

AUSTROADS TEST METHOD ATM-104

Storage Stability of Binders

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# Preface

This test method was prepared by the Bituminous Surfacings Technical Group (BSTG) on behalf of the Austroads Pavements Task Force (PTF). Representatives of Austroads, the National Transport Research Organisation (NTRO) and the Australian Flexible Pavement Association (AfPA) were involved in developing and reviewing this test method.

# Scope

Polymer modified binders (PMBs) are prepared from mixtures of polymers, additives and bitumen selected to achieve their required properties. Some of these binder systems can segregate, leading to variations in measured properties and in some instances significant reductions in desired field performance.

This test method describes a procedure for determining the propensity of components in binders (e.g. polymers) to segregate during prolonged storage at high temperatures. It provides a means by which the storage stability of a binder can be assessed in terms of the segregation of different binder components during hot storage.

The method involves placing a binder sample into an oven at an elevated temperature and, after a prescribed time, measuring the properties (typically softening point) of the top, middle and bottom thirds of the sample. This method is applicable for use on PMBs which contain one polymer type (e.g. styrene‑butadiene‑styrene (SBS) polymer) and hybrid PMBs which contain multiple polymer types (e.g. SBS polymer and crumb rubber).

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

# References

The following normative documents are referred to in this method and are required to perform the test:

| **Austroads Test Method** |
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| ATM 102 | Protocol for handling modified binders in preparation for laboratory testing |
| **Australian Standard** |
| AS 2341.18 | Methods of testing bitumen and related roadmaking products: determination of softening point (ring and ball method) |
| **Australian/New Zealand Standard** |
| AS/NZS 2341.21 | Methods of testing bitumen and related roadmaking products: sample preparation |

The following documents are informative and are not required to perform the test:

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| **Austroads Test Method** |
| ATM 192 | Characterisation of the viscosity of Reclaimed Asphalt Pavement (RAP) binder using the Dynamic Shear Rheometer (DSR) |
| **Australian Standard** |
| AS 2341.12 | Methods of testing bitumen and related roadmaking products: determination of penetration |
| **AASHTO Standard** |
| AASHTO T315 | Standard method of test for determining the rheological properties of asphalt binder using a Dynamic Shear Rheometer (DSR) |
| **ASTM Standard** |
| ASTM D36/D36M | Standard test method for softening point of bitumen (ring‑and‑ball apparatus) |
| **European Committee for Standardization Standard** |
| EN 13399 | Bitumen and bituminous binders – determination of storage stability of modified bitumen |

# Equipment

The following items of equipment are required:

1. Tube – made of thin aluminium which has unvarnished internal surfaces. The tube shall be closed at one end (bottom end), have a minimum height of 160 mm, and a diameter between 25 mm and 40 mm. A photograph of a representative tube (typically referred to as a ‘toothpaste tube’) is shown in Figure 4.1. Tubes meeting the requirements of EN 13399 are suitable for use.
2. Tube holder – a metal frame or metal support which allows the tube to visibly stand vertically.

**Note 1:** Suitable tube holders include:

* 1. a wire frame or metal rack of suitable size for the tube
	2. a metal tin with a metal lid where a circular hole of suitable size has been cut in the metal lid as shown in Figure 4.2.
1. Calliper or ruler – capable of measuring a length up to at least 200 mm, readable to at least 1 mm.
2. Indentation tool – a tool that can be used to place a small indentation mark on the tube. The flat edge of a spatula or the blade of a screwdriver have been found to be suitable.
3. Heat‑resistant gloves, forceps or pliers – for closing the tube filled with the sample.
4. Fan‑assisted oven – an oven in which the air is circulated by a fan. The oven shall be calibrated and validated and capable of maintaining a temperature of 180 ± 2 °C.
5. Heating device – a device for heating the cutting tool (e.g. a gas flame, hotplate or hot air gun).
6. Cutting tool – a sharp heat‑resistant cutting implement (e.g. a sharp knife).
7. Subsample tins – metal tins with lids of suitable size for the top, middle and bottom subsamples that are produced during the test.
8. Softening point apparatus – as described in AS 2341.18.
9. Fridge/freezer (optional) – for sample cooling. A domestic fridge/freezer is satisfactory.

Figure 4.1: Photograph of a representative tube



Figure 4.2: Photograph of a representative tube holder



# Sample Preparation

PMB samples shall be prepared for testing in accordance with ATM 102. Bitumen samples shall be prepared for testing in accordance with AS/NZS 2341.21.

# Procedure

## Filling the Tube

1. Make a small indentation mark on the tube with the indentation tool at the filling height. The filling height shall be 100 mm to 120 mm above the base of the cylindrical part of the tube as measured with the calliper/ruler. The indentation mark shall be visible on the inside of the tube and not create a hole in the tube. Figure 6.1 shows a representative example of an indentation mark and provides an illustration of the filling height.
2. Make marks on the tube at one‑third (⅓) and two‑thirds (⅔) of the filling height, as shown in Figure 6.1. Use the calliper/ruler to measure the distances corresponding to one‑third and two‑thirds of the filling height.
3. Ensure that the tube is visibly vertical. Pour the sample into the tube to the filling height indentation mark, taking care to avoid the incorporation of air bubbles.
4. After the sample is poured, press together the open end of the tube that is close to the top of the binder surface using heat‑resistant gloves or forceps/pliers. Press the remaining upper part of the tube together using heat‑resistant gloves or forceps/pliers so that no air bubbles are present in the top of the tube. This can be performed by progressively closing the tube from the binder surface to the top of the tube.
5. Tightly fold the top of the tube at least two times, using the heat‑resistant gloves or forceps/pilers, to seal the tube from air. An example of a folded tube is shown in Figure 4.2.

