BITUMEN CONTENT AND PARTICLE SIZE DISTRIBUTION OF ASPHALT AND STABILISED SOIL: CENTRIFUGE METHOD

1 SCOPE

This method describes the procedure for determining the bitumen content and particle size distribution of hot mixed asphalt and stabilised soil by the centrifuge method.

2 SAFETY

This method does not attempt to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate occupational health and safety practices that meet statutory regulations.

3 REFERENCED METHODS

The following documents are referenced in this test method:

International Standards

ASTM D 2172 Standard Test Methods for the Quantitative Extraction of Bitumen from Bituminous Paving Materials

Main Roads Western Australia

WA 100.1 Sampling Procedures for Soil and Manufactured Granular Pavement Materials

WA 110.1 Soil and Granular Pavement Material Moisture Content: Convection Oven Method

WA 210.1 Particle Size Distribution of Aggregate

WA 701.1 Sampling and Storage of Asphalt

WA 705.1 Preparation of Asphalt for Testing

4 APPARATUS

(a) **Extraction apparatus** consisting of a bowl, a suitable design is shown in ASTM D 2172 and an apparatus in which the bowl may be revolved at controlled speeds up to 3 600 rpm. The apparatus shall be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion proof facilities and installed in a hood to provide ventilation.

NOTE: The volume of the extraction bowl will be sufficient to contain the minimum test portion depending upon the nominal aggregate size, as shown in Table 1, covered by 10 mm of solvent.

(b) **Filter ring** to fit the rim of the bowl.

(c) **Centrifuge** capable of revolving at approximately 3 000 to 3 500 rpm.

(d) **Centrifuge tubes** of at least 40 mL capacity.

(e) **Measuring cylinder** of 2 000 mL capacity graduated in 20 mL increments.

(f) **Glass measuring cylinder** of at least 100 mL capacity with light coloured graduations in 2 mL increments.

(g) A thermostatically controlled **oven** with good air ventilation capable of maintaining a temperature within the range of 105ºC to 110ºC.

NOTE: Depending on the solvent used during the extraction process the oven may require explosion proof facilities.

(h) **Balance** of at least 3.5 kg capacity readable to at least 0.01 g with a limit of performance (F) of not more than 0.05 g.

(i) A suitable **solvent**.

NOTE: A suitable solvent is one that is totally miscible with bitumen and does not leave a residue on the aggregate.

(j) **Desiccator** with indicator silica gel.

(k) **Oven trays**, spatula, and scoop.

(l) **Sample divider** (optional).

5 PROCEDURE

(a) Obtain a test sample taken in accordance with Test Method WA 701.1 for asphalt or WA 100.1 for bitumen stabilised soils.

(b) **For Bitumen Stabilised Soils** - Dry the stabilised soil to constant mass in an oven at a temperature of 105ºC to 110ºC, as defined in Test Method WA 110.1.

(c) Prepare a test portion in accordance with WA 705.1. The minimum mass of the test portion shall be related to the nominal size of the material and shall be in accordance with Table 1.

NOTE: Samples of asphalt may be heated to 105°C to facilitate sample division.

TABLE 1

Nominal Size	Minimum Test Portion Mass
< 10 mm	750 g
10 – 20 mm	1000 g
> 20 mm	2000 g

(d) Tare the extraction bowl on the balance.

(e) Place the test portion in the extraction bowl and record the mass (m_1) of the test portion to the nearest 0.01 g.

(f) Place the extraction bowl and test portion in the extraction apparatus then add sufficient solvent to cover the test portion by approximately 10 mm.

NOTE: The mixture may be gently stirred and agitated to break up aggregations then evenly distributed and allowed to stand to enhance the bitumen dissolving in the solvent.

(g) Dry a filter ring in an oven at a temperature of 105ºC to 110ºC for a minimum of 20 minutes. Allow the filter ring to cool in the desiccator.

(h) Determine and record the mass (m_2) of the dried and cooled filter ring to the nearest 0.01 g.

(i) Place the filter ring on the bowl in the extraction apparatus, then fit and clamp the cover tightly to the assembly.

(j) Place the 2 000 mL measuring cylinder under the solvent discharge spout.

(k) Start the machine revolving slowly, gradually increasing the speed to a maximum of 3 600 rpm or until solvent ceases to flow from the drainage spout. When the solvent discharge has ceased, allow the machine to stop and add an appropriate quantity of solvent to the extraction bowl.

(l) Repeat Procedure 5(k) two more times and then continue until the discharge is clear. The discharge shall be deemed to be clear when it is not darker than a light straw colour.

(m) Dismantle the extraction apparatus and carefully remove the filter ring avoiding loss of fines and inspect the test portion to verify that all the bitumen has been removed. Should there be visible aggregations within the test portion or indications of bitumen still present repeat Procedure 5(k) until all bitumen and aggregations have been removed.

(n) Record the volume (V_1) of extraction fluid in the 2 000 mL measuring cylinder to the nearest 10 mL.

(o) Remove the filter ring from the extraction apparatus taking care not to lose mineral matter and dry it in an oven at a temperature of 105ºC to 110ºC for a minimum of 20 minutes.

