WEST VIRGINIA DEPARTMENT OF TRANSPORTATION
DIVISION OF HIGHWAYS
MATERIALS CONTROL, SOILS AND TESTING DIVISION

MATERIALS PROCEDURE

DETERMINATION OF BITUMEN CONTENT OF BITUMINOUS CONCRETE

1.0 PURPOSE

1.1 To describe standard test methods and procedures for finding the bitumen content of bituminous concrete mixtures.

2.0 SCOPE

2.1 This procedure is applicable to acceptance testing of bituminous concrete in plant and central laboratories.

3.0 TEST METHODS AND PROCEDURES

3.1 Test methods for determining bitumen content and the applicable test procedures are listed below.

3.1.1 Centrifuge Extraction - per ASTM Designation D 2172, Method A, and Section 4 of this MP.

3.1.2 Reflux Extraction - per ASTM Designation D 2172, Method B, and Section 4 of this MP.

3.1.3 Maryland Extractor - per ASTM Designation D 2172, Method C, and Section 4 of this MP.

3.1.4 Gauging of Storage Tank - per MP 301.03.21.

3.1.5 Pycnometer Method - per Section 5 of this MP.

3.1.6 Nuclear Method - As per ASTM Designation D 4125.

3.1.7 Modification of the above may be used on fully automated plants with print-outs upon approval by the engineer.

4.0 ALTERNATE PROCEDURE FOR ASH DETERMINATION

4.1 The following procedure for testing ash content of extraction solvents may be used in place of the procedure in Paragraph 8 (f) of ASTM Designation D 2172.

4.1.1 Measure the total volume of solvent used in the extraction. Pour all of the solvent into a single container, agitate thoroughly, and immediately measure approximately 100 ml into a previously weighed ignition dish. Evaporate to dryness on a hot plate. Ash residue at a dull red heat (500°C to 600°C), cool, and stir in saturated ammonium carbonate $(NH_4)_2CO_3$ solution in sufficient quantity to form a paste. Wash any ash retained on the stirring rod into the ignition dish with the ammonium carbonate solution. Digest at room temperature for one hour. Dry in an oven at 100°C for a minimum of three hours. Cool in a desiccator, and weigh.

5.0 PYCNOMETER TEST METHOD

5.1 Equipment

5.1.1 Pycnometer - A vacuum desiccator is used as a pycnometer. It consists of two hemispherical sections. The bottom hemisphere must be made of a heat resistant material such as Teflon. The upper hemisphere should be made of transparent plastic and must have an opening near the top through which it can be filled with water and to which the vacuum line can be attached. The pycnometer should be about 225 mm or larger in diameter. A method of clamping the two halves of the pycnometer together must be provided. This should consist of two sheet metal rings which fit over the rims of the pycnometer halves, and which can be clamped together with bolts and wing nuts.

5.1.2 Balance - capable of weighing 20 kg to an accuracy of 1.0 gm.

5.1.3 Vacuum Source - an aspirator or vacuum pump capable of producing at least 381 mm of vacuum.

5.1.4 Thermometer - having a range of at least from 10°C to 15°C and graduated in 1/2 degree divisions. ASTM thermometer 9C is suitable.

5.1.5 Vacuum Gauge

5.1.6 Mixer - a mechanical mixer such as a Hobart Mixer, or a mixing bowl and stirring rod for hand mixing. A three quart sauce pan and a wooden mixing spoon have been found to be suitable for hand mixing.

5.1.7 Hot Plate

5.1.8 Wetting Agent - Triton, manufactured by Rohm and Haas, or Jet Dry, manufactured by Economics Laboratory, Inc., are suitable.

5.1.9 Tubing - rubber or plastic tubing and a Y or T shaped connector.

5.2 Test Data - The following data must be obtained in order to calculate the test results.

5.2.1 Specific Gravity of bitumen at 16°C. This information is usually printed on the shipping invoice. If not, it can be obtained from the supplier. Check the invoice to determine if the specific gravity on the invoice is the gravity at 16°C. The specific gravity of asphalt at 16°C is normally very close to 1.0 and RT-12 tar is usually between 1.15 and 1.25. If the specific gravity is given in some other form, such as at loading temperature or in degrees API, convert to 16°. See instructions in Attachment D of this MP.

