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The Colour Indicator Test for the Presence of Adhesion Agent in Chip Seal Binders

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1 Scope

This procedure indicates the presence of amine type adhesion agents (antistripping agents) in bituminous binders used for the construction of chip seal surfacings. Its efficacy with other adhesion agent chemistry, such as phosphate esters and silanes has not been determined.

2 Referenced Documents

This test method is derived from Works Consultancy Central Laboratories Report 6-83/4, Section A 1.0 (September 1983). Contact WSP Research, Wellington, New Zealand.

3 Principle

Adhesion agent is extracted from a bituminous binder using propan-2-ol. A solution of bromophenol blue in ethanol/water is used to detect the presence of adhesion agent in propan-2-ol. A colour change from yellow to blue indicates that the adhesion agent has been detected.

4 Materials

(a) AR grade propan-2-ol (isopropyl alcohol, isopropanol see also clause 8.1.3 for checking acidity).
(b) AR grade ethanol
(c) Dilute (e.g. 0.025mol/L) hydrochloric acid
(d) Dilute (e.g. 0.025mol/L) sodium hydroxide
(e) Bromophenol blue indicator (see 8.1.2 for method of preparation)
(f) Distilled or deionised water
(g) Standard bitumen of the same grade as the doped binder being tested.
(h) Standard adhesion agent known to be detectable by the test method at a dose rate of 0.5pph (see clause 8.1.1).
(i) Doped bitumen, made from standard bitumen (as 4(g)) doped with 0.5pph standard adhesion agent (as 4(h)).

5 Apparatus

(a) Oven, controlled to 115° ± 10°C
(b) Temperature controlled bath set to 70° ± 5°C
(c) Clean 250mL borosilicate glass beakers
(d) Watch glass or similar covers for beakers
(e) 10mL and 25mL measuring cylinders
(f) Pasteur pipettes
(g) Balance accurate to 0.1g
(h) Laboratory storage bottle with screw cap, of at least 250mL capacity
(i) Spatula (minimum length 150mm)

6 Procedure

Use the following procedure:

(a) Check the propan-2-ol for neutrality (see clause 8.1.3).
(b) Rinse a clean 250mL beaker with propan-2-ol and allow to drain dry.
(c) Place the beaker on the balance and tare.
(d) Loosen, but do not remove, the lid on the container of the binder to be tested, and place in the oven set to 115° ± 10°C until fluid.

Note: Minimise the heating time in case the adhesion agent degrades. One to two hours should be ample time unless the sample volume is large.

(e) Stir the binder with the spatula to ensure homogeneity, taking care not to incorporate air bubbles. Pour 5g ± 1g of the binder into the bottom of the 250mL beaker.
(f) Allow the binder to cool to room temperature, and then add 25mL of propan-2-ol.
(g) Repeat steps (a) to (f) above for the standard bitumen (4 (g)) and the doped bitumen (4 (i)).
(h) Place the beakers containing the test binder, the standard bitumen (blank) and the doped bitumen (control) with propan-2-ol in the bath for 10 minutes. The surface of the propan-2-ol should be at or beneath the bath liquid level to ensure adequate heating.
(i) Remove the beakers from the bath. At this stage the propan-2-ol phase should have a yellow tinge.
(j) Add 2mL of bromophenol blue indicator from the 10mL cylinder. The propan-2-ol phase will change colour from yellow to blue if adhesion agent is detected.

    If the propan-2-ol in the test samples has not turned blue within 5 minutes, gently swirl the contents of the beaker. This will facilitate reaction of the indicator with low dosages of adhesion agent in the binder that would otherwise go undetected. A faint blue colour indicates that the adhesion agent is detected.

(k) Accept the result for the test material if the bromophenol indicator turns blue for the doped bitumen and remains yellow for the standard bitumen. Repeat the test with new standard and doped bitumen samples if the indicator does not show the correct colours as above.

7 Reporting

Report the adhesion agent as “detected” if the bromophenol indicator solution turns blue, or “not detected” if no colour change occurs. Note that a “not detected” result can mean that adhesion agent is present at levels below the limit of detection.

8 Notes

8.1.1 General

The method is capable of detecting most types of adhesion agent in the blended binder at a dose rate of 0.5pph or higher. For any given brand of adhesion agent previous testing may have demonstrated the effectiveness of the method. If such evidence is not available, a sample containing 0.5pph of the agent in a standard chip seal grade bitumen should be prepared, and the colour indicator test applied to it.

It is recommended that as a check on laboratory practice the test is always carried out with a blank prepared from undoped bitumen and another sample prepared with 0.5pph of an adhesion agent known to be detectable by the test.

The method will not detect the presence of very small dosages of adhesion agent, i.e. <0.4pph w/w or v/v.

8.1.2 Bromophenol Blue Solution

Bromophenol blue solution is prepared as follows:

(a) Place approximately 0.1g of solid bromophenol blue into 20mL of ethanol and warm gently with swirling until dissolved.
(b) Make up to 250mL with a mixture of 20% ethanol and 80% distilled or deionised water, and store in a screw cap bottle.

8.1.3 Propan-2-ol Acidity

The acidity of propan-2-ol should be checked before testing. Use the following procedure:

(a) Place slightly more than the quantity needed for planned testing into a clean beaker and add enough indicator to determine the colour.
(b) If the solution turns blue, neutralise it by adding dilute hydrochloric acid solution by Pasteur pipette until it turns a light yellow. If too much acid is added the solution will turn bright yellow. If this happens, add dilute sodium hydroxide solution until the solution turns blue and then re-neutralise

with the hydrochloric acid solution to a light yellow. Let the neutralised propan-2-ol stand for a further 10 minutes. If it reverts to blue repeat the procedure.