Materials Testing Manual

Part 11: Chemical
Test Method Q603: Heat stability of bitumen anti-stripping agent

1 Source
This method was developed in-house using techniques evolved through internal departmental research investigations.

2 Scope
This method describes the procedure for determining the stability to heat treatment of a bitumen anti-stripping agent. It assesses the loss in effectiveness of the agent due to heating through measurement of binder aggregate adhesion using Test Method Q212C – Binder stripping value (immersion tray). The method is applicable to anti-stripping agents which are added directly to bituminous binders.

3 Apparatus

3.1 Test oven, thermostatically controlled at a temperature of 163 ± 1°C.
3.2 Preparation oven, thermostatically controlled at a temperature of 105 ± 5°C.
3.3 Sample containers, five metal containers of approximately 70 mm diameter and 250 mL volume, with one fitted with a tightly fitting level lid.
3.4 Sample lids, metal lids having a diameter of approximately 150 mm and a raised edge around the circumference (Note 8.1).
3.5 Water bath, maintained at a temperature of 40 ± 1°C.
3.6 Hotplate.
3.7 Balance, of suitable capacity, having a resolution of at least 0.01 g and with a limit of performance within the range of ± 0.05 g.

4 Materials

4.1 Aggregate, suitable aggregate of 20 mm nominal size (Note 8.2).
4.2 Bitumen, Class 170 bitumen complying with MRTS 17.
4.3 Cutter, bitumen cutter complying with MRTS 19.

5 Procedure
The procedure shall be as follows:

5.1 Aggregate preparation
Wash and dry at least 150 representative pieces of the dominant size fraction of the aggregate.

5.2 Calibration sample preparation and testing
Prepare four calibration samples covering a range of anti-stripping agent concentrations and test as follows (Note 8.3):
5.2.1 Heat at least 200 g of bitumen on the hotplate until pourable.
5.2.2 Select one of the anti-stripping agent concentrations and calculate the mass of agent required to produce this concentration in 100 to 140 g bitumen. Weigh the calculated mass into a sample container to the nearest 0.01 g.

5.2.3 Weigh the mass of bitumen required to produce the anti-stripping agent concentration into the sample container to the nearest 1 g.

5.2.4 Gently heat the contents of the sample container on the hotplate and stir vigorously for about 5 minutes.

5.2.5 Calculate the mass of cutter (to the nearest 0.1 g) required to produce a 7.5 percent by volume cutback bitumen as follows:

\[
M_c = \frac{7.5D_c M_B}{100D_B}
\]

where:
- \(M_c\) = mass of cutter (g)
- \(M_B\) = mass of bitumen (g)
- \(D_B\) = density of bitumen (t/m³)
- \(D_C\) = density of cutter (t/m³)

5.2.6 Add the mass of cutter calculated in Step 5.2.5 to the sample container with continuous stirring of the contents to achieve complete solution (Note 8.4).

5.2.7 Check the mass of the sample container and contents and if necessary add additional cutter to compensate for any cutter loss. Combine repeated additions of cutter with continuous stirring until the required mass of cutter in the sample (to the nearest 0.1 g) is obtained (Note 8.5).

5.2.8 Pour immediately 25.5 ± 2.0 g of the prepared sample from the sample container onto each of three lids. Spread the sample on each lid as quickly as possible to form a continuous and even film over the lid (Note 8.6).

5.2.9 Allow the three lids to cool to nearly room temperature and then immerse them to a depth of at least 25 mm in the water bath for at least 20 minutes.

5.2.10 Remove the lids from the water bath and then firmly press 10 pieces of aggregate into the binder of each lid. Return each lid to the water bath for a further 10 ± 1 minutes and then remove.

5.2.11 Select one of the lids and pull (by hand) each piece of aggregate in turn from the binder and examine for binder adhesion (Note 8.7).

5.2.12 Visually determine the quantity of binder retained on the underside of each piece of aggregate and score it on a scale of 0 to 10, with 10 being full cover. When all 10 pieces of aggregate have been examined, add the scores and record the total as percent.

5.2.13 Repeat Steps 5.2.11 and 5.2.12 for the remaining two lids.

5.2.14 Repeat Steps 5.2.2 to 5.2.13 for the three remaining anti-stripping agent concentrations.

5.3 Test sample preparation and testing

Prepare the test sample containing the specified anti-stripping agent concentration and test as follows (Note 8.8):

5.3.1 Heat at least 200 g of bitumen on the hotplate until pourable.
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5.3.2 Calculate the mass of agent required to produce the specified anti-stripping concentration in 100 to 140 g bitumen and weigh the calculated mass into a sample container to the nearest 0.01 g.

