Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T542

Identification of tar or pitch in asphalt

NOVEMBER 2012

Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
Ed 1/Rev 0	All	New Issue – John Cunningham	D Dash	July 2001
Ed 2/Rev 0	All	Generally revised and reformatted. Title altered.	J Friedrich	Mar 2009
Ed 3/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T542 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T542

Identification of tar or pitch in asphalt

1. Scope

This test method sets out the procedure for indicating the presence of tar or pitch in asphalt.

2. General

(a) The method identifies the presence of phenol which is contained in tar or pitch

NOTE: Coal tar contains approximately 1% of phenol whereas bitumen does not contain any phenol.

(b) If the sample contains a mixture of tar asphalt and other non-tar material (e.g. bitumen asphalt), the method approaches its detection limit if there is less than 10% tar asphalt or less than 0.5% tar present

(c) OH&S requirements must be followed and include handling of chemicals and avoiding inhalation of dust from the sample. Perform the test in a fume cupboard. Wash hands thoroughly on completion of the test

(d) The test includes the following steps:

(i) The method extracts any weakly acidic phenols from the asphalt with dilute alkali

(ii) An aromatic amine reacts with nitrous acid to produce a diazo compound

(iii) When a diazo compound reacts with the extracted phenol it forms a strongly coloured diazo dye (usually red) which indicates the presence of tar in the original asphalt

3. Apparatus

(a) A balance of suitable capacity with a limit of performance of ±0.05 g

(b) Fume cupboard or hood

(c) Beakers of approximately 400 mL capacity

(d) Option: Erlenmeyer flasks 250 mL

(e) Wide test tubes

(f) Pasteur pipettes (eye droppers)

(g) Hot plate or steam bath

(h) Filter funnel

(i) Stirring rod

(j) Hammer, anvil and chisel

(k) Dish or bucket (for ice)

(l) Gloves (viton and disposable polythene)

(m) The following general purpose laboratory reagent (or better quality) are required for the test:

(i) Sodium hydroxide

(ii) Sulfuric acid

(iii) Para-nitroaniline, also called p-nitraniline

(iv) Sodium nitrite

(n) Expendable laboratory products:

(i) Filter paper, general purpose or No1 porosity

(ii) Litmus paper (red and blue)

(iii) Distilled or deionised water

(iv) Ice or chilled water

4. Preparation

4.1 Diluted solutions

(a) Prepare a 20% by mass sodium hydroxide solution and store in a suitable polythene container

Note: The solution keeps indefinitely.

(b) Prepare a 20% by mass sulfuric acid solution by carefully adding the acid to water

Note: The solution keeps indefinitely.

(c) Prepare a para-nitroaniline reagent freshly each day as follows:

(i) Dissolve 0.2 g para-nitroaniline in a mixture of 20 mL water and 5 mL of 20% sulfuric acid

(ii) Cool the solution by standing the beaker in a dish containing a slurry of crushed ice and water

(iii) After 10 minutes add 0.3 g sodium nitrite and gently stir

(iv) Discard any unused para-nitroaniline reagent after 24 hrs

4.2 Sample

(a) Where the sample is a core:

(i) Mark the extent of sub-samples to be tested

NOTE: Refer to the request document for the extent of testing (e.g. surface course only, assessed to a certain depth, specific layer, etc).

(ii) Trim the sample as required. Take care not to contaminate the samples

NOTE: Ensure that the hammer, chisel and anvil are clean.

(iii) Break up the sub-sample into pieces less than 20 mm and sufficient quantity to provide 100 to 150 g

NOTE: Use appropriate gloves and dust mask when handling the material.

(b) Where the sample is loose (e.g. millings) it is not essential to subsample by quartering. If more samples have been taken than the number of tests required, do not combine samples but select the samples to be tested by a random or deliberate procedure

5. Procedure

5.1 Test for Phenol

(a) Weigh 150 to 200g of the sub-sample and add to a beaker

(b) Add approximately 100 mL of water to the sub-sample in the beaker

(c) Warm the mixture to approximately 90°C in the fume cupboard

(d) Add a 20% solution of sodium hydroxide dropwise until the mixture turns red litmus paper a definite blue. Do not drop the litmus into the beaker

(e) Continue warming the mix for another 30 minutes. Gently agitate to break up any large lumps with the stirring rod

(f) Flute the filter paper and filter the mixture to collect 30 mL of the filtrate in a clean beaker or flask

(g) Add the 20% sulphuric acid dropwise with stirring until the filtrate turns blue litmus red. Decant or filter off the filtrate to remove any precipitate that forms at this stage

(h) Divide the filtrate into 2 portions in test tubes

(i) To one test tube add 2 drops of the p-nitroaniline reagent

(ii) To the other test tube add 20 drops of the same p-nitroaniline reagent

(i) Add a few drops of the sodium hydroxide solution to each test tube

(j) If either test tube solution is a definite red colour, this indicates phenol from the tar. If the solution is pink, this indicates only a small amount of phenol is present

(k) If the solution is not strong red, check the solution with red litmus. If the red litmus does not turn blue then add more drops of sodium hydroxide solution until the red litmus turns blue

(l) Confirm the colour of the solution with another experienced laboratory technician and both are to sign the worksheet

NOTE: The NATA signatory is preferred as the witness.

6. Calculations

There are no calculations.

7. Reporting

Include the following results in the report:

(a) Report the sample details

(b) Report against the sub-sample and its relative position if from a core:

(i) "Tar present" if a definite red colour was observed in Step 5.1(l)

(ii) "Tar absent" if a definite red colour was not observed in Step 5.1(l)

(c) Reference to this test method