

# **COMMENTARY TO AG:PT/T141 - LABORATORY PROTOCOL FOR THE PREPARATION OF CRUMB RUBBER MODIFIED BINDERS**

## **PREFACE**

This modified binder test method was developed by The Bituminous surfacings Research Reference Group on behalf of Austroads. Representatives of Austroads, ARRB Group and the Australian Asphalt Pavement Association have been involved in the review of this test method.

## **FOREWORD**

Crumb rubber modified binders are prepared from mixtures of bitumen, crumb rubber and additives selected to optimise the properties of the digested binder system. The procedure used to prepare these hot digested binder can contribute to the properties of the binder and must be well controlled to ensure repeatability between laboratories. This test method provides a protocol for the preparation of crumb rubber blends in the laboratory.

## **SCOPE**

This test method adopts the principles of AS 2341.21 and AG:PT/T102 and applies to crumb rubber modified binders which are to be tested in accordance with the test methods set out by Austroads.

## **FURTHER DEVELOPMENT**

There are no plans for development of this test method.

# LABORATORY PROTOCOL FOR THE PREPARATION OF CRUMB RUBBER MODIFIED BINDERS

## 1 REFERENCED DOCUMENTS

The following documents are referred to in this test method:

### AS /NZS

2341 Methods of testing bitumen and related road making products

2341.21 Method 21: Sample preparation

### ASTM

E 1 Standard specification for ASTM thermometers

### AUSTROADS

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

AG:PT/T102 Protocol for handling modified binders in the laboratory

## 2 PRINCIPLE

The objective of this procedure is to ensure that crumb rubber modified binder samples are prepared in a uniform manner that reflect the processes used in the field. To do this a well defined digestion process with controlled heating and mixing is provided.

## 3 APPARATUS

Pre-heating Oven - able to maintain a temperature of  $100 \pm 10^{\circ}\text{C}$ . May be fitted with a timer switch.

a) Forced Draught Oven - able to maintain a temperature in the range 100 to  $200^{\circ}\text{C}$ , with a setpoint accuracy better than  $\pm 5^{\circ}\text{C}$ .

Note: No specification for the Forced draught oven has been provided. Appendix A describes how the sample heat-up characteristics are determined. Most general purpose laboratory ovens with fan assistance are suited to this task.

b) Thermometer - conventional mercury-in-glass, partial immersion thermometer for hotplate use and oven monitoring, accurate to  $0.5^{\circ}\text{C}$ . A suitable thermometer is IP 61 C or ASTM 16C, as specified in ASTM E 1, for use at around  $200^{\circ}\text{C}$ .

c) Mechanical Stirrer with impeller described in Appendix B

d) Heating block and temperature controller - Aluminium block, approximately 150 mm diameter, 100 mm high, with a cavity able to accept 500 mL tin cans with a clearance up to 1 mm. The base of the block has provision to accept a Pt100 sensor.

e) Thermoset Hotplate - able to maintain a temperature in the range 135 -  $200^{\circ}\text{C}$ , with a setpoint accuracy of  $\pm 5^{\circ}\text{C}$ .

- f) Temperature Controller - An alternative to the temperature controlled hotplate is described in Appendix C. This system uses a modern oven temperature controller with Pt100 remote temperature sensor. Better temperature control is provided along with a direct temperature readout. No monitoring thermometer is required.
- g) Temperature Probe - a suitable probe based on a thermocouple or Pt100 used in conjunction with a corresponding readout device, with an accuracy of  $\pm 2^{\circ}\text{C}$ . Alternatively, a suitable thermometer (or equivalent) may be used.
- h) Stirrer – Flat blade spatula, suitable for the containers used.

## 4 PROCEDURES

### 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. The hazard of burns with modified binders is greater than with standard bitumens, due to the (normally) higher handling temperatures. It is recommended that notices, describing the action to be taken in the event of bitumen or modified 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**

**HOT** Crumb Rubber modified binders increase the risk of serious burns due to the tendency for these materials to adhere in larger quantities at the elevated digestion temperatures (180°C)

The following precautions should be taken when handling bitumen, multigrade or PMBs:

- a) Eye protection, such as safety glasses and/or face shields, shall be worn when handling hot bitumen, multigrade or PMBs.
- b) Heat-resistant gloves with close-fitting cuffs, and other suitable protective clothing, shall be worn when handling hot bitumen, multigrade or PMBs.
- c) There shall be no smoking while handling hot bitumen, multigrade or PMBs.
- d) A safety protocol should be developed for the digestion of Crumb Rubber modified binders to ensure no operator contact with these hot digested binders during mixing or sample pouring, or while hot samples are present.
- e) Sample sizes should be kept to a minimum.

### 4.2 Sampling

- a) An original sample of the bitumen shall be obtained in accordance with the procedures set out in AS 2341.21 (see Note 1).
- b) Samples of the crumb rubber shall be obtained (see Note 2) in accordance with the procedures set out in AG:PT/T101.

- c) Samples of prepared Crumb Rubber modified binders (see Note 3) shall be obtained in accordance with the procedures set out in AG:PT/T102.