5.2.2 Specific gravity of aggregate. This is determined by testing samples having a known bitumen content. The procedure is explained in Section 6 of this MP.

5.2.3 Temperature calibration chart. The procedure for plotting this chart is given in Section 7 of this MP.

5.3 Test Procedure

5.3.1 A worksheet and sample calculation are shown in Attachment A of this MP.

5.3.2 Weigh the bottom half of the pycnometer, record weight on Line 2 of the worksheet.

5.3.3 Obtain a test sample of sufficient size to almost fill the bottom half of the pycnometer (approximately six kilograms), place sample in bottom half of pycnometer, weigh bottom half of pycnometer containing sample, record weight on Line 1 of the worksheet.

5.3.4 Fill bottom half of pycnometer with water, clamp the top half in place, fill approximately 3/4 full of water, add about two drops of wetting agent, attach vacuum line, apply vacuum sufficient to produce vigorous boiling, and maintain vacuum for approximately 10 minutes.

5.3.5 Remove vacuum line, add water until only a small air space remains in the pycnometer, shake and rotate the pycnometer to remove air bubbles from the sides of the pycnometer, fill completely full of water, insert plug, and wipe all water from outside of pycnometer.

5.3.6 Weigh the pycnometer containing the sample and water, record weight on Line 6 of the worksheet. Immediately after weighing, measure the temperature of the water in the pycnometer, record on Line 8 of the worksheet.

5.3.7 Calculations - Find the weight of the pycnometer plus water for the test temperature from the Temperature Calibration Chart, record on Line 4 of the worksheet. To calculate the bitumen content of the sample, perform the calculations specified in Lines 3, 5, 7, 9, 10, and 11 of the worksheet.

6.0 SPECIFIC GRAVITY OF AGGREGATE

6.1 An example worksheet and sample calculation are shown in Attachment B of this MP.

6.2 Sampling and Sample Preparation.

6.2.1 The specific gravity of the aggregate is determined by testing a sample of the mix which has been prepared in the laboratory and for which the asphalt content has been calculated. In order to obtain accurate results, the gradation and asphalt content of the sample must match, as closely as possible, the gradation and asphalt content of the mix being produced by the plant. The sample should be of about the same size as the samples that are tested for asphalt content.

6.2.2 Obtain a sample of the asphalt and an aggregate sample from each hot bin. If it is not possible to sample from the hot bins, mix a dry batch and either sample it from the pug mill, or dump it into an end loader or truck and sample it there.

6.2.3 Calculate the weight of each material needed in the sample. Formulas and example calculations are given in Attachment D.

6.2.4 Place the mixing bowl on the balance and weigh into it the required quantity of each material. Heat on a hot plate to about 150°C. Mix either by hand, or with a mechanical mixer, until the aggregate is well mixed and completely coated with asphalt.

6.2.4.1 Note: If the sample is mixed by hand, it will usually be easier to mix it in two batches. Each batch should contain 1/2 of the required quantity of each material.

6.3 Test Procedure

6.3.1 Weigh the bottom half of the pycnometer, then transfer the sample from the mixing bowl to the pycnometer half.

6.3.2 If the sample is being mixed in two batches, mix the second half and place in the pycnometer half.

6.3.3 Scrape the mixing bowl and stirring rod as clean as possible. Add this material to the sample in the pycnometer half. Weigh the sample in the pycnometer half.

6.3.4 At this point, there will still be some material stuck to the mixing bowl and stirring rod. To determine the weight of this material, place the stirring rod in the mixing bowl and weigh on the balance, clean with a solvent, such as naphtha, wipe dry, and reweigh.

6.3.5 Test the sample, following steps 5.3.4 through 5.3.6 of this MP.

6.3.6 Calculate the specific gravity of the aggregate following the instructions given on the worksheet, or use the formula in Attachment D.

