COMMENTARY TO AG:PT/T102 – PROTOCOL FOR HANDLING MODIFIED BINDERS IN PREPARATION FOR LABORATORY TESTING

PREFACE

This Modified Binder Test Method was prepared by the Bituminous Surfacings Research Reference Group BSRRG (formerly National Bituminous Surfacings Research Group) on behalf of Austroads. Representatives of Austroads, ARRB Group and Industry through the Australian Asphalt Pavement Association (AAPA) have been involved in the development and review of this test method.

FOREWORD

Modified binders are heat-sensitive materials and may undergo significant changes in test results and ultimately to their properties as a result of exposure to high temperatures for extended periods. An additional potential problem is that they may separate from the modified binder dispersion. Improper heating and homogenisation of samples can, therefore, affect the test results obtained on sampled binders, which may thus not truly represent those of the original material.

Recent testing experience, particularly with those binders formulated with SBS polymer at concentrations greater than four percent suggests that the manner of binder handling prior to testing can significantly influence the (empirical) test results. This laboratory handling protocol represents agreed best practice based on an extensive period of review and evaluation.

Estimates of precision reported by Austroads and included in test methods released since 2002 were obtained on modified binders handled with this protocol.

SCOPE

This test method adopts the principles of AS 2341.21 and applies these to modified binders that are to be tested in accordance with the test methods set out by Austroads. Such binders include polymer modified bitumen and multigrade bitumen.

Further Development

This test method is a key component of laboratory best practice for the testing of modified binders. Further improvements will be introduced as they become available.

PROTOCOL FOR HANDLING POLYMER MODIFIED BINDERS IN THE LABORATORY

1 REFERENCED DOCUMENTS

The following documents are referred to in this method:

AUSTROADS

AG:PT/T101	Method of sampling polymer modified binders, polymers and crumb rubber

AS

2341	Methods of testing bitumen and related road making products
2341.21	Sample preparation

ASTM

E 1	Standard specification for ASTM thermometers

IP	Methods for analysis and testing, Part 1, Vol. 2, Appendix A

2 PRINCIPLE

The objective of this procedure is to ensure that modified binder samples, when tested, are representative of the product as sampled. To achieve this, samples must undergo a minimum and controlled amount of heating. A flow chart of the prescribed handling procedure options is set out in Appendix A.

3 APPARATUS

a. Pre-heating oven - able to maintain a temperature of 160 ±10 °C. May be fitted with a timer switch.

b. Forced draught oven - able to maintain a temperature in the range 100 to 200 °C, with a set-point accuracy better than ±5 °C.

c. Thermometer - conventional mercury-in-glass, partial immersion thermometer for hotplate use and oven monitoring, accurate to 0.5°C. A suitable thermometer is IP 61 C or ASTM 16C, as specified in ASTM E 1, for use at around 200 °C.

d. Mechanical stirrer - with impeller able to maintain the stirring characteristics described in Appendix B

e. Heating block - aluminium block, e.g. 150 mm diameter, with a cavity able to accept a nominal 400 mL beakers with a clearance up to 2 mm.

f. Beaker – nominal 400 mL (or equivalent container) to fit the heating block with suitable lid e.g. standard 500 mL paint container with lid is suitable and can be discarded after use.

g. Temperature controllable hotplate - able to maintain a temperature in the range 135 - 200°C, with a set-point accuracy of ±5°C.

h. Temperature controller - an alternative to the temperature controllable hotplate is described in Appendix C. This system uses an oven temperature controller with Pt100 remote temperature sensor. Better temperature control is provided along with direct temperature readout. No monitoring thermometer is required after system calibration check.

i. Temperature probe - a suitable probe based on a thermocouple or Pt100 used in conjunction with a corresponding readout device, with an accuracy of ±2 °C. Alternatively, a suitable thermometer (or equivalent) may be used.

j. Stirrer – flat blade spatula, suitable for the containers used.

k. Beaker(s) - 250 - 300 mL sub-sample containers.

