Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T522

Penetration and properties of binder recovered from asphalt

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Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
Ed 1/Rev 0	All	Reformatted and Revision Summary Added. Safety notes altered/ 4. altered./ Note 5 Added/ Note 3 altered	D Dash	Jan 2000
Ed 2/Rev 0	All	New issue. Title changed. The method has been expanded to include penetration and viscosity testing.	J Friedrich	January 2010
Ed 3/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T522 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T522

Penetration and properties of binder recovered from asphalt

1. Scope

This test method sets out the procedure for recovering a sample of bituminous binder from an asphaltic concrete mix and carrying out subsequent penetration and viscosity testing.

NOTE: The Test is derived from and is complimentary with method ARRB 6 (ARR 66).

2. General

(a) The method uses a sample solution or prepares a solution from an asphalt sample to:

(i) Recover about 21 g binder from the sample solution

NOTE: Where the total amount of recovered binder is less than 6 g, an alternative test, such as ARR 66, should be used.

(ii) Test the penetration of the recovered binder

(iii) Carry out the specified testing on the recovered binder. The options for testing viscosity and other properties are listed in Table 1

(b) Most PMBs are recovered but the interpretation of the results requires care and appropriate control binders

(c) If tar is present, not all the pitch component will be recovered and the reported viscosities will be low

(d) The following documents are referred to in this Test Method:

(i) T521 Polymer Content of Polymer Modified Binders by Infra-red Spectrophotometry

(ii) AS/NZS 2341 Methods of testing bitumen and related road making products

- AS 2341.2 Determination of dynamic (coefficient of shear) viscosity by flow through a capillary tube
- AS/NZS 2341.5 Determination of apparent viscosity by 'Shell' sliding plate micro-viscometer
- AS/NZS 2341.10 Determination of the effect of heat and air on a moving film of bitumen (rolling thin film oven (RTFO) test)
- AS 2341.12 Determination of penetration
- AS/NZS 2341.13 Long-term exposure to heat and air
- AS 2341.18 Determination of softening point (ring and ball method)
- AS/NZS 2341.23 Determination of residue from evaporation

(iii) AS 2891.3.1 Bitumen content and aggregate grading - Reflux method: Sections h, i, j, k and l.

(iv) AS 3529 Solvents – Toluene

(v) MBT27 Brittle Point of Polymer Modified Binders (Fast Fraass).

(vi) AG:PT/T111 Handling Viscosity of Polymer Modified Binders (Brookfield Thermosel) http://www.austroads.com.au/pdf/TestMethod/T111_Brookfield.pdf

(vii) AG:PT/T121 Consistency, Stiffness, Elastic Recovery and Tensile Modulus of Polymer Modified Binders (ARRB Elastometer) http://www.austroads.com.au/pdf/TestMethod/T121_Elastometer.pdf

(viii) Brookfield manual that describes the Brookfield Thermosel (other mechanically equivalent brands may be substituted)

3. Apparatus

(a) A balance of about 5 kg capacity with a limit of performance of ±0.1 g

(b) Fume cupboard

(c) For Method A Step 5.2:
Quickfit type glassware suitable for simple distillation preferably with 1 L round bottom flask with matching heating mantle, Liebig condenser, thermoregulator and adjustable platform

(d) Centrifuge with peripheral radius of approximately 180 mm and maximum speed at least 4500 rpm capable of accepting centrifuge tubes, either 2 of at least 50 mL capacity or 4 of at least 25 mL capacity

NOTE: If there is any doubt as to the suitability of the centrifuge or the centrifuging technique, test approximately 2 g of the recovered binder by ASTM D 482 for ash content. The ash content should be below 0.5%. This must be done every two years and if a different centrifuge is used.

(e) Rolling thin film oven (AS 2341.10) with the facility to switch the power supplied to the heaters off while the fan and carriage remain turning

NOTE: This may be accomplished with dual range ovens, i.e. those which have switched ranges for maintaining both 100 °C and 163 °C simply by switching to the lower range.

(f) A thermostatically controlled oven, with good air circulation and capable of maintaining temperatures of 135° ± 5°C

(g) Hot plate

(h) Carbon dioxide source as described in AS 2341.13

(i) At least 3 RTFO Bottles

NOTE: Cylinders lubricated with silicone compounds such as "Mollycote" may be tested. As much contaminant as possible should be scraped off with a flexible knife or spatula. The RTFO bottles used will become contaminated and must be either reserved for this work or thoroughly cleaned.

