

Australian/New Zealand Standard®

Methods of testing bitumen and related roadmaking products

Method 30: Recovery of residue from bituminous emulsion

1 SCOPE This Standard sets out a procedure specifically for the recovery of residue from bituminous emulsion for further testing.

NOTE: Where binder content is required, AS/NZS 2341.23 is preferred.

2 PRINCIPLE The water phase of a bitumen emulsion is evaporated from a weighed sample on a hotplate to recover the binder phase for further testing. The test is carried out under an inert atmosphere to minimize binder hardening during evaporation.

3 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

1160 Bituminous emulsions for construction and maintenance of pavements

AS/NZS

2341 Methods of testing bitumen and related roadmaking products

2341.23 Method 23: Determination of residue from evaporation

4 REAGENT A 2:1 mixture of industrial methylated spirits and water.

5 APPARATUS The following apparatus is required:

- (a) *Balance* A balance having a minimum capacity of 250 g, accurate to 0.3 g and readable to 0.1 g.
- (b) *Carbon dioxide gas bottle* A carbon dioxide gas bottle with pressure regulator, connected by flexible tubing through a 500 mL gas washing bottle to a nozzle of glass tubing having an internal diameter of about 6 mm. The nozzle shall have the delivery end bent through approximately 180° so that it may be hooked over the side of the metal container and extended down the inside about 50 mm.

NOTES:

- 1 The purpose of this apparatus is to direct the flow of gas down the inside of the container to form a non-oxidizing atmosphere without hindering the escape of volatile matter.
- 2 An inert gas, e.g. nitrogen, may be used.
- (c) *Electric hotplate* An electric hotplate with a variable temperature control.
- (d) *Metal containers* Metal containers, up to 1 L capacity. The container size and shape shall be such that material is not lost during evaporation. Containers having a diameter of 90 mm and depth 105 mm with double-crimped base and side seams have been found to be suitable.
- (e) *Shallow dish* A shallow dish to hold cooling water.

- (f) *Tongs or thermally insulated gloves* Tongs or thermally insulated gloves to hold the metal containers.
- (g) *Stirring thermometer* A stirring thermometer reading to 200°C, graduated in intervals of 1°C.

6 PROCEDURE The procedure shall be as follows:

- (a) Weigh container and thermometer to the nearest 0.1 g (*B*).
- (b) Tare the balance with the container and thermometer and weigh to the nearest 0.1 g, about 60 g of the thoroughly mixed sample (*m*).

NOTE: Where subsequent testing requires larger quantities of residue, a larger mass of emulsion may be used.

- (c) Add 25 mL of the reagent and place the assembly on the hotplate.
- (d) Connect the gas nozzle, adjust the rate of flow of gas at about 2 bubbles per second (when passed through gas washing bottle), and commence the evaporation process.
- (e) Adjust the temperature of the hotplate and, in order to control boiling, rock the container from side to side with tongs, while stirring.

NOTE: It may be advantageous to place a piece of wire gauze over the hotplate as an additional means of regulating the rate of heating, but the airspace caused by the raised rim of the base is usually adequate.

- (f) Remove container from the hotplate when frothing subsides and temperature rises to about 130°C. Cool to about 50°C by immersing the container in a shallow dish of water, maintaining the gas flow into the container.
- (g) Remove gas nozzle, dry the container, and weigh to the nearest 0.1 g.
- (h) Repeat evaporation with further additions of reagent until the loss in mass between successive evaporations is less than 0.2 g.

NOTE: The residue obtained may be used to determine viscosity and insoluble matter given in AS 1160. Some hydrocarbon solvents may not be completely removed even after repeated evaporations and thus affect the viscosity of the residue.

- (i) Record the final mass of container, stirrer and residue to the nearest 0.1 g (*A*).

7 CALCULATION Calculate the percentage of residue as follows:

$$\text{Residue (\%)} = \frac{100}{m} (A - B)$$

where

A = mass of metal container, stirring thermometer and residue, in grams

B = mass of metal container and stirring thermometer, in grams

m = mass of sample, in grams

8 REPORT The test report shall contain the following:

- (a) Percentage of residue to the nearest 0.1%.
- (b) Reference to this Australian/New Zealand Standard, i.e. AS/NZS 2341.30.

9 PRECISION The following criteria should be used for judging the acceptability of the results of this test:

- (a) *Repeatability* Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.4% of residue by mass.
- (b) *Reproducibility* The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 1.0% of residue by mass.