Note 1 Some apparatus items are optional depending on method selected

4 PROCEDURE

4.1 General

Modified binders can be complex mixtures of polymers and a variety of petroleum products. If handled in accordance with the directions of the suppliers, there should be no significant safety risk. It is recommended that notices, describing the action to be taken in the event of hot binder burns, should be displayed in the laboratory in the areas where bitumen, multigrade and PMBs are handled. A suitable warning could be as follows:

WARNING: HOT BITUMEN, MULTIGRADE & PMBs CAN CAUSE BURNS

The following precautions should be taken when handling hot bituminous binders, such as bitumens, multigrade bitumens, polymer modified bitumens and crumb rubber modified bitumens:

a. Eye protection, such as safety glasses and/or face shields, shall be worn when handling hot bituminous binders.

b. Heat-resistant gloves with close-fitting cuffs, and other suitable protective clothing, shall be worn when handling hot bituminous binders.

c. There shall be no smoking or the presence of other ignition sources in close proximity while handling hot bituminous binders.

d. Bituminous binders heated in the presence of small quantities of water may foam excessively and spatter or overflow the sample containers. Samples should be checked for the presence of water while the material is still cold. First loosen the lid of the sample container, if necessary by gently heating the container lid, and examine

the cold sample for the presence of water. If water is found to be present, drain off as much as possible and allow to dry at room temperature or blow-dry with clean compressed air.

4.2 Sampling

a. An original sample shall be obtained in accordance with the procedures set out in AGPT/T101.

> Note 2 Samples taken from, production, bulk storage, and transport tankers or during transfer from or into these vessels shall be treated as original samples.

b. If the original sample has just been taken and the viscosity is low enough (i.e. temperature is 160 ºC or higher), it can be reduced, after suitable stirring, into sub-samples for later testing. Note that these samples must be allowed to cool to below 60°C before reheating and testing (testing of hot poured original samples will generally give higher test results). These smaller sub-samples shall be re-heated only once prior to testing (see 4.5 and 4.6).

4.3 Sample Reduction – Cold Bulk (Sample < 160 °C)

4.3.1 Hotplate method

a. Preheat the bulk sample in the preheating oven set at 160ºC

> Note 3 Heating times vary according to oven type, sample size and initial temperature. A minimum of four hours is generally required if the samples are larger than 4 L. Samples must not be heated for more than ten hours at 160 °C. The use of a timer switch is recommended to allow preheating to commence overnight.

b. Place the sample onto the hotplate and commence stirring with a spatula without entraining air, occasionally scraping the sides and the bottom of the container. When the sample is of uniform consistency, check its temperature with a thermometer and continue stirring.

c. When the sample reaches 160 °C, divide the bulk sample into sub-samples using smaller containers (1 L or less). Remove the bulk container from the hotplate.

> Note 4 At this stage, check the material for any unusual or unexpected physical properties, such as extreme fluidity, cutter odour, fume, or high consistency. If the presence of cutter, or other volatile oil, is suspected, a thin film dish or RTFO treatment bottle may be poured at this stage for further testing (mass loss determination).

4.3.2 Oven method

a. Set the fan forced oven to 180 °C.

b. Heat the bulk sample to 160 °C, allowing sufficient time to reach this temperature. This temperature is check by inserting a temperature probe into the sample or timed according to the appropriate heating time as determined in procedure set out in Appendix B.

c. Remove the bulk sample from the oven and thoroughly stir it for 30 seconds with a spatula without entraining air.

d. Divide the bulk sample into sub-samples in containers of 1 L, or less. Some or all of these samples may be further heated in preparation for pouring into test moulds for testing by the procedure in 4.5. At this stage, check the material for any unusual or unexpected physical properties (see Note 3 above).

4.4 Sample Reduction – Hot Bulk (Sample ≥ 160 °C)

4.4.1 Hotplate method

a. If the bulk sample is 160 °C or hotter it may be sub-sampled without heating by stirring for at least 30 seconds with a spatula and decanting into containers, 1 L or smaller, and allowed to cool.

> Note 5. If immediate testing is required the sub-samples must first be allowed to cool to below 60 °C before reheating according to 4.5 or 4.6 for pouring of test moulds. Sample reduction from bulk (see Note 9)

4.5 Sub-sample Preparation for Testing (Hotplate Method)

a. Take sub-sample as already decanted at stage 4.3.1 (c) or 4.3.2 (d).

b. Set the temperature controlled hotplate to 180 °C and allow temperature to stabilise.

c. Heat the sub-sample(s) to 180 °C in the preheated aluminium block. Do not hold at 180 °C for longer than 30 minutes.