(j) Scraping tool suitable for scraping RTFO bottles radially in a direction parallel to their axes

NOTE: A suitable scraper has a handle with razor blade mounted axially at one end.
Old dinner knives are a convenient way of scraping the binder off the razor blade.

(k) Beakers 600 mL and 50 mL, stirring rods, graduated cylinders, and pipette (optional)

(l) Hammer and chisel, and diamond or masonry saw

(m) Penetration rings: Elliptical brass rings 11 mm high and 31 mm major axis and made from brass plate 5 mm thick with tripod supports

NOTE: Penetration rings of greater height to diameter allow the binder to form a concavity on cooling which makes observation difficult and hence should not be used. Heights less than 11 mm may damage needles when soft samples are recovered.

(n) For Method B Step 5.3: steam rings (Bain Marie)

(o) Moulds and tubes as appropriate to AS 2341.2, AS/NZS 2341.5 and AS 2341.18

(p) Stop watch, aluminium foil, torch or lamp for viewing RTFO

(q) Heat lamp

(r) (Optional) 75 μm AS sieve

(s) The following materials are required for the test:

(i) Toluene conforming to AS 3529, or distilled toluene from this test method may be re-used

(ii) Option in Step 5.3(h) uses Pyridine

4. Preparation

(a) The minimum quantity of recovered binder required for each test is listed in Table 1.

Table 1 – Tests and Approximate Mass of Binder Required

Description	Test Reference	Mass
Penetration:	AS 2341.12	7.5 g
Viscosity and other tests:		
▪ Sliding plate	AS/NZS 2341.5	0.3 g
▪ Softening point (one ring)	AS 2341.18	2.3 g
▪ Asphalt Institute (AI) viscosity *NOTE: AI 800R requires a larger sample*	AS 2341.2	6 g
▪ Fast Fraass (triplicate)	MBT27	1.2 g
▪ Brookfield Thermosel	Brookfield manual or AGPT/T111	10 g
▪ Infra-red spectrophotometry *NOTE: For testing multigrade, PMB or anything doubtful.*	T521	0.5 g
▪ ARRB Elastometer C mould	AG:PT/T121	6 g

(b) Where the sample is a toluene solution:

(i) Do not stir before taking a sample

(ii) Determine the non-volatile content (*N*) of the toluene solution using either AS 2891.3.1 or AS 2341.23

NOTE: If the non-volatile content (N) of the toluene solution has already been determined use that value.

(iii) Calculate the mass (*M*) of toluene solution required for distillation in g as follows:

$M = \frac{2400}{N}$ for 3 RTFO bottles OR $M = \frac{3150}{N}$ for 4 RTFO bottles.

(c) Where the sample is asphalt (e.g. loose, core or cylinder):

(i) For an asphalt core or cylinder, cut the core to obtain the required sample size. Where more than one layer of asphalt is present in the core, ensure that the sample represents the required layer

NOTE: Cutting may be diamond saw or masonry saw.
Asphalt cylinders coated with wax are not suitable for this test method and must be rejected.

(ii) Break up the asphalt into small fragments

NOTE: This may be helped by heating the sample in an oven at 60°C for 15 to 20 min.

(iii) Measure out a sub-sample of at least $\frac{700n}{B}$ g,

where *n* is number of RTFO bottles and *B* is the % binder content.

(E.g. Asphalt with about 5% bitumen content requires at least $\frac{700 \times 3}{5} = 420g$).Procedure

5. Procedure

5.1 General

(a) The amount of bituminous binder recovered per RTFO bottle must not exceed 8.0 g. If more than about 21 g recovered binder is required for testing then additional RTFO bottles are required. The number of RTFO bottles *n = required mass of binder / 7*

(b) The sample is tested depending on its form:

(i) If the sample is a toluene solution, recover the binder according to Steps 5.2 (Method A) and 5.4, and test the binder according to Step 5.5

(ii) If an asphalt sample is provided, recover the binder according to Steps 5.3 (Method B) and 5.4, and test the binder according to Step 5.5

5.2 Sample as a solution (Method A)

(a) Set up the distillation apparatus for simple distillation with a Liebig type condenser to cool the distillate and a graduated cylinder receiver

(b) Decant a quantity of solution prepared in Step 4(b) to less than half-fill the distillation flask. Do not add the sediment to the distillation flask
If *M* is more than half the capacity of the distillation flask, the distillation will need to be interrupted and the remaining solution added at Step 5.2(e)

(c) Add ceramic chips to the flask. Adjust the rate of heating so that distillation proceeds at a smooth but rapid rate

NOTE: The vapour phase temperature may rise to about 115°C.

