

Australian/New Zealand Standard™

AS/NZS 2341.21:2015

**Methods of testing bitumen and related
roadmaking products****Method 21: Sample preparation**

PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee CH-025, Bitumen and Related Products (for Roadmaking), to supersede AS/NZS 2341.21:1995.

Bitumen and related products are heat-sensitive materials. Excessive heating can permanently alter properties and test results.

The adoption of a uniform and practical sample preparation protocol as described in this Standard will improve the reproducibility of test results obtained for bitumen (including multigrade bitumen), bituminous emulsion and cutback bitumen samples. This protocol also applies to small samples of binder that are tested following a pre-treatment or extraction procedure.

The term 'informative' has been used in this Standard to define the application of the appendix to which it applies. An informative appendix is only for information and guidance and is not an integral part of the Standard.

METHOD

1 SCOPE

This Standard sets out sample preparation protocols applying to all samples of bitumen (including multigrade bitumen), bituminous emulsion and cutback bitumen that are to be tested in accordance with the test methods in the AS/NZS 2341 series (see AS/NZS 2341.0) and for all test methods specified in AS 2008, AS 1160 and AS 2157.

WARNING: THE USE OF THIS STANDARD MAY INVOLVE HAZARDOUS MATERIALS, OPERATIONS AND EQUIPMENT. THIS STANDARD DOES NOT PURPORT TO ADDRESS ALL OF THE SAFETY ISSUES ASSOCIATED WITH ITS USE. IT IS THE RESPONSIBILITY OF THE USER OF THIS STANDARD TO ESTABLISH APPROPRIATE SAFETY AND HEALTH PRACTICES, AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE.

2 APPLICATION

This protocol shall take precedence over any sample preparation related clauses contained within individual Standards. This Standard shall not be used to prepare hot polymer modified binders, or binder residues obtained from polymer modified bituminous emulsion.

3 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

- 1160 Bituminous emulsions for the construction and maintenance of pavements
- 2008 Bitumen for pavements
- 2157 Cutback bitumen
- 3568 Oils for reducing the viscosity of residual bitumen for pavements
- 2341 Methods of testing bitumen and related roadmaking products
- 2341.15 Method 15: Distillation of cutback bitumen

AS/NZS

- 2341 Methods of testing bitumen and related roadmaking products
- 2341.0 Part 0: General introduction and list of standards
- 2341.10 Method 10: Determination of the effect of heat and air on a moving film of bitumen (rolling thin film oven (RTFO) test)

ASTM

- D2872 Standard Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)

NZTA

- M/1 Specification for Rounding Bitumens

4 PRINCIPLE

This sample preparation protocol aims to ensure that cold samples, when reheated and tested, are representative of the product as sampled.

To achieve this, the original samples undergo minimal and controlled heating and adequate stirring.

NOTE: Typical temperatures at which different binders are sufficiently fluid to be stirred freely, and so can be split into sub-samples or poured into test equipment are listed in Appendix A.

The standard sample size described in AS 2008 and AS 2157 is 1 L and in AS 1160 is 2 L. Sample sizes smaller than 1 L and bulk samples of up to 20 L may also be supplied to laboratories.

This sample preparation protocol is used to split bulk samples into sub-samples and to prepare smaller samples for testing. Smaller samples include samples which are tested after a pre-treatment or extraction procedure.

5 APPARATUS

5.1 General items

The general items of apparatus are as follows:

- (a) *Containers*—with suitable capacity, such as metal tins and lids, or beakers (for stored sub-samples). Metal lids should not contain rubber seals as they can melt during heating and contaminate the sample. Bituminous emulsions should be stored in plastic containers or lined metal tins.
- (b) *Metal spatula*—with a flat metal blade, suitable for the containers used.
- (c) *Temperature probe*—a thermocouple or Pt₁₀₀ sensor used in conjunction with a readout device, with a readability and accuracy of at least 2°C or better. Alternatively, a thermometer may be used (e.g. a 200°C general purpose mercury in glass thermometer with a readability and accuracy of 2°C or better).

5.2 Hotplate method

The items of apparatus required if the hotplate method is used are as follows:

- (a) *Preheating oven*—capable of maintaining a temperature accuracy of at least 10°C or better at the desired temperature. The preheating oven may be fitted with a timer switch. The preheating oven is optional if a fan-assisted oven, as specified in Clause 5.3, is available.
- (b) *Hotplate*—a temperature controllable hotplate capable of maintaining a surface temperature within 10°C or better at the set temperature. If a temperature probe is used to monitor temperature it should be placed in firm contact with the hotplate surface.

