

SPECIFIC GRAVITY OF COKE AND POWDERED ASPHALTS

UOP Method 822-81

SCOPE

This method is for determining the specific gravity of coke derived from either petroleum or coal. Other granular materials can be analyzed if they can be ground to pass a No. 200 sieve, are essentially insoluble in ethylene glycol and have a specific gravity greater than ethylene glycol. The method is also applicable to hard asphalts and similar materials that can be ground to pass through a No. 100 sieve and are essentially insoluble in *tert*-amyl alcohol.

OUTLINE OF METHOD

A representative sample of the material is ground to pass through either a No. 100 or 200 sieve. Coke samples are ground to pass through a 200 sieve to eliminate small voids. Asphalts are ground to pass through a 100 sieve because it is not practical to grind them finer. A portion is placed in a tared Hubbard-Carmick pycnometer and weighed. A displacement liquid is added to the pycnometer and the pycnometer is reweighed. The specific gravity is then calculated from the values obtained.

APPARATUS

Balance, capable of weighing to 0.1 mg

Bath, refrigerated, -15 to 100 C range, VWR Scientific, Cat. No. 13276-944, or equivalent

Beaker, one-liter

Desiccator, vacuum, plastic, 250-mm ID, Sargent-Welch Scientific Co., Cat. No. S-25136, or equivalent

Gauge, vacuum, dial type, Sargent-Welch Scientific Co., Cat. No. S-39706-20, or equivalent

Grinding container, Spex Industries Inc., Cat. No. 8004WC, or equivalent

Grinding mill, Mixer Mill, Spex Industries Inc., Cat. No. 8000, or equivalent

Mortar and pestle, Diamonite, Fisher Scientific Co., Cat. No. 12-952-5, or equivalent

Mortar and pestle, heavy, porcelain, Coors size 2/52011 and 2/52012, Sargent-Welch Scientific Co., Cat. Nos. S-62250-50E and S-62251-E, or equivalent

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Pump, high vacuum, Sargent-Welch Scientific Co., SarVac Model 8803B, or equivalent

Pycnometer, Specific Gravity Bottle, Hubbard-Carmick, Sargent-Welch Scientific Co., Cat. No. S-2365, or equivalent

Sieve, 8-inch diameter, USA Series Nos. 100 and 200, with cover and bottom pan, Sargent-Welch Scientific Co., or equivalent

Thermometer, ASTM 63 C, -8 to 32 C

REAGENTS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified.

Ethylene glycol, 99.9% minimum purity

tert-Amyl alcohol, 99.9% minimum purity

CALIBRATION OF PYCNOMETER

Calibrate the pycnometer at a temperature of 15.6 or 25 C according to ASTM D 70 with the following exception: fill the pycnometer with either ethylene glycol or *tert*-amyl alcohol (see *PROCEDURE*) instead of water. Do not submerge the pycnometer.

PROCEDURE

The Sample taken for analysis should be representative of the total batch from which the sample was obtained.

Coke

Crush 10 to 15 g of the sample in any convenient manner and then reduce it in the grinding mill to pass through a No. 200 (75 μ m) sieve. If any of the sample does not pass through the sieve further reduce it using the Diamonite mortar and pestle until all of it passes through the sieve.

Place 3 to 4 g of the ground sample into a clean, dry, calibrated, tared pycnometer and weigh it to the nearest 0.1 mg. Add enough ethylene glycol (see *NOTE*) to cover the sample by approximately 5 mm. This is best done by using a plastic wash bottle and directing a stream of ethylene glycol around the interior wall of the pycnometer. Place the pycnometer into a vacuum desiccator. Slowly evacuate the desiccator to a vacuum of 3.3 kPa (25 mm) or less to remove air from the sample. Use caution to prevent excessive bubbling from degassing of the sample. Slowly allow the desiccator to return to atmospheric pressure. Repeat this cycle 4 or 5 times. Remove the pycnometer from the desiccator and fill it with ethylene glycol. Insert the stopper making certain that the glycol fills the capillary in the stopper. Carefully place the pycnometer in a beaker containing enough water so that the water level will be within 3 to 4 mm of the top of the body of the pycnometer. Partially immerse the beaker containing the pycnometer in a constant temperature bath at 25 ± 0.1 C. Keep the capillary filled with glycol while the pycnometer is in the bath by maintaining a drop of glycol on the stopper top. Allow the pycnometer to remain in the bath at the required temperature for a minimum of 30 minutes. Remove the pycnometer, wipe the drop of glycol from the top of