(d) Calculate the expected volume (*V*) of distillate in mL as follows:
$V = 1.14M - 100$ for 3 RTFO bottles
OR
$V = 1.14M - 135$ for 4 RTFO bottles

(e) As the volume in the distillation flask is reduced progressively add any solution remaining from Step 5.2(b)

(f) As the volume of distillate nears *V*, decrease the heat setting so that distillation ceases at V ± 10 mL

(g) Lower the heating mantle on the adjustable platform and allow the flask to cool for at least 15 min

(h) Proceed to Step 5.4

5.3 Sample of asphalt (loose, cores or cylinders) (Method B)

(a) Place the asphalt prepared in Step 4(c) in a suitable beaker

(b) Add 20 mL of toluene for every 140 g of asphalt

NOTE: The solution may be soaked overnight to improve dissolution of binder

(c) Heat the solution for about 1.5 h on steam rings, stirring or agitating about every 30 min

(d) Decant the toluene into a 600 mL beaker

NOTE: A glass rod at the lip of the beaker may assist decantation.

Decantation is to reduce the amount of aggregate in the centrifuge, some fine aggregate is inevitable.

(e) Add 10 mL toluene per 140 g asphalt and stir the solution

(f) Heat the solution for 0.5 h on steam rings

(g) Decant the toluene into a 600 mL beaker

(h) Add 5 mL toluene per 140 g asphalt and immediately decant into a flask.

NOTE: If tar is believed to be present the 5 mL of toluene may be replaced with pyridine (consult MSDS and work only in fume cupboard) followed by additional warming of the material on the steam rings.

(i) Proceed to Step 5.4.

5.4 Recovery of binder

5.4.1 Centrifuge

(a) Transfer all the solution from the flask to centrifuge tube(s)

(b) Where required to balance the centrifuge:

(i) Fill empty tubes with solution either from rinsing the flask with toluol or unconcentrated toluene solution from the same sample

(ii) If the centrifuge tubes are small, more than 2 may be required

(iii) Use a dummy tube to balance an odd number of sample tubes

(c) Centrifuge the solution under conditions of time and peripheral speed that have been found to be satisfactory (refer to Note for Step 3(d))

(d) Transfer the binder solution from the centrifuge tubes into a graduated cylinder without allowing the dregs to enter the cylinder. Use either of the following methods:

(i) Decant the solution using a glass rod, stop the transfer if cloudy filler is seen near the top of the centrifuge tube

(ii) A pipette with a guide mark to prevent it reaching within 10 mm of the base of the centrifuge tube

5.4.2 RTFO Oven

(a) Transfer no more than 30 mL of supernatant solution to each of the RTFO bottles. Mark the bottles if others will be in the oven

(b) Place the RTFO bottles in a cold or almost cold RTFO oven

(c) Start the flow of carbon dioxide at 4 L per minute. Turn on heat set to the 163°C range and cause the carriage to rotate

NOTE: Binder solution from two different specimens can conveniently be tested simultaneously. Both the centrifuge and rolling thin film oven work better when full. If not full, dummy bottles and tubes are used to balance the instrument. Mark the tubes and bottles carefully and distinctively.

(d) When the oven temperature passes 100°C, start to observe the contents of the bottles through the oven window with a lamp or torch. Continue observation until the bottles contents appear dry (i.e. the 'dry point'). Record the heating time and temperature at the dry point

NOTE: It is expected that the bottles will be dry at this stage. If they are not dry within another 5 min, carefully check the gas plumbing and the oven temperature.

(e) From the dry point, continue heating until all the following 3 criteria are achieved:

- Maximum temperature at least 130°C but no more than 136°C
- Heating for a period of 30 to 40 min
- Time heated above 125°C is at least 12 min

(i) Record the time when the temperature reaches 125°C

(ii) If the temperature rises above 135°C, reduce the heating to maintain the range 130° to 136°C

NOTE: Turn the switch to the 100°C range and then back up again as required.

