

# NZTA T22: 2020

## QUANTITATIVE EXTRACTION OF BINDER FROM ASPHALT MIXES

# 1 SCOPE

This document provides a test method that determines the binder content of an asphalt mix. It is based on, and updates, widely used in-house test method developed by the (then) Ministry of Works Auckland testing laboratory.

This update is intended to standardise and modernise the test method and publish it so that further update and amendment can be done when necessary.

## 1.1 Safety Disclaimer

The use of this test method may involve hazardous materials, operations and equipment. This document does not purport to address all the safety issues associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

# 2 RELATED DOCUMENTS

- (a) American Society for Testing and Materials, Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates, ASTM C136.
- (b) American Society for Testing and Materials, Standard Practice for Sampling Bituminous Paving Mixtures, ASTM D979.
- (c) Standards New Zealand, Methods of Sampling and Testing Road Aggregates, NZS 4407.
- (d) Standards New Zealand, Safety in Laboratories – Part 8: Fume Cupboards AS/NZS 2243.8.

# 3 MATERIALS

- (a) Non-flammable chlorinated solvent such as 1-1-1 trichloroethane (methyl chloroform), dichloromethane (methylene chloride) or stabilised trichloroethene (trichloroethylene).

# 4 APPARATUS

The following apparatus is required:

- (a) A suitable wide-mouthed flask and stopper or lid made from a material resistant to chlorinated solvents and suitable for use on a hotplate. Stainless steel is recommended. The flask should be of a volume such that the test sample and solvent can be safely contained. A volume up to 7 litres has been found to be suitable for most asphalt mixes.
- (b) A ventilated drying oven capable of maintaining a temperature within  $\pm 5^\circ \text{C}$  of its set point.
- (c) A balance of appropriate capacity to weigh the flask, asphalt sample and solvent. A capacity of at least 10 kg and readability of 0.1 g is recommended. The balance shall have a limit of performance not exceeding  $\pm 0.5 \text{ g}$ .
- (d) A fume hood or cupboard complying with the requirements of AS/NZS 2243.8.
- (e) A balance of appropriate capacity to weigh the evaporation dish, lid, solvent and bitumen. A capacity of 200 g and readability of 0.1 mg is recommended. The balance shall have a limit of performance not exceeding  $\pm 0.5 \text{ mg}$ .
- (f) A hot plate of sufficient size and power such that the flask, sample and solvent can be heated to boiling.
- (g) A hot plate of sufficient size to carry at least two evaporation dishes and able to maintain a plate surface temperature between  $140^\circ \text{C}$  and  $160^\circ \text{C}$ .

**Note:** The two hot plates above may be the same device if they comply with the requirements this test method.

- (h) A centrifuge, capable of holding tubes of volume between nominally 20 mL and 50 mL, such that at least 1 g of bitumen is recovered from the evaporated solvent in step 5 (n) below. The centrifuge shall be capable of at least 3600 revolutions per minute.
- (i) Test sieves, 0.150 mm and 0.075 mm mesh size.
- (j) Metal evaporation dishes with lids. The dishes shall be nominally 45 mm high and diameter 70 mm. The dishes shall be of sturdy construction so that the bases remain flat when heated. Brass or stainless steel has been found to be suitable.
- (k) Heat resistant trays, spatulas, stirring rods and other equipment suitable for handling asphalt mix samples.

## 5 PROCEDURE

- (a) Obtain a representative sample of asphalt mix using the method of ASTM D979. Reduce the sample by quartering, or other means such as a sample divider (riffle box) so that a test sample complying with the minimum masses in Table 1 is obtained.
- (b) If the sample is received cold reheat it in an oven set to nominally 110° C until the sample is loose enough to be handled and a representative test sample obtained. Minimise binder hardening and moisture loss by limiting the reheating time to 4 hours maximum and covering the sample with a heat resistant cover or material such as aluminium foil.

**Note:** Asphalt mixes made using stiff binders, such as polymer modified bitumen, may need to be reheated at a higher temperature. Reheating temperatures and durations should be minimised to reduce damage to the binder by oxidation.

