

Chapter 15

Tex-215-F, Determining Asphalt Content of Rock Asphalt by Hot Solvent Extraction

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Section 1

Overview

Effective Dates: August 1999 to November 2004.

This method provides a procedure to determine, by hot solvent extraction, the percentage of asphalt in native rock asphalt aggregate. Other methods to determine asphalt content that correlate satisfactorily to the Soxhlet procedure results may be used.

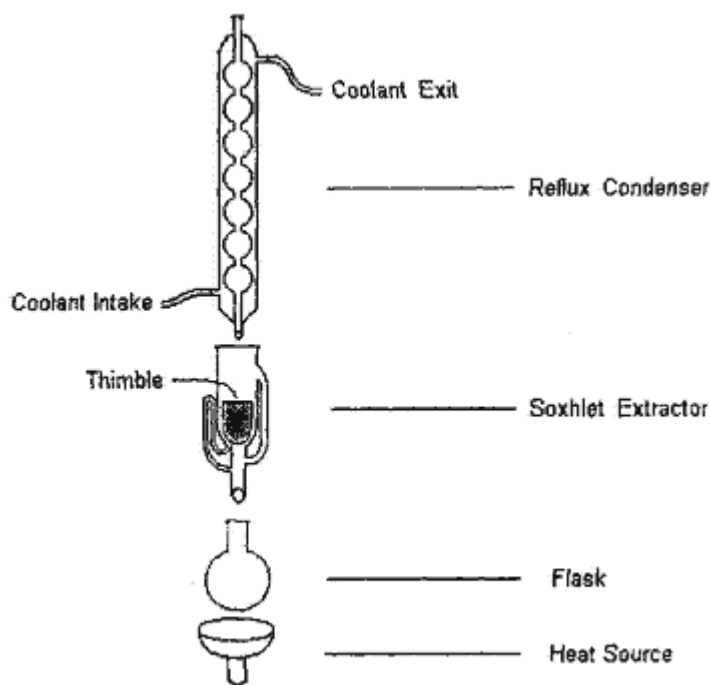
Units of Measurement

The values given in parentheses (if provided) are not considered to be standard and may not be exact mathematical conversions. Each system of units shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

Section 2 Apparatus

The following apparatus is required:

- ◆ 'Soxhlet apparatus,' an extractor consisting of a vertical glass cylinder with two tubes (one a siphon tube) attached at the upper end to a reflux condenser and to a 500 mL (17 fl. oz.) Pyrex glass flask at the lower end.
- ◆ Whatman fat-free filter thimble (or equivalent) 80 mm (3.15 in.) in length and 33 mm (1.3 in.) in diameter
- ◆ electric hot plate, heat lamp or suitable source of heat
- ◆ burner or muffle furnace
- ◆ electric oven capable of maintaining a temperature of 60 to 110 °C (140 to 230 °F)
- ◆ a balance readable to 0.01 g and accurate to 0.01 g
- ◆ mechanical pulverizer
- ◆ desiccator
- ◆ silica ignition dish, 125 mL (4.3 fl. oz.) capacity
- ◆ miscellaneous items, such as tubing, small scoop, spatula, brush, tongs, etc.



Soxhlet Apparatus

Figure 15-1. Soxhlet Apparatus.

Section 3

Materials

The following materials are needed:

- ◆ solvent: trichlorethylene or methylene chloride
- ◆ ammonium carbonate solution – saturated solution of ACS Grade $(\text{NH}_4)_2\text{CO}_3$.

Section 4

Preparing Sample

Use the following procedure to prepare the sample.

- ◆ Mix the rock asphalt aggregate and quarter to obtain a representative sample weighing 1000 g.
- ◆ Thoroughly blend the material and take small portions from several places in the pan.
- ◆ Pulverize this material in mechanical pulverize to pass the 2.00 mm (No. 10) sieve.

Section 5 Procedure

Follow these steps to determine the asphalt content of rock asphalt by hot solvent extraction.