(iii) If the temperature does not rise to 130°C in 30 min, increase the set point and maintain in the range 130° to 136°C

(f) Turn the oven and gas flow off

(g) Remove one RTFO bottle at a time from the oven and without delay scrape with special scraper and collect the scraped material in the same 50 mL beaker

(h) Proceed to Step 5.5

5.5 Penetration of recovered binder

(a) Prepare the sample for penetration testing as follows:

NOTE: The aim is to achieve glossy a bubble-free surface for the penetration test.

(i) Heat the beaker of scraped binder in an oven maintained at 135° ± 5°C for about 10 min

(ii) Place the beaker on a hotplate and while heating stir the binder with a thermometer. Do not allow the temperature to exceed 140°C in the first 5 min and never exceed 165°C. If bubbling is rapid, decrease the temperature until bubbling ceases

(iii) When bubbling has almost ceased remove the heat and record the final temperature and the total time period over 140°C

(iv) Pour the sample into the penetration rings. Ensure the ellipse is centred when pouring so that when set up for penetration measurement the assembly does not rock

(v) Allow to cool in air to ambient temperature

(vi) Break any bubbles in the surface of the cooled penetration specimen by lightly touching the bubble with a bare finger, followed by a few seconds under a heat lamp

(b) Measure penetration at 25°C according to AS 2341.12 except for the following amendments:

NOTE: As penetrations may be small, the operator should be well trained in observing the needle and its reflection, being aware of looseness and backlash in the measuring and needle placement mechanism.

(i) The cooling in air phase Clause 5(c) may be reduced to 30 min but the 1 h in the water bath must not be reduced

NOTE: Cooling rate is greater for a smaller sample and the large area of brass.

(ii) Duplicate penetrations are to be measured approximately at the foci of the ellipse to conform to Clause 6(g) 10 mm apart and 10 mm from edge

(iii) If the average penetration is less than 12 mm/10, dry the ring and place under a heat lamp until the indentations have just flowed out. Repeat Step 5.5(b) but at 35°C or some higher temperature so that a penetration > 12 mm/10 is determined

(c) Following the test return the binder to the sample in the beaker

NOTE: Remove the penetration ring from the base plate and heat it on its side on a hotplate so that the binder can be recovered.

(d) If there was nil total time period over 140°C at Step 5.5(a), check the sample preparation at the conclusion of testing using the procedure in Step 5.7

5.6 Testing of recovered binders

(a) Carry out the tests specified on the recovered binder using the appropriate methods and requirements listed in Table 2

Table 2 – Requirements for Testing Recovered Binders

Method	Criteria	Requirement
Sliding plate viscometer AS/NZS 2341.5	All	Appendix A Use 25° for unaged binder and fresh asphalt or 45° on aged binders. Appendix B If the slope or 'c' value is reported, the viscosity can be extrapolated for any shear rate.
	Penetration > 25 mm/10	Viscosity at 25°C except for the following: Appendix C Prepare a single sliding plate sandwich. Appendix D Record to 0.005 s^{-1} and the 'C' value.
	Penetration ≥ 12 & < 25 mm/10	Appendix E Use a single sliding plate sandwich at 45°C. Appendix F Record to 0.005 s^{-1} and the 'C' value.
	Penetrations ≥ 7 & < 12 mm/10	To reduce the time required to record sliding plate results: Appendix G Do not exclude the data from the highest loads as suggested by 9c of the method. Appendix H Do not drop loads > 2 kg onto the hook. Appendix I Ignore Appendix A2 as this applies only when a viscosity of 5.67 is targeted.