NOTE: It may be necessary to carefully brush off any larger material adhering to the filter ring ensuring any material dislodged is returned to the test portion.

(p) Place the filter ring in the desiccator and allow it to cool. Determine and record the mass (m_3) of the filter ring and mineral matter to the nearest 0.01 g.

(q) Determine and record the mass (m_4) of a clean dry oven dish. Remove the extraction bowl and contents from the extraction apparatus and carefully transfer all of the contents into the oven dish.

(r) Dry the contents to constant mass in an oven at a temperature of 105ºC to 110ºC. Constant mass shall be deemed to have been achieved when successive weighing of a test portion, after additional oven drying for intervals of not less than twenty minutes, differ by not more than 0.1 percent of the original mass of the test portion. Record the constant mass (m_5) to the nearest 0.01 g. If required retain the material for particle size distribution.

NOTE: Refer to note 4 (g).

(s) Dry two centrifuge tubes in an oven at a temperature of 105ºC to 110ºC for a minimum of 20 minutes.

(t) Remove the centrifuge tubes from the oven and allow them to cool in the desiccator. Determine and record the mass of each tube (m_6 and m_7 respectively) to the nearest 0.01g.

(u) Agitate the extraction fluid thoroughly and quickly obtain an approximate 80 mL aliquot of the extraction fluid into the 100 mL glass measuring cylinder.

NOTE: Mineral matter will rapidly settle in the extraction fluid. Hence agitation and sampling must be thorough and rapid if a representative aliquot is to be obtained. For volumes of 2000 mL or less pouring from one 2 litre measuring cylinder to another to thoroughly mix the extraction fluid has been found suitable. For volumes greater than 2 litres mixing in a capped 4 litre plastic bottle has been found suitable for solvents of low vapour pressure.

(v) Determine the volume (V_2) of the aliquot to the nearest 1 mL.

(w) Pour approximately half of the aliquot into each centrifuge tube.

NOTE: The mass of each centrifuge tube and contents must be approximately equal to prevent the centrifuge being out of balance whilst centrifuging.

(x) Place the centrifuge tubes and contents into the centrifuge then centrifuge at approximately 3 000 to 3 500 rpm for approximately 20 minutes.

(y) Remove the centrifuge tubes from the centrifuge and carefully decant the fluid from the tubes in one continuous motion. The sediment that has settled should not be decanted by this operation.

(z) Dry the centrifuge tubes and contents in an oven at a temperature of 105°C to 110°C for a minimum of 20 minutes.

(aa) Remove the centrifuge tubes and sediment from the oven and allow them to cool in the desiccator. Determine and record the mass of each tube (m_8 and m_9 respectively) to the nearest 0.01 g.

(bb) If required, determine the Particle Size Distribution of the material retained in the extraction bowl in accordance with WA 210.1 but including the mass of matter on the filter ring (m_{FR}) and the mass of matter in the extraction fluid (m_{EF}) in the mass of material passing the 0.075 mm sieve.

NOTE: The mass of the test portion is likely to be less than stated in Test Method WA 210.1 for the nominal size of the material.

6 CALCULATONS

(a) Calculate the mass of matter on the filter ring using the formula:

$$m_{FR} = m_3 - m_2$$

Where:

m_{FR} = mass of matter on filter ring in grams

m_2 = mass of filter ring in grams

m_3 = mass of filter ring and matter in grams

(b) Calculate the mass of matter in the extraction fluid using the formula:

$$m_{EF} = [(m_8 + m_9) - (m_6 + m_7)] \times \frac{V_1}{V_2}$$

Where:

m_{EF} = mass of matter in extraction fluid in grams

m_6 and m_7 = mass of centrifuge tubes in grams

m_8 and m_9 = mass of centrifuge tubes and sediment in grams

V_1 = volume of extraction fluid in mL

V_2 = volume of aliquot in mL

(c) Calculate the percentage bitumen using the formula:

$$Bit = \frac{m_1 - [(m_5 - m_4) + m_{FR} + m_{EF}]}{m_1} \times 100$$

Where:

Bit = bitumen content as a percentage

m_1 = mass of test portion in grams

m_4 = mass of oven dish in grams

m_5 = mass of the oven dish and contents in grams

m_{FR} = mass of matter on filter ring in grams

m_{EF} = mass of matter in extraction fluid in grams

(d) Calculate the percentage binder for crumbed rubber binder using the formula:

$$BitCR = Bit \times f$$

$BitCR$ = binder content with Crumb Rubber as a percentage

Bit = binder content as a percentage from 6(c)

f = f factor determined as part of mix design

7 REPORTING

Report the bitumen content and when required the crumb rubber binder content to the nearest 0.1%. If required, report the particle size distribution in accordance with the requirements of WA 210.1.

8 ISSUING AUTHORITY

Document Owner:
Pavements Manager

9 REVISION STATUS RECORD

Page No.	Section	Revision Description / Reference
3	6 & 7	Calculation and reporting for crumbed rubber asphalt added
All	All	General Formatting