

AUSTROADS TEST METHOD ATM 191 [AGPT/T191]

Extractions of Bituminous Binder from Asphalt

Commentary

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**Preface**

This test method was prepared by the Bituminous Surfacings Working Group (BSWG) and the Asphalt Research Working Group (ARWG) acting on behalf of the Austroads Pavements Task Force (PTF). Representatives of Austroads, ARRB Group and the Australian Asphalt Pavement Association (AAPA) have been involved in the development and review of this test method.

**Foreword**

This method is based on ARRB method M07. The objective of the method is to provide a sample of binder representative of its in-service state primarily in terms of measured viscosity. Since the method is based on a solvent extraction step followed by thin film solvent removal, the effectiveness of the procedure will depend on achieving complete removal of the solvent while maintaining minimal oxidative hardening in the extracted binder.

**Scope**

This method has been developed for extracting bituminous binders from bituminous mixtures i.e. asphalt field samples, loose plant mix or reclaimed asphalt pavement.

**Further Development**

This test method is under development and some changes in the test parameters may occur in the future.

**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 all of the safety problems associated with its use. It is the responsibility of the user of this Austroads test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

# References

The following documents are referred to in this method:

| **Austroads Test Methods** | |
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| ATM-192 | Characterisation of the Viscosity of Reclaimed Asphalt Pavement (RAP) Binder Using the Dynamic Shear Rheometer (DSR). |
| **Australian/New Zealand Standards** | |
| AS/NZS 2341.5 | Methods of testing bitumen and related roadmaking products, Method 5: Determination of apparent viscosity by ‘Shell’ sliding plate micro-viscometer. |
| AS/NZS 2341.10 | Methods of testing bitumen and related roadmaking products, Method 10: Determination of the effect of heat and air on a moving film of bitumen (rolling thin film oven (RTFO) test). |
| AS/NZS 2341.12 | Methods of testing bitumen and related roadmaking products, Method 12: Determination of penetration. |
| AS/NZS 2341.13 | Methods of testing bitumen and related roadmaking products, Method 13: Long-term exposure to heat and air. |
| AS/NZS 2243.8 | Safety in laboratories, Part 8: Fume cupboards. |
| **ASTM International** | |
| ASTM D482 | Standard Test Method for Ash from Petroleum Products |
| ASTM D2872 | Standard Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-film Oven Test) |

# Materials

1. Carbon Dioxide (may be substituted with Nitrogen gas in this test method).
2. Toluene.

*Note: As toluene vapour is flammable and toxic all working areas should be adequately ventilated and free from sources of ignition.*

# Equipment

1. Heating oven – thermostatically controlled to operate at a temperature of 100°C ± 5°C.
2. Rolling Thin Film Oven (RTFO) – as specified in AS/NZS 2341.13, including the additional safety requirements to ovens used for the evaporation of toluene specified in the standard. The oven shall be thermostatically controlled to operate at a temperature of 100°C ± 5°C.
3. Centrifuge – with a speed capacity of at least 3000 RPM.

*Note: If there is any doubt as to the suitability of the centrifuging technique, test approximately 2 g of the recovered binder by ASTM D482 for ash content. The ash content should be below 0.5%.*

1. Metal or glass gas tight centrifuge tubes – of suitable capacity for the centrifuge.
2. Balance – of at least 1000 g capacity, with a limit of performance not exceeding 0.1 g.
3. RTFO bottles – as specified in AS/NZS 2341.10 or ASTM D2872.
4. RTFO bottle scraper – suitable to remove extracted bitumen from inside of RTFO bottles.
5. Funnel – made of metal, fitted with a 75 µm mesh.
6. Timer – suitable timing device readable and accurate to 1 minute, or better.
7. Sample containers – such as glass beakers of suitable capacity; i.e. 250 mL beaker.
8. Sample pans – such as steel trays of suitable capacity, i.e. 2 L stainless steel tray.
9. Brush – sieve or paint brush suitable for removing fine material from asphalt samples.
10. Metal spatula – with a flat metal blade, of suitable size for the containers used.
11. Fume Cupboard – compliant with AS/NZS 2243.8.
12. Dish – Teflon or metal dish of suitable size.
13. Pipette (optional) – of suitable volume to decant centrifuged binder solution.
14. Filter Paper (optional) – Whatman No 44 or equivalent.