#### **4.3 Blending**

#### **4.4 Testing**

### **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.

### **Notes**

- 1. Where routine testing is undertaken it is convenient to prepare several 350 mL samples of the selected bitumen in 500 mL tins matched to the cavity in the Aluminium block.
- 2. To obtain a representative sample of the crumb rubber from a larger sample the use of a small laboratory riffle box is recommended.
- 3. Since Crumb Rubber modified binders are sensitive to further heating (the digestion process continues), the use of a lower temperature for reheating is recommended. Where the most reliable data is required, laboratory preparation and insitu sampling for testing is recommended.

## APPENDIX A

(Normative)

### HEAT-UP RATE OF SAMPLE IN FORCED DRAUGHT OVEN

#### **A1 GENERAL**

During the preparation of bulk samples for sub-sampling, the minimum time required for a bulk sample to reach 160°C must be known.

In preparing subsamples 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.

#### **A2 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 on the lid.
  - III. Allow the sample to cool until it is semi-solid.
  - IV. Insert the temperature probe to half the sample depth and secure it to ensure the probe tip does not move as the sample softens during the testing 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 subsamples.
- 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 through 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 (c) and (d) (see Notes A1, A2 & A3).

#### **A3 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 subsample containers intended to be used to reach 175°C (for 180°C set point).

**Notes**

- A1 Alternatively, a suitable thermometer can be used in place of the temperature probe.
- A2 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.
- A3 Glass beakers and metal containers with cut-off rims will require lids.

## APPENDIX B

(Normative)

### Mechanical Stirrer and Impeller

#### B1 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.

#### B2 EQUIPMENT DEFINITION

For the 1997 PMB inter-laboratory precision exercise, a commercial paint stirrer (impeller) was chosen for its availability and simple design. Figure B1 describes the paddle and provides the important dimensions. This impeller (or its equivalent) is recommended. At the time of the precision exercise the paddle described was available from Kmart stores.

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 entrapment of air into the sample. Figure B2 describes the ideal mixing condition.

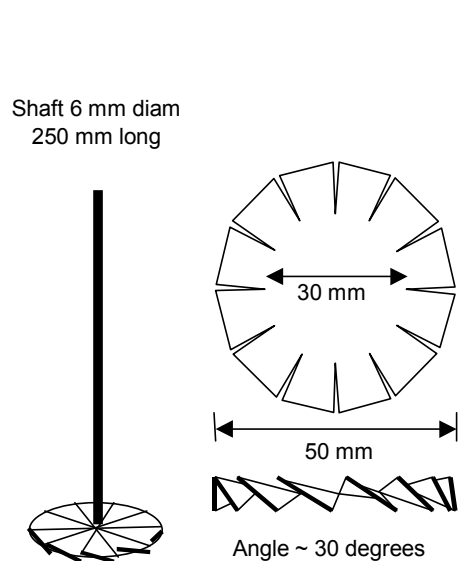
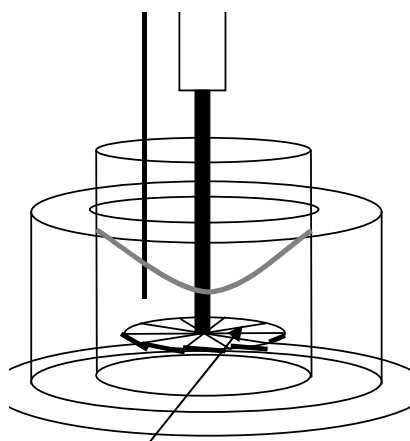


Figure B1. Impeller assembly  
(Informative)



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

Figure B2. Incipient Vortex

## APPENDIX C

(Informative)

### Electronic Temperature Controller

#### **C1 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.

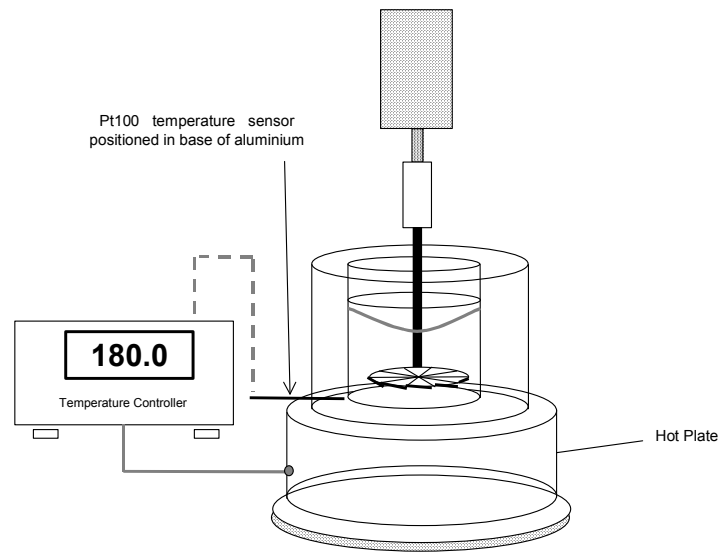
#### **C2 EQUIPMENT**

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 C1 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.

In this application it is recommended that the Pt100 sensor be located in the base of the Aluminium block.



**Figure C1. Temperature Control Module and Hotplate Assembly**

## AMENDMENT RECORD

Amendment No.	Clauses amended	Action	Date
1	This method was developed from AG:PT/T131 2002.	New	May 2002
2	Commentary Page	New	June 2005
	Footer and header	Format	
	Applied revised test method number	Format	
	Applied new styles	Format	
3	Applied new test method numbers	Substitution	March 2006
	Moved notes to the end of the method	Format	

### Key

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