

AS 2341.30:2021



STANDARDS
Australia



Methods of testing bitumen and related roadmaking products

Method 30: Recovery of residue from bituminous emulsion



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- Australian Airports Association
- Australian Chamber of Commerce and Industry
- Australian Flexible Pavement Association
- Australian Institute of Petroleum
- Australian Road Research Board
- Austrroads
- AustStab
- Department of Transport, Vic
- Engineers Australia
- National Association of Testing Authorities Australia

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Method 30: Recovery of residue from bituminous emulsion

Originated as part of AS 1160–1981.
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Revised and redesignated as AS 2341.30:2021.

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Preface

This Standard was prepared by the Australian Members of Joint Standards Australia/Standards New Zealand Committee CH-025, Bitumen and Related Products (for Roadmaking), to supersede AS/NZS 2341.30:1996.

After consultation with stakeholders in both countries, Standards Australia and Standards New Zealand decided to develop this document as an Australian Standard rather than an Australian/New Zealand Standard.

The objective of this Standard is to specify procedures for the recovery of residue from a bituminous emulsion so that the residue can be subjected to further tests. A list of all parts in the AS (AS/NZS) 2341 series can be found in the Standards Australia online catalogue.

The major changes in this edition are as follows:

- (a) The apparatus has been updated to reflect current industry standard apparatus and practise.
- (b) The stirring thermometer previously used in the test has been replaced by a digital infrared thermometer.
- (c) The calculation of a residue from evaporation result for the bituminous emulsion sample is now optional.

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NOTES

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Methods of testing bitumen and related roadmaking products

Method 30: Recovery of residue from bituminous emulsion

1 Introduction

The water phase of a bituminous 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.

This method is applicable to bituminous emulsions used in the construction and maintenance of pavements (as specified in AS 1160).

This method may also be used to determine the residue from evaporation of a bituminous emulsion, if specified.

2 Scope

This document sets out a procedure for the recovery of the residue from a bituminous emulsion for further testing. It also provides a means by which the residue from evaporation of a bituminous emulsion can be determined.

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

3 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document.

NOTE Documents referenced for informative purposes are listed in the Bibliography.

AS 1160, *Bituminous emulsions for the construction and maintenance of pavements*

AS/NZS 2341.1, *Methods of testing bitumen and related roadmaking products, Part 1: Precision data — Definitions*

AS/NZS 2341.21, *Methods of testing bitumen and related roadmaking products, Method 21: Sample preparation*

4 Terms and definitions

For the purpose of this document, the terms and definitions in AS/NZS 2341.1 and the following apply.

4.1

may

indicates the existence of an option

4.2

shall

indicates that a statement is mandatory

4.3

should

indicates a recommendation

5 Reagent

A 2:1 by volume mixture of methylated spirits and water.

NOTE Domestic grade methylated spirits has been found to be suitable.

6 Apparatus

The following apparatus is required:

- (a) *Balance*, with a minimum capacity of 250 g, readable to at least 0.1 g, with a limit of performance not exceeding 0.3 g.
- (b) *Inert gas delivery system* consisting of a carbon dioxide gas bottle (food grade) with pressure regulator, connected by flexible tubing through a 500 mL glass gas washing bottle to a nozzle of glass tubing having an internal diameter of about 6 mm. The gas 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.

NOTE 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.

NOTE 2 Another inert gas, e.g. nitrogen (food grade), may be used.

- (c) *Hotplate*, with variable temperature control.
- (d) *Wire gauze* (optional).
- (e) *Container*, metal with an open top, up to 1 L capacity.

NOTE 3 A food can having a diameter of appropriately 75 mm and a depth of appropriately 105 mm has been found to be suitable.
- (f) *Shallow dish*, to hold cooling water.
- (g) *Tongs or thermally insulated gloves*, to hold the metal container.
- (h) *Stirrer*, suitable for the containers used, such as a metal spatula or glass rod.
- (i) *Digital infrared thermometer*, which is readable to at least 1 °C and accurate to at least ± 5 °C, and able to measure temperatures in the range between 20 and 200 °C.

7 Procedure

The procedure shall be as follows:

- (a) Prepare the bituminous emulsion sample for testing in accordance with AS/NZS 2341.21.
- (b) Place the stirrer into the container and weigh the container and stirrer on the balance (*A*).
- (c) Tare the balance with the container and stirrer and add about 60 g of the bituminous emulsion sample. Record the mass of bituminous emulsion added to the container (*M*).

NOTE 1 Where subsequent testing requires larger quantities of residue, a larger mass of emulsion may be used.
- (d) Add approximately 25 mL of the reagent and place the container on the hotplate.

- (e) Connect the inert gas delivery system to the container, adjust the rate of flow of gas at about 2 bubbles per second (when passed through the gas washing bottle), and commence the evaporation process.
- (f) Adjust the temperature of the hotplate and, in order to control boiling, rock the container from side to side using the tongs or thermally insulated gloves, while stirring the sample with the stirrer.

NOTE 2 It may be advantageous to place a piece of wire gauze over the hotplate as an additional means of regulating the rate of heating.

- (g) When frothing of the sample subsides, monitor the temperature of the sample using the digital infrared thermometer. Remove the container from the hotplate when the temperature of the sample is about 130 °C.
- (h) Place the container in a shallow dish of water while maintaining the gas flow into the container. Monitor the temperature of the sample using the digital infrared thermometer. Allow the sample to cool to about 50 °C.
- (i) Remove gas nozzle, dry the outside of the container, and weigh the container, stirrer and residue on the balance.
- (j) Repeat the evaporation with further additions of reagent until the loss in mass between successive evaporations is less than 0.2 g. Take the final mass recorded to be the mass of container, stirrer and residue after evaporation (B).

NOTE 3 Some hydrocarbon solvents may not be completely removed even after repeated evaporations. This may affect the test results obtained for the residue.

8 Calculation of residue from evaporation (if required)

If it is required to calculate the residue from evaporation of the bituminous emulsion, the following equation shall be used:

$$\text{Residue from evaporation (\% mass)} = \frac{100}{M} (B - A)$$

where

- A = mass of container and stirrer (g)
- B = mass of container, stirrer and residue after the loss in mass between successive evaporations is less than 0.2 g (g)
- M = mass of bituminous emulsion sample added to the container (g)

9 Report

If the residue obtained in this test is subjected to further testing, reference this document, i.e. AS 2341.30, and the test methods used to characterize the residue.

If the method described in this document is used to determine a residue from evaporation test result, then the test report shall contain the following:

- (a) Residue from evaporation to the nearest 0.1 % mass.
- (b) Reference to this document, i.e. AS 2341.30.

10 Precision

The following criteria should be used for judging the acceptability of residue from evaporation results obtained using this test (95 % probability):

- (a) *Repeatability* Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.4 % 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 % mass.

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