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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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# Standard Test Method for Solvent-Extractable Material in Black Pigments[1]

This standard is issued under the fixed designation D 305; 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 test method covers the determination of the solvent-extractable material in black pigments such as carbon black, lampblack, and bone black.

1.2 *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 329 Specification for Acetone[2]

## 3. Significance and Use

3.1 This test method is used by black pigment producers and users for product acceptance.

## 4. Apparatus

4.1 *Extraction Apparatus*, consisting of a flask, siphon cup, and a condenser similar to the apparatus shown in either Fig. 1 or Fig. 2.

NOTE 1—A Sophlet apparatus may be used as an alternative.

4.2 *Extraction Thimbles*—The thimble must be made of greaseless paper and be of correct size to fill the selected apparatus.

NOTE 2—The recommended thimble size for each of the apparatus shown in Fig. 1 is listed below:

| | Fig. 1 | Fig. 2 |
|---|---|---|
| Height | 70 mm | 62 mm |
| Inside diameter | 28 mm | 22 mm |
| Outside diameter | 30 mm | 24 mm |

4.3 The thimbles are available in various heights and widths with two thickness levels, single thickness or double thickness. Single-thickness thimbles are recommended for this extraction procedure.

## 5. Reagents and Material

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

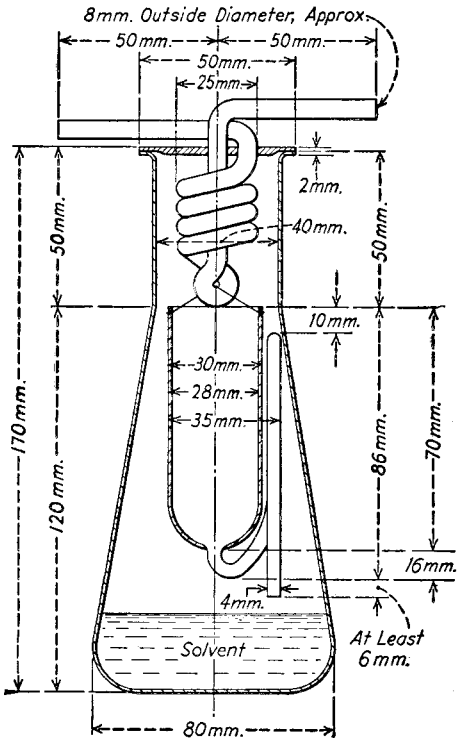


**FIG. 1 Extraction Apparatus**

5.2 *Solvent*—Acetone and benzene are the most commonly used solvents (see Specification D 329); however, other solvents may be used as agreed upon.

5.3 *Glass Wool.*

## 6. Procedure

6.1 Weight approximately 10 g of pigment (moisture-free)

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

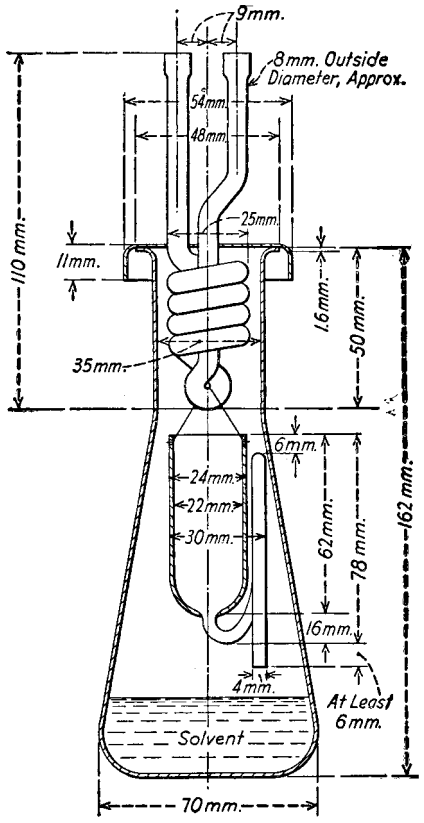


**FIG. 2 Extraction Apparatus**

into a weighed thimble and record the weight to 0.1 g. For pigments with high apparent density (bone black, iron oxides, etc.) a 40-g sample is recommended. Plug the open end of the thimble with glass wool and place in the siphon cup. Add 200 to 250 mL of the solvent to the previously dried flask.

6.2 When using acetone as the extraction solvent, extract continuously for 4 h, heating at a rate such that the time required to fill and empty the siphon cup will not exceed 8 min. With other solvents extract for 16 h.

NOTE 3—If particles of carbon have escaped from the cup into the extraction liquid, filter the liquid, wash the paper with solvent, and return the total filtrate to the extraction flask, prior to continuing with 6.3.

6.3 Transfer quantitatively the extract solution to a 400-mL beaker that has been weighed to the nearest 0.1 g, and evaporate off the solvent on a steam bath or plate. Remove the beaker from the bath or hot plate just before the last trace of the solvent disappears. Dry the dish for 1 h at 105°C (221°F). Cool and weigh to the nearest 0.1 g.

## 7. Calculation

7.1 Calculate the percent extractable material, *E*, to the nearest 0.05 % as follows:

$$E = [(R - B)/(S - W)] \times 100$$

where:

*R* = weight of extractable material and weighed dish, g,
*B* = weight of weighed dish, g,
*S* = weight of thimble and specimen, g, and
*W* = weight of thimble, g.

## 8. Precision

8.1 Precision data are not available at this time. When they are available the appropriate precision statements will be added.

## 9. Keywords

9.1 black pigment; solvent extractable