> Note 6 The Aluminium block can be brought to the working temperature on the hotplate or by pre-heating in an oven.

d. Stir the sub-sample with a mechanical stirrer and suitable impeller for 30 seconds without entraining air (refer to Appendix C).

e. Fill the required test containers.

f. Discard the remnants of the heated sub-sample(s). Do not add to current working *stock.*

4.6 Sub-sample Preparation for Testing (Oven method)

a. Take sub-sample as already decanted at stage 4.3.1 (c) or 4.3.2 (d). and place in an oven set at 180 ºC

b. The samples are to be heated for a minimum time as determined under the procedure set out in Appendix B2 to achieve a temperature of at least 170 ºC.

c. Remove the sub-sample tin from the oven and immediately transfer to a hot plate and commence stirring (manual or slow mechanical) until a binder pouring temperature of 180 ºC has been reached (time taken must not exceed 30 minutes).

d. Pour the test moulds without delay and schedule testing according to the respective test method.

> Note 7. For multiple sample testing ensure that only that number of sub-samples is preheated as can be poured within 15 minutes (i.e. oldest sub-sample is held for no more than 15 minutes at 180 ºC).

4.7 Testing of Hot Production Samples

a. If the sample is below 175 ºC heat the sample tin in a forced draught oven or on a hotplate until the sample reaches 180 ±5 ºC. For samples at or above 175 ºC proceed to 4.7(b).

b. Stir and fill the required test containers and allow the binder to cool. Unused binder handled at or above 175 ºC (i.e. without reheating) may be reheated one more time for testing if required i.e. can be treated as a sub-sample under 4.5 or 4.6.

> Note 8. Where disputes arise between manufacturer and user of the binder, test properties shall be determined on samples prepared from cold product. Manufacturers testing hot poured samples under 4.7 must ensure that the relevant specifications can also be met by testing of samples cooled to below 60 °C.

5. REPORT

The following information shall be reported:

a. Sample history including the preparation method.

b. The presence of fumes or cutter odour.

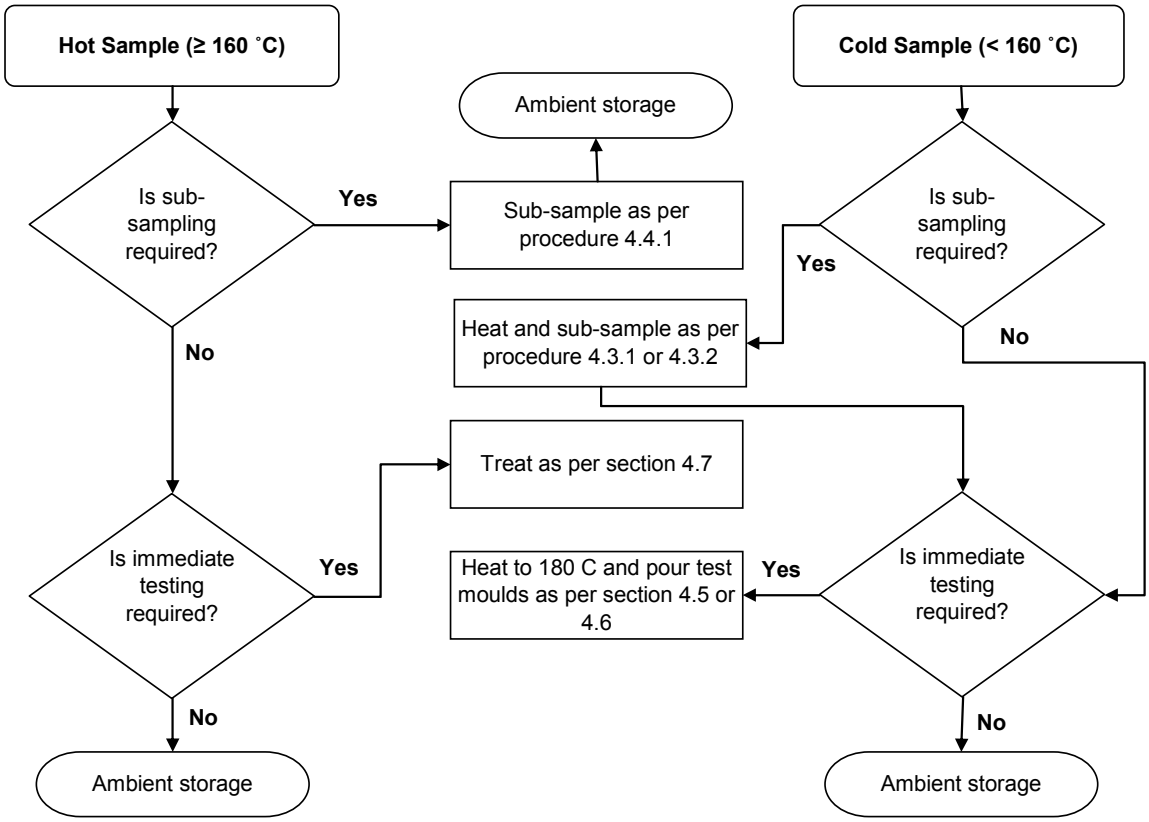
c. The presence of foaming.

d. Unusually high consistency or inhomogeneity.