Table 1: Minimum test sample masses

| Nominal Maximum Aggregate Size (mm) | Minimum Test Sample Mass (kg) |
|-------------------------------------|-------------------------------|
| 7                                   | 1.0                           |
| 10                                  | 1.5                           |
| 14                                  | 2.0                           |
| 20                                  | 2.5                           |
| 28                                  | 3.0                           |

- (c) Weigh a test container capable of holding the test sample to the nearest 0.1g. Record this as M1.
  - (d) Place the test sample in the container and weigh the container, and test sample. Record this as M2.
  - (e) Place the container and test sample in the drying oven set to a temperature between 105° and 160° C and dry to constant mass. The sample shall be deemed to be dry when the change in mass of the sample does not exceed 0.1% of the mass of the sample after drying. Remove the container and sample from the oven and cover it with a lid or heat resistant material such as aluminium foil and allow it to cool such that the mass of the container and sample can be determined accurately. Weigh the container and dry asphalt mix sample and record this as M3.
- Note:** The determination of moisture can be omitted provided the laboratory has evidence that the asphalt mix has been produced dry.
- (f) Weigh the wide-mouthed flask and lid to the nearest 0.1 g and record this as M4. Transfer the dried asphalt test sample into the flask and weigh the flask, test sample and lid. Record this as M5.
  - (g) Transfer the flask to the fume cupboard and add solvent. Approximately 1 kg of solvent per kg of asphalt mix is adequate. Heat the flask and solvent on the hot plate until the solvent is boiling.
  - (h) Stir the boiling solvent and asphalt mix with a metal stirring rod. Remove the stirring rod and place the lid or stopper on the flask. Remove the flask from the hot plate and cool the flask and contents to room temperature.

**Note:** A water bath may be used to cool the flask.

- (i) Weigh the cooled flask, asphalt mix test sample and solvent to the nearest 0.1g and record this as M6. If a water bath was used to cool the flask carefully dry the flask with a cloth or paper towel prior to weighing.
- (j) Immediately after weighing the cool dry flask and contents, transfer the flask to the fume cupboard. Remove the lid or stopper and carefully stir the contents. Decant a portion of the solvent and bitumen solution through the 0.150 mm sieve into two centrifuge tubes until they are approximately two thirds full. Seal the centrifuge tubes. Place the tubes in the centrifuge and centrifuge them at 3600 revolutions per minute for 10 minutes. After 10 minutes allow the centrifuge to gradually slow and stop. Do not brake the centrifuge.

**Note:** The bitumen and solvent aliquot may be passed through the 0.150 mm sieve and temporarily stored in a sealed bottle prior to charging the centrifuge tubes if required.

- (k) Weigh two of the evaporating dishes and lids to the nearest 0.1 mg. Record this as M7.
- (l) Quickly decant a portion of the centrifuged bitumen and solvent solution into the evaporation dishes so that between 1 g and 2 g of bitumen remains after evaporation of the solvent and replace the lids. Quickly weigh the dishes and lids to the nearest 0.1 mg and record this as M8.

**Note:** Passing the solvent and bitumen solution through the 0.150 mm sieve in step (j) above can be carried out while charging the evaporation dishes instead if desired.

- (m) Set the hot plate so that the plate surface temperature is between 140° and 160° C. Place the evaporating dishes on the hot plate with the lids partly off to aid evaporation of the solvent. Leave the evaporating dishes on the hot plate for 30 ± 1 minutes.
- (n) Remove the evaporating dishes from the hot plate and allow them to cool to ambient temperature. Weigh the dishes, lids and residual bitumen to the nearest 0.1 mg and record as M9.
- (o) Wash the aggregate in solvent decanting the solvent over a 0.075 mm sieve until the solvent is a straw colour or clear. Dry the aggregate on a hot plate or an appropriately ventilated oven and cool to room temperature. Determine the particle size distribution using the methods of NZS 4407, ASTM C136 or equivalent method.

## 6 CALCULATIONS

Carry out the following calculations:

- (a) The moisture content of the asphalt mix =  $(M2 - M3)/(M3 - M1) \times 100$ .....(%)
- (b) Total mass of bitumen (B) =  $((M9 - M7) \times (M6 - M5))/(M8 - M9)$ .....(g)
- (c) Total mass of aggregate (A) =  $(M5 - M4) - B$ .....(g)
- (d) Calculate the bitumen content for the asphalt sample for each of the two evaporating dishes using the calculation below. If the bitumen content for the two evaporating dishes differs by more than 0.10% reject the test.  
Percentage of bitumen (T) =  $B \times 100/(M5 - M4)$ .....(%)

## 7 REPORTING

Report the following:

- (a) The bitumen content for the sample, as the average of the two evaporating dishes, to the nearest 0.1%.

## 8 PRECISION AND ACCURACY

The precision of this test method is generally assumed to be within  $\pm 0.2\%$ , and the accuracy to be within  $\pm 0.3\%$ , of the reported result. However, these assumed values require confirmation by an interlaboratory trial with this version of the test method.