Determining Asphalt Content of Rock Asphalt by Hot Solvent Extraction	
Step	Action
1	Place a filter thimble and cotton plug (sufficient to plug the open end of thimble) in a 60 °C (140 °F) oven to dry for approximately 30 minutes.
2	<ul style="list-style-type: none"> ◆ Allow to cool in desiccator and obtain the weight to nearest 0.01 g. ◆ Record this weight as B under 'Calculations.' NOTE: A suitable weighing bottle will support the thimble, or the bottom of the thimble can be indented so it will stand erect while weighing.
3	Transfer 50 ± 3 g of the pulverized rock asphalt into the filter thimble (about ½ full).
4	Place the loose cotton plug in the top of the thimble, and place in an oven and dry to a constant weight at a minimum temperature of 60 °C (140 °F). <ul style="list-style-type: none"> ◆ Avoid overheating. ◆ Drying may be done in a pan in an oven prior to placing in filter thimble.
5	Weigh the thimble, contents, and cotton plug to the nearest 0.01 g.
6	Record weight as A under 'Calculations.'
7	Place the thimble and contents directly into the Soxhlet extraction apparatus.
8	<ul style="list-style-type: none"> ◆ Fill the flask about ½ full of solvent and place a ball of cotton loosely in the open end at top of the condenser. ◆ Have plenty of cool water flowing continuously through the condenser.
9	Heat the flask just enough to vaporize the solvent.
10	The solvent vapor condenses in the condenser and drops back upon the sample through which it filters, thus dissolving out the asphalt.
11	The solvent siphons over from the cylinder back into the flask where it is distilled again.
12	The asphalt collects in the bottom of the distillation flask.
13	Continue the extraction process until the solvent becomes colorless as it fills the vertical glass cylinder surrounding the filter.
14	The time of extraction will depend upon the amount of asphalt in the sample.
15	Remove the heat and allow the apparatus to cool.
16	Lift the thimble, allow it to drip dry, then dry the thimble and contents in an oven at a minimum temperature of 60 °C (140 °F).
17	<ul style="list-style-type: none"> ◆ Cool the contents to room temperature (preferably in desiccator) and weigh. ◆ Record the weight as E under 'Calculations.'
18	Pour the solution from the flask into a tared silica dish.
19	<ul style="list-style-type: none"> ◆ After the solvent has evaporated, ignite the residue over a burner to ash the bituminous material. ◆ If a muffle furnace is available, place ashed material in the furnace at approximately 650 °C (1200 °F) for 15 or 20 minutes.
20	Cool the dish and contents and add just enough ammonium carbonate (NH ₄) ₂ CO ₃ to wet the ash remaining in the dish.
21	Evaporate the ammonium carbonate from sample on a hot plate until the ash is completely dry.
22	◆ Cool the dish and contents to room temperature in a desiccator and weigh.

Determining Asphalt Content of Rock Asphalt by Hot Solvent Extraction	
Step	Action
	◆ Record the weight to nearest estimated 0.01 g as C under 'Calculations.'
23	◆ Use the net weight (C-D) of the ash to correct for the fine mineral matter which is carried through the filter thimble during extraction. (Omit the use of ammonium carbonate if prior tests do not indicate a need for the re-carbonation of the ash.) ◆ Determine the frequency of ash determination necessary for daily control testing.

Section 6

Calculations

Use the following to calculate the percent of asphalt in native rock asphalt:

$$\text{Percent asphalt} = \frac{A - F}{A - B} \times 100$$

Where:

- ◆ A = weight of sample, filter thimble and cotton
- ◆ B = weight of filter thimble and cotton
- ◆ C = weight of silica dish and ash
- ◆ D = weight of silica dish
- ◆ E = weight of sample, filter thimble and cotton after extraction
- ◆ F = E + C - D = total weight of extracted sample, thimble and cotton plus ash.