APPENDIX A

(Normative)

Sample Handling Protocol Options



APPENDIX B

(Normative)

Heat-up rate of sample in forced draught oven

B1 GENERAL

During the preparation of bulk samples for sub-sampling, the minimum time required for a bulk sample to reach the desired temperature (e.g. 160 °C or 180 °C) must be known.

In preparing sub-samples for testing, the minimum time required for the particular size of container to attain the filling temperature must be known.

The procedure below shall be carried out, as required, for each size of container used during the forced draught oven sample preparation in this method

B2 PROCEDURE

The procedure shall be as follows:

a. Insert a thermocouple into the sample as follows:

   (i) Punch a hole through the lid of the container.

   (ii) Fill the container with the sample and secure the lid.

   (iii) Allow the sample to cool until it is semi-solid.

   (iv) Insert the temperature probe through the hole to half the sample depth and secure it to ensure the probe tip does not move as the sample softens during the heating and monitoring phase.

   (v) Cool the sample to room temperature.

b. Set the oven to the required temperature, viz. 160 °C when using bulk samples, or 180 °C when using sub-samples.

c. When the temperature has stabilised, place the sample in the oven on the shelf and in the position normally used for heating bitumen samples. Leave the temperature probe in place and close the door on the thermocouple wire (these wires can be passed though an oven top vent if required).

d. Monitor the temperature with time and establish the minimum time required for the sample container to reach within 5 ˚C of the required temperature.

e. Remove the container, cool to room temperature and repeat Steps (a) through (d) for other relevant container sizes.

Note B1: Alternatively, a suitable thermometer can be used in place of the temperature probe.

Note B2: If the laboratory sometimes tests several samples at the one time, the above procedure should also be carried out with the monitored sample surrounded by bitumen-filled dummy containers.

Note B3: Glass beakers and metal containers with cut-off rims will require lids.

B3 RECORDING

The following information shall be recorded:

a. The minimum time required for a particular size of bulk sample container to reach 155 °C (for 160 °C set point).

b. The minimum time required for sub-sample containers intended to be used to reach 175 °C (for 180 °C set point).

APPENDIX C

(Normative)

Mechanical stirrer and impeller

C1 GENERAL

For sample mixing to be repeatable and reproducible between laboratories, the rotational speed, paddle design and sample container should be adequately defined. By defining the sample size at 1 Litre, and specifying the impeller the geometry of the system is established. The chosen system is described as a low shear rate mixer and is not intended to blend the polymer into the binder. Its purpose is to ensure a homogeneous system for well manufactured binders by overcoming any segregation which may have occurred in the original sample.

C2 EQUIPMENT DEFINITION

For the 1997 PMB inter-laboratory precision exercise, a commercial paint stirrer (impeller) was chosen for its availability and simple design. Figure C1 describes the paddle and provides the important dimensions (informative only). A simple impeller should be selected to ensure the incipient vortex described in Fig. C2 can be sustained with the available mechanical stirrer. A high shear mixer e.g. Silverson type, is not suitable for this test method.

The mechanical stirrer is a general purpose laboratory stirrer with variable speeds. The combination of stirrer and paddle with an appropriate rotational speed ensures that during the stirring process, the surface of the sample will be on the verge of forming a vortex. The maintenance of this incipient vortex ensures adequate mixing without the entrainment of air into the sample. Figure C2 describes the ideal mixing condition.