the stopper, and dry the rest of the pycnometer. Do not redry the top of the stopper. Immediately weigh the pycnometer to the nearest 0.1 mg.

Asphalt

Crush about 10 g of sample in any convenient manner and then reduce it, using the Coors mortar and pestle, to pass through a No. 100 (150 μm) sieve. Place 2 to 3 g of the ground sample into a clean, dry, calibrated, tared pycnometer and weigh it to the nearest 0.1 mg. Add enough *tert*-amyl alcohol to cover the sample by approximately 5 mm.

Place the pycnometer into a vacuum desiccator. Slowly evacuate the desiccator to remove air from the sample. Use caution to prevent excessive bubbling from degassing of the sample or from evaporation of the alcohol which boils at 102 C at 101 kPa (760 mm). Slowly allow the desiccator to return to atmospheric pressure. Repeat this cycle 4 or 5 times. Remove the pycnometer from the desiccator and fill it with alcohol. Insert the stopper making certain that the alcohol fills the capillary in the stopper. Carefully place the pycnometer in a beaker containing enough water so that the water level will be within 3 to 4 mm of the top of the body of the pycnometer. Partially immerse the beaker containing the pycnometer in a constant temperature bath held to within ± 0.1 C of the desired temperature (15.6 or 25 C). Keep the capillary filled with alcohol while the pycnometer is in the bath by maintaining a drop of alcohol on the stopper top. Allow the pycnometer to remain in the bath at the required temperature for a minimum of 30 minutes. Then remove the pycnometer, wipe the drop of alcohol from the top of the stopper, and dry the rest of the pycnometer. Do not redry the top of the stopper. Immediately weigh the pycnometer to the nearest 0.1 mg.

CALCULATIONS

Calculate the specific gravity to the nearest third decimal as follows:

$$\text{Specific Gravity} = \frac{(C - A)E}{[(B - A) - (D - C)]}$$

where:

- A** = weight of empty pycnometer, g
- B** = weight of pycnometer filled with glycol or alcohol at test temperature, g
- C** = weight of pycnometer plus sample, g
- D** = weight of pycnometer plus sample plus glycol or alcohol at the test temperature, g
- E** = specific gravity of glycol or alcohol at the test temperature

NOTE

Keep the ethylene glycol container tightly covered when not in use, since it rapidly picks up moisture from the air. Use fresh glycol for each test.

PRECISION

Based on the number of replicates indicated, the estimated standard deviations (esd) for a sample of coke and a sample of asphalt were calculated and are shown in the following table.

Duplicate results by the same operator should not differ by more than the allowable difference shown (95% probability).

<u>Sample</u>	<u>Number of Replicates</u>	<u>Specific Gravity</u>	<u>esd</u>	<u>Allowable Difference</u>
Coke	10	2.078	0.0012	0.004
Asphalt	6	1.102	0.0028	0.010

TIME FOR ANALYSIS

The elapsed time for one analysis is 3 hours. The labor requirement is 1.5 hours.

REFERENCE

ASTM Method D 70, www.astm.org

SUGGESTED SUPPLIERS

Fisher Scientific, 1600 Glenlake Ave., Itasca, IL 60143
 Sargent-Welch Scientific Co., 7300 Linder Ave., P.O. Box 1026, Skokie, IL 60077
 Spex Industries, Inc., P.O. Box 798, Metuchen, NJ 08840
 VWR Scientific, 2619 Congress St., Bellwood, IL 60104