Method	Criteria	Requirement
	Penetration < 7 mm/10	Appendix J A single load of 3.058 kg will apply a stress of 5 x 10^4 Pa. Do not apply any higher loads. Appendix K Apply the load for sufficient time to establish a slope after any initial curvature is ignored. Appendix L Do not use software designed to interpolate to a fixed shear rate. Appendix M An alternative is to run the sliding plate at 60°C. *NOTE: Cotton gloves worn under rubber gloves can help avoid burns when installing a sample.* *It is permissible to run the same plate at 2 temperatures: first run the lower temperature, then invert the sandwich so that the shear is in the reverse direction and run at the higher temperature.*
Softening point AS 2341.18	All	Appendix N Use a single ring to conserve material for other tests. Appendix O Two rings of different materials may be run side-by-side. Duplicates are permissible.
	Penetrations ≥ 7 & < 14 mm/10	Appendix P Prepare 2 rings (run one in water first) as the softening point may be ≥ 80°C. Appendix Q Record the water softening point if < 80°C. Appendix R Record glycerol softening point if the water softening point ≥ 80°C.
	Penetration < 7 mm/10	Appendix S Run the test in glycerol.
AI viscosity AS 2341.2	Penetration ≥ 45 mm/10	Appendix T Use AI 100
	penetration ≥ 25 & < 45 mm/10	Appendix U Use AI 200
	Penetration ≥ 15 & < 25 mm/10	Appendix V Use AI 400
	Penetration < 15 mm/10	Appendix W Use AI 800R (requires a larger sample)
Fast Fraass MBT27		Appendix X Carry out in triplicate
Brookfield Thermosel Brookfield manual or AGPT/T111		Appendix Y Recommended conditions, LVT spindle 31, 135, 150 and 165° at fastest achievable speed. *NOTE: Fully digital Brookfields (e.g. the DV-II+Pro) run at speeds as low as 0.01 RPM are capable of measuring viscosity at 60°C. Older style Brookfields have minimum speeds of 0.3 or 0.5 RPM. If an estimate of the viscosity at 60°C is required, the RVT Brookfield is preferable & should be used with Thermosel spindle SC4-29.* Appendix Z Equilibrate sample at each temperature for 30 min before measuring. Appendix AA Measure the viscosity at 105°C and lower temperatures down to the lowest temperature possible. Appendix BB Take logarithms of the viscosities and extrapolate to predict a viscosity at 60º; if desired an additional datum may be added due to the softening point temperature which can be taken to be at a viscosity of 1310 Pa.s.

Method	Criteria	Requirement
Infra-red spectrophoto-metry T521		Appendix CC Binder solution may be used.
ARRB Elastometer C mould AG:PT/T121		Appendix DD Use a shear strain rate of 0.1 and a strain of 1 at temperature either 60° (45° and 70° and lower strain rate may be used in extreme cases). Appendix EE Use the testing software to report the viscosity extrapolated to zero strain N=100.

5.7 Check on Sample Preparation

(a) If there was nil total time period over 140°C at Step 5.5(a), check the sample preparation using the following procedure:

(i) Collect any remaining sample, including that recovered from the Thermosel tube

(ii) Overheat the sample to 200°C for 5 min either in the apparatus of AS/NZS 2341.5 or in a small beaker stirred with a thermometer on a hotplate

(iii) Determine the penetration and softening point on the overheated sample. The penetration is measured prior to cutting the meniscus of binder from the ring (Part 6d of AS 2341.18). If the amount of sample is too small for the penetration ring and if the original penetration was < 25 mm, then both the penetration and the softening point may be measured from a single softening point ring

(b) Compare the mean penetration results for the standard and the overheated samples:

(i) If the test results differ by ≤ 30%, do not report the overheated penetration or softening point

(ii) If the difference is > 30%, report the penetration and the softening point of both the standard preparation and the overheated sample

6. Calculations

Calculations are included in the referenced Test Method

7. Reporting

Include the following results in the report:

(a) Report the sample details and original form of sample (i.e. solution or type of asphalt sample)

(b) Penetration at each temperature and average penetration rounded to the nearest 1 penetration unit (mm/10). Where required in Step 5.7, the overheated sample penetration

(c) The results for each of the tests specified:

(i) Sliding Plate Viscosity (log Pa.s) of a single plate to three significant figures and the temperature to the nearest degree. In addition, either the slope or "c" value where the viscosity can be interpolated to a shear rate of 0.005 s^{-1}, or the actual shear rate (s^{-1})

(ii) Softening point (mean if duplicates) to the nearest 0.5ºC. Where required in Step 5.7, the Softening point of the overheated sample

NOTE: It is not necessary to identify water or glycerol.

(iii) Asphalt Institute Viscosity (Pa.s) to three significant figures

(iv) Fast Fraass report the mean of triplicates rounded to the nearest 1°C

(v) Brookfield Thermosel Viscosity (Pa.s) and the measurement temperature (°C) to three significant figures. Where a 60°C viscosity is estimated, the source viscosities and measurement temperature

(vi) Infra-red spectrophotometry results:

- For SBS/SBR types, the % SBS/SBR resin by mass on the total mass of bitumen plus resin
- For the EVA/EMA types, the % carbonyl functionality and the estimated % EVA or EMA resin

(vii) ARRB Elastometer C Mould (Pa.s) to three significant figures. Identify if temperatures and strain conditions other than those recommended were used

(d) Reference to this test method

RMS 12.624