5.3.3 Weigh the mass of bitumen required to produce the anti-stripping agent concentration into the sample container to the nearest 1 g.

5.3.4 Gently heat the contents of the sample container on the hotplate and stir vigorously for about 5 minutes.

5.3.5 Allow the sample container to cool to room temperature and then fit its lid.

5.3.6 Place the sample container in the test oven for 360 ± 5 minutes.

5.3.7 Remove the sample container from the test oven and allow to cool to room temperature.

5.3.8 Calculate the mass of cutter (to the nearest 0.1 g) required to produce a 7.5 percent by volume cutback bitumen as follows:

\[ M_C = \frac{7.5D_C M_B}{100D_B} \]

where \( M_C \) = mass of cutter (g)

\( M_B \) = mass of bitumen (g)

\( D_B \) = density of bitumen (t/m³)

\( D_C \) = density of cutter (t/m³)

5.3.9 Remove the lid and gently heat the sample container on the hotplate until the contents are fluid.

5.3.10 Add the mass of cutter calculated in Step 5.3.8 to the sample container with continuous stirring of the contents to achieve complete solution (Note 8.4).

5.3.11 Check the mass of the sample container and contents and if necessary add additional cutter to compensate for any cutter loss. Combine repeated additions of cutter with continuous stirring until the required mass of cutter in the sample (to the nearest 0.1 g) is obtained (Note 8.5).

5.3.12 Repeat Steps 5.2.8 to 5.2.13.

6 Calculations

Calculations shall be as follows:

6.1 Stripping values

Calculate the stripping values for each set of three sample lid results as follows:

6.1.1 Record the total score obtained for each sample lid as the binder adhesion result.

6.1.2 Calculate the stripping result for each sample lid by subtraction of the binder adhesion result from 100.

6.1.3 If the difference between the stripping results of the three sample lids is less than 20 %, calculate the average of the three results and record the average as the stripping value to the nearest 1 %.
6.1.4 If the difference between the stripping results of the three sample lids exceeds 20 % but two of the results agree to within 10 %, calculate the average of these two results and record this average as the stripping value to the nearest 1 %.

6.1.5 If the difference between the stripping results of the three sample lids exceeds 20 % and no two results agree within 10 %, repeat the test.

6.2 Calibration samples

Plot the stripping values obtained for the four calibration samples against the respective anti-stripping agent concentrations and apply the best fit curve.

6.3 Test sample

6.3.1 Using the calibration curve obtained in Step 6.2, determine the effective anti-stripping agent concentration corresponding to the stripping value obtained for the test sample.

6.3.2 Calculate the heat stability of the test sample to the nearest 1 % as follows:

\[ S = \frac{100C_2}{C_1} \]

where

- \( S \) = heat stability (%)
- \( C_1 \) = specified anti-stripping agent concentration (%)
- \( C_2 \) = effective anti-stripping agent concentration (%) determined in Step 6.3.1.

7 Reporting

Report the heat stability of the sample to the nearest 1%.

8 Notes on method

8.1 Press-on lids from 4 litre capacity tins have been found suitable.

8.2 A suitable aggregate is one which provides a wide range of bitumen stripping values over the selected range of anti-stripping agent concentration.

8.3 The anti-stripping agent concentrations selected for the calibration samples should include 0% and that specified for the test sample. The remaining two concentrations should be selected between these two extremes at equidistant intervals.

8.4 If the contents have cooled so that thorough mixing is difficult, gentle warming of the contents is permitted.

8.5 It is important to reasonably ensure that the correct quantity of cutter is added to the binder. Varying quantities of cutter will lead to varying binder viscosities which have considerable influence on the stripping value obtained.

8.6 If the sample on the lid has cooled so that spreading of the sample to form a continuous film over the lid is difficult, minimal warming of the lid in the oven is permitted. However, under no circumstances shall a hotplate be used for this purpose.

8.7 Extract pieces of aggregate evenly with a direct upward pull without any twisting or shoving motion. Where only collar or edge adhesion has taken place, exercise care so that the ductile binder retained at the edge of the aggregate does not contact or overlay the underside of the aggregate before an assessment of stripping.
8.8 The specified anti-stripping agent concentration will normally be that concentration recommended for field use. However, where the heat stability of a number of anti-stripping agents is to be compared, the one specified concentration should be adopted for all such agents.
Test Method Q604: Pourability of a liquid bitumen anti-stripping agent

1 Source
This method was developed in-house using techniques evolved through internal departmental research investigations.