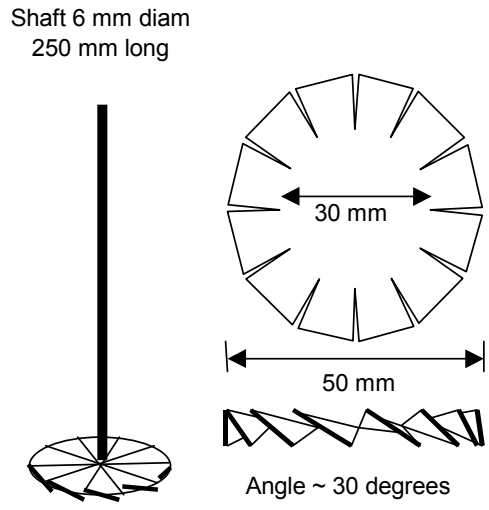
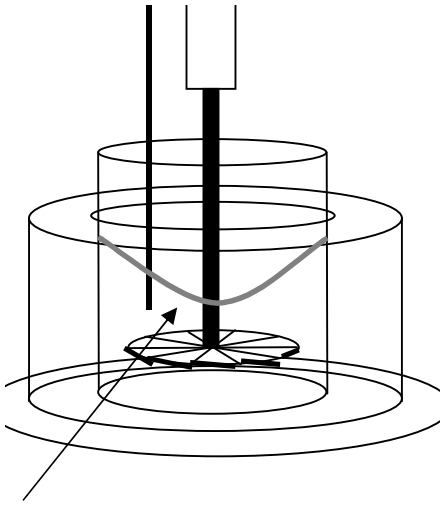


Fig. B1. Impeller assembly (Informative)



Vortex must not be turbulent at any stage in the stirring process. Speed of stirrer motor adjusted to point short of air entrapment.

Fig. B2. Incipient Vortex

APPENDIX D

(Informative)
Electronic Temperature Controller

D1 GENERAL

Sample heating using the temperature controlled hotplate, heating block and thermometer can be difficult to manage both in terms of control precision and temperature stability. The lack of a direct temperature measurement link between the sample and the hotplate is the primary cause of these deficiencies.

D2 EQUIPMENT

Several electronic controller manufacturers have released cost effective modules for temperature control in a variety of application. These devices can be seen in recently manufactured laboratory ovens, MATTA test cabinets and Durability ovens and provide a high level of stability through the use of a Platinum resistance (Pt100) sensor. Other features are:

- Low noise (zero crossing) power switching to eliminate interference with other laboratory equipment (computers etc.)
- Direct selection of program temperature set point
- Display of actual sample temperature to 0.1 °C (can be calibrated to ensure precision).
- Long term stability

Although not commercially available, the arrangement presented in Figure D1 can be readily assembled. This module can be applied to a variety of temperature control applications and can serve as a temperature monitor without the using the control function

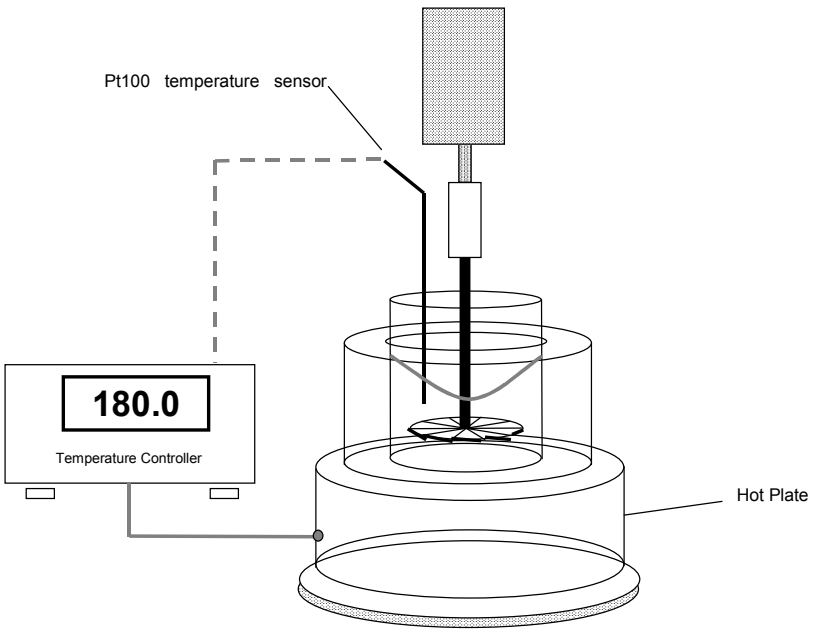


Fig. C1. Temperature Control Module and Hotplate Assembly

AMENDMENT RECORD

Amendment No.	Clauses amended	Action	Date
1	Commentary Page	New	June 2005
	Footer and header	Format	
	Applied revised test method number	Format	
	Applied new styles	Format	
2	Applied new test method numbers	Substitution	March 2006
	Moved notes to the end of the method	Format	
3	Changes to the Foreword and scope	Substitution	October 2007
4	Extensive rewording throughout	Substitution	July 2008

Key

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