6.4 Testing Frequency - The specific gravity of the aggregate may change during production. The specific gravity should be redetermined if the type or source of any of the aggregates being used in the mix is changed, or if the gradation of the mix changes enough to require a change in the plant mix formula.

7.0 TEMPERATURE CALIBRATION CHART

7.1 Find the weight of the pycnometer filled with water, using the test procedure below at six different temperatures, fairly uniformly spaced, between approximately 25°C and 45°C.

7.2 Test Procedure - Fill the pycnometer about 3/4 full of water, add two drops of wetting agent, and apply vacuum for approximately 10 minutes. Add water until only a small air space remains, shake and rotate the pycnometer to remove air bubbles, fill completely with water, insert plug, and wipe all water from the outside of the pycnometer. Weigh and immediately measure the temperature of the water.

7.3 Calibration Chart - On a piece of graph paper, plot all test results as shown in the example in Attachment C. Using a transparent plastic ruler, draw the straight line which comes closest to passing through all of the points. This graph is used to find the weight of the pycnometer filled with water at test temperature.

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GLR:b

Attachments

Attachment A

WEST VIRGINIA DIVISION
OF HIGHWAYS

MATERIALS CONTROL, SOIL AND TESTING DIVISION

REPORT NO.

ASPHALT CONTENT OF PAVING MIXTURES - PYCNOMETER METHOD

PROJECT NO.	DATE SAMPLED
TYPE MIX	PLANT
COARSE AGGREGATE	LOCATION
FINE AGGREGATE	TYPE BITUMEN

	TEST NUMBER								
1	Wt. Pyc. + Mix								
2	Wt. Pyc. Empty								
3	(1 - 2) Wt. Sample								
4	[1]Wt. Pyc. + Water								
5	(3 + 4) Total								
6	Wt. Pyc. + Sample + Water								
7	(5 - 6) Wt. Displaced Water								
8	Temp. ^{o}C @ (6)								
9	(3 ÷ 7)								
10	(A ÷ 9) - 1								
11	(D x 10) Percent AC								

A	Effective Specific Gravity - Aggregate	Tested By: _____ Date: _____
B	Specific Gravity - Asphalt	
C	A - B	Submitted By: _____ Date: _____
D	100B ÷ C	

[1] Weight of Pycnometer + Water at same temperature as at (6) when containing sample.

SPECIFIC GRAVITY OF AGGREGATE

Composition of Test Sample

Data

(a)	Wt. of aggregate used in sample	_____	gm
(b)	Wt. of asphalt used in sample	_____	gm
(c)	Wt. of mixing equipment before cleaning	_____	gm
(d)	Wt. of mixing equipment after cleaning	_____	gm

Calculations

(e)	Theoretical Wt. of sample (a + b)	_____	gm
(f)	Theoretical asphalt content $\left(\frac{100b}{e}\right)$	_____	%
(g)	Material left in mixing bowl (c - d)	_____	gm
(h)	Correction factor $\left(\frac{20g}{e}\right)$	_____	%
(i)	Corrected asphalt content (f - h)	_____	%