# Procedures

## Asphalt Sample Preparation

1. Place the asphalt sample into a labelled clean sample pan and put into the heating oven at 100°C ± 5°C for about 10 minutes to soften the sample.

*Note: If the sample is large additional heating may be required to soften the sample. If extra heating is required do not exceed 60 minutes of heating time. If asphalt field samples are comprised of more than one layer, appropriate action should be taken to separate the layers; i.e. saw cutting.*

1. Break up the sample with the spatula.
2. Divide the asphalt sample by quartering until about 50 g is obtained.

*Note: If a second sample is required repeat step 3.*

## Binder Extraction

1. Pour toluene into the sample container until the level is about 25 mm above the level of the asphalt sample.

*Note: Toluene vapour is flammable and toxic. All operations using toluene should be completed in the fume cupboard.*

1. Cover the sample container to slow the toluene evaporation and leave the sample in solution for about 1 hour.
2. Decant the bitumen solution into a clean sample container by passing it through the funnel.
3. Pour an additional amount of toluene into the sample container containing the asphalt sample until the level is about 25 mm above the level of the test portion.
4. Cover the sample container and leave the sample in solution for a further 15 minutes.
5. Decant the second portion of the bitumen solution through the funnel into the sample container used in step c.
6. Transfer the appropriate amount of bitumen solution to the centrifuge tubes ensuring that the tubes are of the same approximate weight typically within 0.5 g is sufficient.
7. Secure the lids to the centrifuge tubes and place into the centrifuge, ensuring that the tubes are counterbalanced.
8. Begin centrifuging the binder solution by slowly increasing the speed to approximately 3000 RPM and run for 15 minutes.
9. Decant the bitumen solution into a labelled sample container taking care not to transfer any of the fine material separated from the centrifuging process.

*Note: To minimize the filler being transferred into the sample container a pipette and/or 3 µm filter paper can be used.*

1. Decant the bitumen solution into four labelled RTFO bottles ensuring not to exceed 25 mL per bottle.
2. Insert the four RTFO bottles into the cold RTFO.

*Note: some RTFO ovens may be fitted with an internal carriage cabinet that aids in the concentration of carbon dioxide in and around the bottles. If the RTFO oven is fitted with a carriage cabinet it should be closed at this step.*

1. Fill the carriage with empty bottles where necessary.

*Note: always ensure that the carriage is full.*

1. Connect the Carbon Dioxide to the inlet pipe of the RTFO oven.
2. Turn on the Carbon Dioxide.

*Note: the flow of CO2 into the oven shall be 4 Litres per minute ± 0.5 L/minute.*

1. Turn on the RTFO oven (including the carriage rotation).
2. After the oven has reached the set temperature of 100°C ± 5°C treat sample for a further 45 minutes ± 5 minutes.
3. After the 45 minutes remove the labelled RTFO bottles containing residual bitumen from the oven.
4. Using the RTFO bottle scraper remove the residual bitumen from the inside of the bottles and collect in a dish.

*Note: This provides sufficient sample to characterise the viscosity properties of binder using the Dynamic Shear Rheometer (ATM 192).*

1. When multiple binder recoveries are to be performed repeat steps a to s and ensure the entire sample is collected in a single dish.

*Note: this allows for testing of other binder properties that require larger volumes of binder e.g. Penetration AS 2341.12.*

1. Homogenise the entire sample by gentle stirring with the spatula at a temperature not exceeding 100°C for a period no longer than 5 minutes ± 1 minute.

*Note: temperature to be checked regularly using suitable device, such as temperature gun.*

1. Cover the dish to ensure that the sample is not contaminated.

# Information to be Reported

Where binder property determinations are made from extracted samples reference to this Austroads method is required i.e. ATM .

# Amendment Record

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| Substitution | Old clause removed and replaced with new clause |
| New | Insertion of new clause |
| Removed | Old clauses removed |