Designation: D4930 – 06 (Reapproved 2011)

Standard Test Method for
Dust Control Material on Calcined Petroleum Coke[1]

1. Scope

1.1 This test method covers the determination of the amount of material applied to calcined coke to control dust associated with coke handling and transportation.

1.2 This test method is limited to those materials that are soluble in a solvent that can be used in a Soxhlet extraction type of apparatus such as methylene chloride (dichloromethane).

NOTE 1—Methylene chloride is the most popular solvent for removing dust control oil at the time this procedure is being written. Toluene and methyl chloroform, however, have been used with equal results on all cokes tested which have included only those sprayed with aromatic or waxy materials.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.* For specific warning statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D2013 Practice for Preparing Coal Samples for Analysis
D2234/D2234M Practice for Collection of a Gross Sample of Coal
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions:*

3.1.1 *analysis sample, n*—the reduced and divided representative portion of the bulk sample, prepared for use in the laboratory.

3.1.2 *bulk sample, n*—the reduced and divided representative portion of the gross sample as prepared for shipment to and received by a laboratory, to be prepared for analysis.

3.1.3 *test sample, n*—the weighed portion of the analysis sample actually used in a test.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *de-dusting material, n*—a substance, such as a petroleum oil, a petroleum wax, an aqueous latex emulsion, and the like, that is applied to (sprayed on) calcined petroleum coke to control dust associated with coke handling and transport.

4. Summary of Test Method

4.1 The dust control material is dissolved from a weighed dry representative sample of 6.3-mm maximum sized coke by methylene chloride in a Soxhlet extraction apparatus. The mass of the residue remaining after distilling and evaporating the solvent is the mass of dust control material. The amount of residue as a percent of the original mass is calculated.

5. Significance and Use

5.1 The test is designed to quantify the amount of dust control material added to calcined coke. The dust control material is applied to calcined coke to help maintain a dust-free environment. It generally serves no other useful purpose. It adds weight to the coke and can have a negative effect on the quality of carbon and graphite artifacts made from the treated coke. For these reasons the coke customer wants to know the amount of dust control material on the coke and can specify a maximum level.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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[2] 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.

6. Apparatus

6.1 *Extractor,* with condenser, Soxhlet, 200 mL capacity.[3]

6.2 *Flask,* round bottom with short neck, 250-mL.

6.3 *Heating Mantle,* for 250-mL flask.

6.4 *Boiling Chips.*

6.5 *Thimbles, Cellulose Extraction,* 43 mm in diameter by 123 mm in height (single thickness; 9 to 11 µm for *liquid* retention).

6.6 *Analytical Balance,* capable of weighing to 0.1 mg.

6.7 *Hot Plate,* with temperature control ±3°C between 90 and 110°C.

6.8 *Hooked Glass Tube,* 3-mm inside diameter, 225-mm long, bent such that about 75 mm extends into the flask when connected into the neck of the flask.

6.9 *Flexible Tubing.*

6.10 *Drying Oven, Forced Draft or Vacuum* (vacuum preferred if toluene is used).

6.11 *Desiccator.*

6.12 *Rifflers,* encased with hoppers.

6.13 *Hood.*

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.[4] 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.

7.1.1 *Methylene Chloride,* reagent grade with *residue after evaporation* specification of 0.002 % or less (any other solvent used shall be reagent grade with no greater residue). (**Warning—**The use of methylene chloride is extremely volatile (BP = 40°C) and mildly toxic.) (**Warning—***All* handling of methylene chloride, including volumetric measurement and transfer of the solvent to the extraction apparatus, must be done in an approved fume exhaust hood. Extractions and subsequent methylene chloride drying procedures *must* also be performed in this same hood.[5])

7.1.2 *Acetone,* reagent grade. (**Warning—**Acetone is extremely flammable.) (**Warning—**Keep away from heat, sparks, and flame and keep container closed when not actually pouring acetone. Use only in a well-ventilated area. Avoid inhalation and contact with the eyes or skin.)

8. Sample Preparation

8.1 Riffle divide the bulk sample to obtain a 200 g analysis sample.

8.2 Reduce the analysis sample to pass a 6.3-mm sieve by one pass through a jaw crusher set to not crush smaller than 6.3 mm (a roll crusher can be used by stage crushing to 6.3 mm).