Specific Gravity

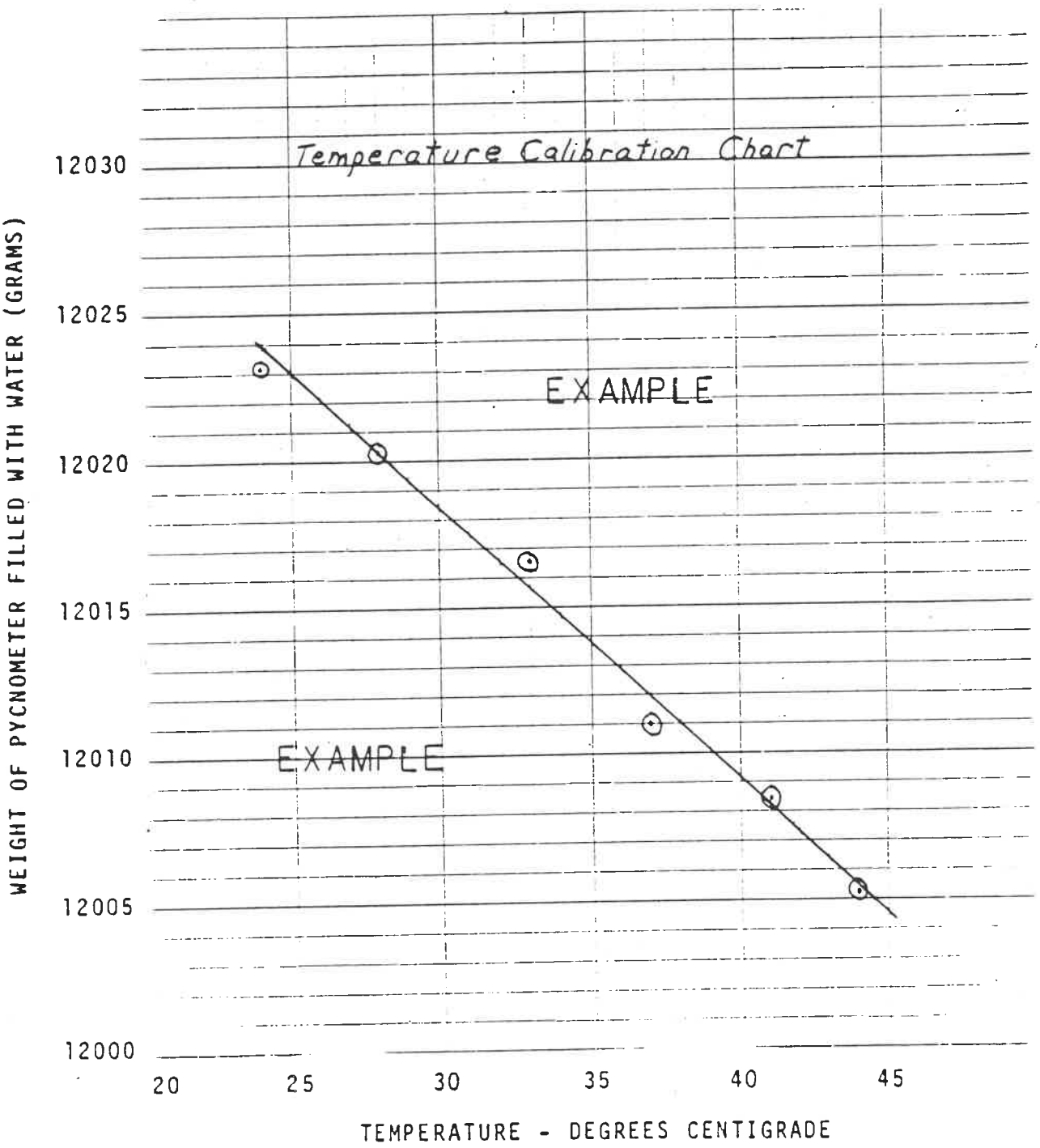
Data

(j)	Wt. of pycnometer, empty	_____	gm
(k)	Wt. pycnometer + sample	_____	gm
(m)	Wt. pycnometer + sample and water	_____	gm
(n)	Temperature of water	_____	°C
(p)	Wt. pycnometer filled with water at test temperature (from Temp. Calibration Chart) . .	_____	gm
(q)	Specific gravity of asphalt (from shipping invoice)	_____	

Calculations

(r)	Actual Wt. of sample (k - j)	_____	gm
(s)	Wt. pycnometer filled with water + sample Wt. (r + p)	_____	gm
(t)	Wt. displaced water (s - m)	_____	gm
(u)	Specific gravity of sample $\left(\frac{r}{t}\right)$	_____	
(v)	$\left(\frac{100}{u}\right)$	_____	
(w)	$\left(\frac{1}{q}\right)$	_____	
(x)	(v - w)	_____	
(y)	(100 - i)	_____	
(z)	Specific gravity of aggregate $\left(\frac{y}{x}\right)$	_____	

ATTACHMENT C



FORMULAS AND EXAMPLE CALCULATIONS

ASPHALT SPECIFIC GRAVITY

Information on Shipping Invoice	To Convert to Specific Gravity at 16°C
Specific gravity at loading temperature	Divide by temperature correction factor*
Degrees API	S.G. at 16°C = $\frac{141.5}{\text{Degrees API} + 131.5}$
Kilograms per liter at 16°C	Divide by 8.33
Kilograms per liter at loading temperature	Divide by 8.33 x temperature correction factor*

*Temperature correction factors may be obtained from Table A-1 of Asphalt Institute Publication MS-3 (Asphalt Plant Manual).