Figure 6.1: Photograph of a representative tube



## Hot Storage

1. Place the sealed tube in the tube holder so that the sealed nozzle is the lowest part of the tube. Ensure that the plastic cap (if supplied with the tube) is removed.

**Note 2:** If the plastic cap is not removed from the tube it may melt during hot storage.

1. Immediately after pouring and sealing the tube, place the sealed tube and tube holder in an oven set to 180 °C, taking into account the temperature correction of the oven after calibration/validation. Ensure that the tube appears visually vertical in the tube holder. An example of a tube placed in a tube holder is shown in Figure 4.2.

**Note 3:** The test result can be affected if the sample is allowed to cool down before hot storage.

1. Heat the tube and tube holder in the oven for 72 ± 1 hours. Ensure the tube stays visually vertical during hot storage.

## Producing Subsamples for Testing

1. Carefully remove the tube and tube holder from the oven after the required heating time. Ensure that the sample is minimally disturbed as it is removed from the oven.
2. Allow the tube to cool to room temperature in the tube holder. Ensure that the tube remains visibly vertical during cooling.

**Note 4:** To facilitate cutting of the tube, the sample may be cooled to below ambient temperature using a fridge/freezer. However, the sample should always be allowed to cool slowly to ambient temperature before any further cooling is conducted.

1. When the tube has cooled to room temperature (or below room temperature if the fridge/freezer is used), place the tube horizontally on a hard, flat and clean surface.
2. Cut the tube into three equal parts at the ⅓ and ⅔ filling height marks with the heated cutting tool to yield subsamples which represent the top, middle and bottom thirds of the sample when it was present in the oven. The cutting tool shall be heated using the heating device before cutting.
3. Mark three subsample tins with labels which differentiate the top, middle and bottom subsamples (e.g. label the different tins, ‘Top’, ‘Mid’ and ‘Bot’, respectively).
4. Place the top, middle and bottom subsamples separately into their respective tins. Place the lids loosely on the tins.
5. Prepare (i.e. heat) PMB samples for testing in accordance with ATM 102. Prepare (i.e. heat) bitumen samples for testing in accordance with AS/NZS 2341.21. Remove the metal tube from each subsample tin when the binder has flowed out of the metal tube.

## Subsample Testing

1. Test the top, middle and bottom subsamples for softening point in accordance with AS 2341.18.

**Note 5:** If the bath liquid to be used in softening point tests is not known, the top, middle and bottom subsamples may each be poured into four softening point rings. If the initial softening point result obtained for a particular subsample indicates that the bath liquid (e.g. water) was not appropriate for the test, the second set of two softening point rings may be used to conduct the test in the alternative bath liquid (e.g. glycerol).

**Note 6:** Thetop, middle and bottom subsamples may be subjected to tests other than softening point using AS 2341.18 if agreed with the customer. Examples of other tests include penetration at 25 °C (using AS 2341.12), Dynamic Shear Rheometer (DSR) complex viscosity at 60 °C (using ATM 192), softening point (using ASTM D36/D36M) or DSR dynamic shear modulus or phase angle (using AASHTO T315). Tests other than those listed may also be used if agreed with the customer.

# Calculation

Calculate the storage stability result for the binder using Equation 1.

|  |  |
| --- | --- |
| $$SS = \left|T\_{t}\right. - \left.T\_{m}\right| + \left|T\_{b}\right. - \left.T\_{m}\right|$$ | 1 |
| where |  |  |  |
| $$SS$$ | = | storage stability (°C) |  |
| $$T\_{t}$$ | = | softening point of top subsample (°C) |  |
| $$T\_{m}$$ | = | softening point of middle subsample (°C) |  |
| $$T\_{b}$$ | = | softening point of bottom subsample (°C) |  |
| $$\left|T\_{t}\right. - \left.T\_{m}\right|$$ | = | absolute value of (Tt − Tm) (°C) |  |
| $$\left|T\_{b}\right. - \left.T\_{m}\right|$$ | = | absolute value of (Tb − Tm) (°C) |  |

**Note 7:** If tests other than softening point are used to characterise the properties of a sample, Tt, Tm and Tb in Equation 1 can be taken to be the test properties obtained for the top, middle and bottom thirds of a sample, respectively, after hot storage. SS in Equation 1 will then be expressed in the units of the chosen test property.

# Test Report

The following shall be reported:

1. Storage stability test result. Storage stability results shall be reported to the nearest 0.5 °C if softening point tests are used to characterise the sample.
2. Test results obtained for the top, middle and bottom subsamples. If softening point tests are used to characterise the sample and the AS 2341.18 method is used, the following information shall be reported:
	1. Softening points of the top, middle and bottom subsamples using the reporting requirements of AS 2341.18
	2. The bath liquid used to perform softening point tests on the top, middle and bottom subsamples.
3. Oven storage temperature (°C).
4. Oven storage time (hours).
5. Any deviations, by agreement or otherwise, from the specified procedure.

# Precision

1. The criteria given in AS 2341.18 should be observed for each of the duplicate softening point determinations. No precision data exist for the measurement of storage stability.

Amendment Record

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| **Amendment no.** | **Clauses amended** | Action | Date |
| – | New test method | New | January 2025 |

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| --- | --- |
| **Key** |  |
| Format | Change in format |
| Substitution | Old clause removed and replaced with new clause |
| New | Insertion of new clause |
| Removed | Old clauses removed |