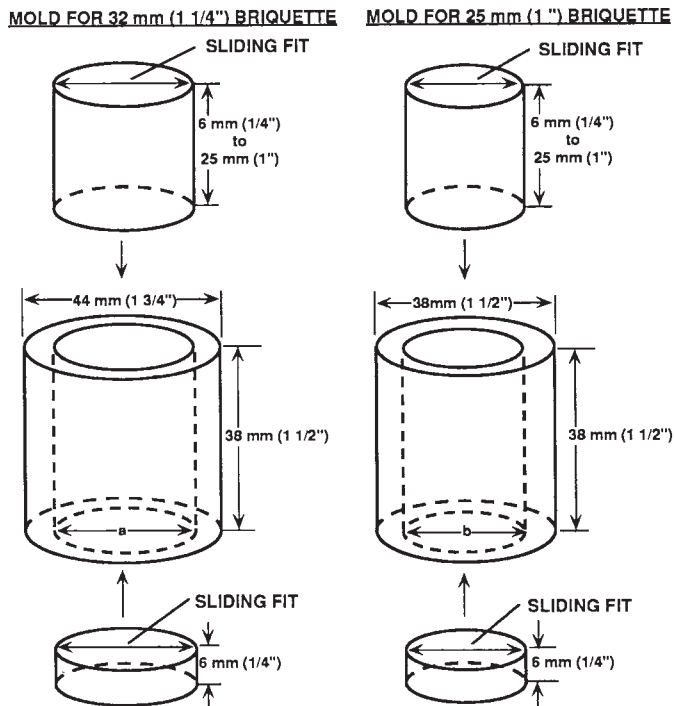
6.5.2 The mold shall be large enough to provide a plane area of 4 cm<sup>2</sup> or more on one side of the briquette. (Designs of suitable 25- and 32-mm (1- and 1¼-in.) inside-diameter molds are shown in Fig. 1).

6.6 *Hydraulic Press*, capable of producing a pressure up to 28 MPa (4000 psi) on the briquette with an attachment to eject the briquette after hardening of the binder.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.05.



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

**FIG. 1** Molds Suitable for Briquetting Coke Samples

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

NOTE 1—Equipment that has 203- or 305-mm (8- or 12-in.) diameter disk laps, gear-driven at 160 to 170 r/min, and has an automatic sample holder attachment is recommended.

6.8 *Sample Cleaner*, essential for cleaning coke briquettes between the different grinding or polishing stages. This may be a simple stream of water or an airjet, but an ultrasonic cleaner is recommended.

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

## 7. Materials

7.1 *Binder*—Any binding material, such as epoxy resin, fulfilling the following requirements may be used for preparing the coke briquette:

7.1.1 The binder shall hold all coke particles securely during grinding, polishing, and observation.

7.1.2 The binder shall not react with the coke or the atmosphere.

7.1.3 Under the microscope, the binder shall contrast markedly with the coke being observed when immersed in oil.

7.1.4 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 coke and the binder, is undesirable for microscopic observation. Relief depends a great deal on the polishing technique.

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

7.3 *Grinding Abrasives*—Water-resistant, adhesive-backed silicon carbide papers of grit Nos. 120, 240, 400, and 600. The grinding and polishing sequences recommended are listed in Table 1.

NOTE 3—It is acceptable to use commercially available diamond impregnated wheels as substitutes particularly for the coarse grinding abrasive papers if the same polish quality requirements are met as specified in Section 10.

7.4 *Polishing Abrasives*—Aluminum oxide slurries in 0.3- and 0.05- $\mu$ m sizes. The grinding and polishing sequences recommended are listed in Table 1.

NOTE 4—It is acceptable to use commercially available colloidal silica as a substitute for aluminum oxide if the same polish quality requirements are met as specified in Section 10.

7.5 *Lap Coverings*—Nap-free cloths or chemotextile material backed with water-resistant adhesive.

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

## 8. Sampling

8.1 Obtain gross samples of coke in accordance with Test Method D 346, when appropriate.

8.2 If the coke appears wet, oven-dry the coke until no surface moisture is evident.

8.3 Crush and divide the gross sample in accordance with Test Method D 346 to obtain a subsample of 500 g of – 2.36-mm (No. 8) sieve size. Particle size reduction is done in such a manner that the production of fines is minimized.

8.4 Pass the dried, sieved coke through the medium riffle sampler (see 6.3) to obtain a sample of about 50 g for a set of two briquettes about 25 mm (1 in.) in diameter.

8.5 A reserve coke sample should be stored as described in 6.9.

## 9. Preparation of Coke Briquettes

9.1 Prepare a coke briquette using a binder that meets the requirements of 7.1 in such a manner that, when polished, at least 60 % of the cross-sectional area will be coke. A suitable procedure for use with epoxy resin is as follows:

**TABLE 1** Suggested Abrasive Sequences for Grinding and Polishing Coke Briquettes<sup>A</sup>

NOTE 1—Alternative abrasives and techniques are permissible if the same polish quality requirements are met as specified in Section 10.

Sequence I—grinding with silicon carbide paper:

1. No. 120 grit paper
2. No. 240 grit paper
3. No. 400 grit paper
4. No. 600 grit paper

Sequence II—polishing with alumina on cloths:

1. 0.3- $\mu$ m slurry on chemotextile
2. 0.05- $\mu$ m slurry on chemotextile

9.1.1 Add activator or hardener, in the amount recommended by the supplier, to about 4 g of epoxy resin for each 10 g of coke. Mix thoroughly with a spatula or disposable wooden stirring stick. Place the riffled coke sample in a suitable small disposable container. Thoroughly stir a few drops of resin into the coke sample. Continue to add resin, a few drops at a time, and stir until all coke particles are wetted and the coke-resin mixture coheres when pressed to the side of the container with the spatula.

9.1.2 Coat the internal surfaces of a mold (including plungers if applicable) with a release agent and insert the lower plunger. Fill the mold with the coke-resin mixture. Insert the upper plunger, place the mold in the hydraulic press, and apply pressure on the briquette for 3 to 5 s. Pressures of less than 28 MPa (4000 psi) have been found to be adequate. Release and reapply the pressure; repeat this cycle three times to expel air bubbles.

9.1.3 Allow the briquette(s) to harden overnight at room temperature or apply heat to harden as recommended by the manufacturer of the epoxy.

9.1.4 Eject the coke briquette from the mold using the special attachment on the press and label the briquette.

**NOTE 5**—It is acceptable to use 32- or 38-mm (1¼- or 1½-in.) preformed one-piece molds that do not include separable parts and do not require pressure during the coke-briquetting procedure. This type of briquette is commonly referred to as a loose mount. It is also acceptable to use other types of binding material other than the commonly used activator/resin-based epoxy. Binding powders used in the proper ratio of

coke to binder with proper heat treatment have also been found acceptable.

## 10. Preparation of Briquette Surface

10.1 Grind and polish one of the flat surfaces of the briquette on a lap to obtain a surface suitable for microscopical examination. Grinding and polishing can 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 the one described in Table 1. The surface so obtained shall meet the following requirements:

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

10.1.2 The surface shall be free of pits caused by loss of grains of coke or mineral matter.

10.1.3 The surface shall be reasonably free of scratches when examined at a magnification of  $\times 500$  or more under oil immersion.

10.1.4 The surface shall be reasonably free of relief.

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

10.2 After each grinding or polishing step, clean the briquette(s) to remove all abrasive and dislodged coke and mineral cuttings. Use of an ultrasonic cleaner filled with water and detergent is recommended.

## 11. Keywords

11.1 briquettes; coke; microscopy

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