8.3 Dry the reduced analysis sample at 110 to 115°C to constant weight (about 8 h may be required; overnight drying preferred when convenient). Store in a desiccator until ready to test.

NOTE 2—The recommended practices for collecting samples and the equipment and procedures for crushing and dividing are described in Practice D346, D4057, and Practice D2013 and Test Methods D2234/D2234M. Specifications for sieves are described in Specification E11.

9. Procedure

9.1 From the dried analysis sample, riffle to obtain a test sample of about 75 g. Weigh the test sample to the nearest 0.01 g and record the mass as W_s.

9.2 Charge an extractor thimble with the test sample, place the thimble in the extractor, and assemble extractor and condenser.

NOTE 3—Do not use grease on joints. Use TFE-fluorocarbon sleeves to control leaking problems.

9.3 Put two or three clean boiling chips into a clean, dry boiling flask and weigh to the nearest 0.1 mg. Record the mass as W_1.

NOTE 4—Because of the required weight precision, handle the flask with clean tongs or gloves.

9.4 Pour 210 mL of methylene chloride into the boiling flask and assemble it to the extractor.

9.5 Start the cooling water flow to the condenser. Raise the heating mantle to the flask and adjust the power to bring the solvent to a boil. Adjust the heat to control the reflux rate, being careful not to override the capacity of the condenser. About 2 drops/s reflux to the thimble should be satisfactory. An aluminum foil tent around the upper portion of the flask and lower portion of the extractor will help speed up reflux rate.

9.6 After the extractor has gone through four dump cycles, continue heating until the chamber around the sock is two-thirds full. Note the color of the solvent in the thimble region. If it is not clear, continue refluxing through the fifth cycle (dump and fill sock until two-thirds full). Continue in this fashion until the solvent in the thimble region is clear. Several dump cycles can be necessary when excessive fines are present or when the de-dust oil is only mildly soluble (asphalt or "heavy" vacuum reside).

9.7 Bring approximately 200 mL of deionized water to a boil in the 300-mL beaker.

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[3] The sole source of supply of the apparatus known to the committee at this time is the Soxhlet extraction apparatus, Kimax (glass), 24005 series, catalog No. 393-2711, available from Curtin Matheson Scientific, Inc., 9999 Veterans Memorial Dr., Houston, TX 77038. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] *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 *Annual 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.

[5] Occupational Health Guidelines for Chemicals "Methylenechloride," DHHS (NIOSH) Publication No. 81-123, January 1981.

9.8 Remove the flask from the extractor and place it on top of the beaker of boiling water under a fume hood. Start a small flow of air through the bent glass tube, then insert the tube into the neck of the flask being careful not to touch the liquid. Air flow shall be low enough to avoid splattering of the liquid.

9.9 After the methylene chloride has evaporated, dry the outside of the flask, rinse the outside with acetone, then place the flask with its contents in a drying oven (or vacuum oven) at 110 ± 2°C (and approximately 100 mm Hg), for 15 min.

9.10 Remove the flask from the drying oven and cool to room temperature in a desiccator. Weigh the flask and contents to the nearest 0.1 mg and record the weight as W_2.

9.11 Repeat the procedure from 9.1-9.10 with a second test sample.

10. Calculation

10.1 Calculate the amount of residue as a percent of the original sample weight as follows:

$$\text{mass\% residue} = \frac{W_2 - W_1}{W_s} \times 100 \quad (1)$$

where:

W_s = mass of the test sample, g.
W_1 = mass of the boiling flask and chips.
W_2 = mass of the boiling flask, chips, and residue.

11. Report

11.1 Report the average of duplicate results to the nearest 0.01 % of dust control material on the coke.

12. Precision and Bias[6]

12.1 Precision is dependent on the absolute amount of oil on the coke as well as the efficiency of the sampling and riffling procedure used (due to variability of the oil application).

12.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.04$$

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.15$$

12.2 *Bias*—No justifiable statement can be made on the bias of the procedure in this test method because it depends on conformance to the empirical conditions of this test.

13. Keywords

13.1 calcined petroleum coke; de-dusting material; dust control material

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1242.