NOTE: A power controllable hotplate may be used during heating of a 20 L container of bitumen for sub-sampling.

5.3 Oven method

Fan-assisted oven—an oven in which the air is circulated by a fan. The oven shall be capable of maintaining a temperature accuracy of 5°C or better, at the set temperature (it may also be used as the preheating oven).

5.4 High viscosity bituminous emulsions

Water bath (optional)—a water bath capable of maintaining a temperature accuracy of 5°C or better, at the set temperature, may be used as an alternative to the fan-assisted oven.

6 PROCEDURES

6.1 Testing of bitumen

6.1.1 General

WARNING: HOT BITUMEN CAN CAUSE SERIOUS BURNS.

Precautions to be taken when heating or handling bitumen include the following:

- (a) Wearing of appropriate personal protective equipment.
- (b) No ignition sources in close proximity.
- (c) Care when heating bitumen samples which contain water.

WARNING: HEATING BITUMEN CONTAMINATED WITH WATER PRESENTS ADDITIONAL HAZARDS.

Bitumen heated in the presence of small quantities of water may foam excessively and spatter or overflow the sample containers. Therefore, samples should always be checked for the presence of water while still cold by examining whether a layer of condensate is evident, either on the surface of the sample or on the underside of the container lid, or if the binder surface exhibits a pitted appearance. If rust is found on the inner surfaces of a sample container, it should be assumed that the sample contains water.

In such cases where water is found to be present, drain off as much as possible and dry the sample at room temperature or blow-dry with clean compressed air. Where moisture contamination has been detected or is suspected, the sample may be heated with extra care as follows:

- (i) Transfer the sample container onto a tray of sufficient capacity to capture any material which may overflow.
- (ii) Place the tray and container in the preheating or fan-assisted oven set at approximately 120°C. Position the lid off-centre on the sample container so that there is an air gap for water vapour to escape. Heat the sample until it is fluid enough to stir.

- (iii) At regular intervals not exceeding half-hourly, stir the sample with a circular and upward motion to release any generated steam. Mild foaming is to be expected. Place the lid off-centre on the sample container after returning it to the oven.
- (iv) Continue step (iii) until there is no obvious frothing or foaming, the sample volume remains constant and the binder appears homogeneous.

6.1.2 Observations

Check the material for any unusual or unexpected physical properties, such as extreme fluidity, elastic or rubbery behaviour, unusual odour, cutter oil odour or fume. If unusual behaviour is suspected refer to the client for advice before proceeding with further testing.

NOTE: Binders which contain cutter oil typically show low results in viscosity tests. If the presence of volatile oil is suspected, the sample may be tested for mass change by AS/NZS 2341.10 or ASTM D2872. Samples which contain volatile oils will show negative mass change results which will be typically lower than -0.6% .

6.1.3 Sampling of bitumen

The sample shall be obtained in accordance with AS 2008, or as specified by the customer.

NOTE: Samples taken from production, bulk storage, transport tankers or during transfer from or into these vessels should be treated as original samples.

6.1.4 Sub-sampling of bitumen

6.1.4.1 General

Representative sub-samples can be prepared using either a hotplate or oven method.

Loosen the lid of the sample container and remove any rubber seal in the lid. If the rubber seal is not removed it can melt during heating and contaminate the sample.

6.1.4.2 Hotplate method

If sub-samples are prepared using the hotplate method, the procedure shall be as follows:

- (a) Set the preheating oven to 100°C and place the bulk sample in the oven with the lid loose. Heat the sample for between 4 h and 10 h.

NOTE: Four hours is sufficient preheating time for a 1 L sample but a 20 L sample should be warmed for 8–10 h. A programmable timer can be used to allow preheating to occur overnight.

- (b) After preheating, remove the lid from the container and place the sample on the hotplate. Commence stirring with a spatula without entraining air, occasionally scraping the sides and bottom of the container.

- (c) Check the temperature of the sample from time to time with the thermometer or temperature probe to ensure that the temperature does not rise above 150°C . When the sample can be stirred freely and no lumps can be felt with the spatula, stir thoroughly for 30 s without entraining air. Remove the bulk sample from the hotplate. Pour the number of sub-samples required into containers of appropriate size (normally 1 L or less).

NOTE: In the case of M1000 binders the binder temperature during hotplate heating should not rise above 165°C .

6.1.4.3 Oven method

If sub-samples are prepared using the oven method, the procedure shall be as follows:

- (a) Set the fan-assisted oven to a temperature not exceeding 150°C .

NOTE: In the case of M1000 binders the oven can be set to a temperature not exceeding 165°C .