2 Scope
This method describes the procedure for determining the pourability of a liquid bitumen anti-stripping agent. It may be used to indicate how readily a liquid bitumen anti-stripping agent will pour from a supply container at low temperature.

3 Apparatus
The following apparatus is required:

3.1 Measuring cylinders, three unstoppered measuring cylinders of 25 mL capacity, graduated in 0.5 mL increments and conforming to ISO 4788.
3.2 Receivers, two crow receivers of 100 mL capacity conforming to BS658.
3.3 Water bath, maintained at a temperature of 5 ± 0.5°C.
3.4 Thermometer, a partial immersion liquid in glass thermometer with a range of at least 0-10°C, graduated in subdivisions of 0.5°C or less, with an uncertainty of no more than 0.2°C (for example, ASTM 90C).
3.5 Retort stand, boss head and clamp.
3.6 Travelling side arm stop (as illustrated in Figure 1 and detailed in Figure 2).
3.7 45° set-square or protractor.
3.8 Digital stopwatch.
3.9 Steel rule, of at least 300 mm length and accurate and readable to 1 mm.
3.10 Pipette, a pipette of 10 ml capacity.
3.11 Stirring rods, 2 glass stirring rods.

4 Procedure
The procedure shall be as follows:

4.1 Thoroughly mix the sample and add approximately 30 mL to each of two 25 mL measuring cylinders (that is, approximately 5 mL above the 25 mL mark).
4.2 Place the cylinders containing the samples into the water bath.
4.3 Stir the samples regularly with the stirring rods until they attain a temperature of 5 ± 0.5°C.
4.4 Assemble the clamp, travelling side arm stop, boss head, retort stand and remaining 25 mL measuring cylinder on a level bench as illustrated in Figure 1.
4.5 Adjust the cylinder location within the clamp so that the clamp is positioned near the base of the cylinder.
4.6 Loosen the grub screw and set the side of the measuring cylinder at 45° to the horizontal using the set-square or protractor. Retighten the grub screw.
4.7 Check the angle to ensure that the measuring cylinder is at 45° to the horizontal when stopped by the travelling side arm stop. The cylinder is now in the pour position.

4.8 Return the cylinder to the vertical position and then adjust the cylinder within the clamp so that the 20 mL mark on the cylinder is level with the upper edge of the clamp.

4.9 Return the cylinder to the pour position. Adjust the boss head, travelling sidearm stop and clamp so that the lower edge of the cylinder mouth is 255 ± 5 mm above the bench. This is the height at which the apparatus is to be set during testing.

4.10 Position a 100 mL crow receiver on the bench in a suitable location to receive sample draining from the measuring cylinder.

4.11 Return the cylinder to the vertical position and remove the cylinder.

4.12 Remove any excess liquid above the 25 mL mark on each measuring cylinder in the water bath using the pipette.

4.13 Remove one of the measuring cylinders from the bath, quickly dry and clamp into position so that the 20 mL mark on the cylinder is level with the upper edge of the clamp.

4.14 Rapidly move the cylinder to the pour position and immediately start the stopwatch.

4.15 At the 5 second time increment, rapidly return the measuring cylinder to the vertical position, and then remove it from the clamp.

4.16 Record the volume of sample in the receiver \( V_1 \) to the nearest 1 mL.

4.17 Position the second 100 mL crow receiver on the bench in a suitable location to receive sample draining from the measuring cylinder.

4.18 Repeat Steps 4.13 to 4.15 for the remaining measuring cylinder.

4.19 Record the volume of sample in the receiver \( V_2 \) to the nearest 1 mL.

4.20 If the difference between \( V_1 \) and \( V_2 \) exceeds 4 mL, repeat the test.

5 Calculations

Calculate the pourability of the sample as follows:

\[
P = \frac{2(V_1 + V_2)}{2}
\]

where

- \( P \) = pourability (%)
- \( V_1 \) = volume of sample drained in the first receiver (mL)
- \( V_2 \) = volume of sample drained in the second receiver (mL)

6 Reporting

Report the pourability of the sample to the nearest 1%.
Figure 1 - Pourability apparatus configuration
Figure 2 - Travelling side arm stop