AGGREGATE SPECIFIC GRAVITY - SAMPLE CALCULATIONS FOR BLENDING TEST SAMPLES

Example 1 - Batch Plant (Hot Bin Samples)

Procedure

1. Sample aggregate from hot bins, and sample asphalt.
2. Select a test sample size (usually 6000 grams).
3. Find the batch weights being used to produce the mix.
4. Blend test sample in proportion to batch weights.

EXAMPLE PROBLEM

Sample Size . . . . . 6000 grams
Batch Weights: #1 Bin. . . . . 1135 kg
#2 Bin. . . . . 590 kg
Asphalt . . . . . 91 kg
Total . . . . . 1716 kg

Solution

The 6000 gram test sample should consist of:

From the #1 bin: $\frac{1135 \times 6000}{1716} = 3750$ grams

From the #2 bin: $\frac{590 \times 6000}{1716} = 1950$ grams

Asphalt: $\frac{91 \times 6000}{1716} = 300$ grams

Total Sample Weight = 3750 + 1950 + 300 = 6000 grams

Example 2 - Continuous Plant (Hot Bin Samples) or Batch Plant (Dry Batch Sample)

Procedure

1. If sampling from hot bins, sample from all bins at the same time and for the same number of revolutions. Combine into a single sample.

2. If sampling a dry batch, sample several portions from different locations and combine into one sample.

3. Select a sample size (usually 6000 grams).

4. Split or quarter the sample to approximately the weight needed. Do not attempt to quarter to an exact weight.

5. Calculate the weight of asphalt needed in the sample.

Example Problem

Blend a test sample containing 5.0% asphalt and weighing approximately 6000 grams.

Solution

The amount of aggregate needed is:

$$\frac{6000 \times (100 - 5.0)}{100} = 5700 \text{ grams}$$

The aggregate is quartered to approximately 5700 grams, since it is not practical to quarter to an exact weight. Assume that, after quartering, the aggregate actually weighs 5540 grams. The weight of asphalt needed to give an asphalt content of 5.0% is found from the formula:

$$\text{weight of asphalt} = \frac{\text{wt. of aggregate} \times \% \text{ asphalt}}{(100 - \% \text{ asphalt})}$$

$$= \frac{5540 \times 5.0}{100 - 5.0} = 292 \text{ grams}$$

SPECIFIC GRAVITY FORMULAS

Listed below are the formulas on which the pycnometer test method is based and which were used to devise the worksheets in Attachments A and B.

(1) $G_m = \frac{A}{A + D - E}$

(2) $P = \frac{100G_b}{G_a - G_b}\left[\frac{G_a}{G_m} - 1\right]$

(3) $G_a = \frac{100 - P}{\frac{100}{G_m} - \frac{P}{G_b}}$

(4) $P = \frac{100\ B}{B + S} - \frac{20\ W}{B + S}$

Where:

G_m = specific gravity of sample

A = weight of sample in air

D = weight of pycnometer filled with water at test temperature

E = weight of pycnometer filled with water and sample at test temperature

G_b = specific gravity of asphalt at test temperature.
NOTE: in the test procedure, for convenience, the gravity at 16°C is substituted for the gravity at test temperature. The difference between the two gravities is not significant.

G_a = specific gravity of aggregate

P = percent of asphalt by weight

B = weight of asphalt used in mixing sample of known asphalt content.

S = weight of aggregate used in mixing sample of known asphalt content.

W = weight of material left in mixing bowl after transferring sample of known asphalt content to pycnometer.