COMMENTARY TO AG:PT/T151 - FUME ASSESSMENT OF MODIFIED BINDERS

PREFACE

This test method was prepared by ARRB Group on behalf of the Austroads. Representatives of Austroads, ARRB Group and the Australian Asphalt Pavement Association have been involved in the development and review of this test method.

FOREWORD

Modified binders are heat-sensitive materials and may undergo significant changes to their properties as a result of exposure to high temperatures for extended periods particularly where added polymer can be damaged and/or separate from the modified binder dispersion. Improper heating of samples can, therefore, affect the properties of binders, resulting in sample test results which may not truly represent the characteristics of the original material.

SCOPE

The scope of this method is limited to the generation and collection of bitumen binder fumes for subsequent external laboratory analysis. Crumb rubber binder blends can also be prepared and fume samples collected during the digestion process.

Fume sampling can be limited to discrete time windows to avoid sampler overload where cutter oil or other significant volatiles are present in the binder.

Further Development

There are no development plans for this test method.

FUME ASSESSMENT OF MODIFIED BINDERS

1 REFERENCED DOCUMENTS

The following documents are referred to in this 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 polymers, crumb rubber and polymer modified binders

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

2 PRINCIPLE

Bituminous binder (including PMBs) is heated and held at a specified temperature (usually 180°C), in a closed glass reaction vessel.

The controlled gas flow (CO_2) is sampled at defined intervals, using BHP Environmental Health Laboratory (EHL) samplers to collect generated fume. The exposed samplers are then sent to the BHP to determine the Total Volatile Organics and Polynuclear Aromatic Hydrocarbons (PAH).

3 OCCUPATIONAL HEALTH AND SAFETY

3.1 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 & PMBs CAN CAUSE BURNS

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. While the material is still cold, loosen the lid of the sample container (invert the can and warm the lid, if necessary), or punch a hole in the lid.

e. Examine the cold sample for the presence of water. If water is thought to be present, drain most of it out, or blow with clean compressed air to evaporate the free water.

3.2 Sampling

An original sample shall be obtained in accordance with the procedures set out in AG:PT/T101 (see Note 1).

If the original sample has just been taken and the viscosity is low enough, it can be reduced, after suitable stirring, into sub-samples. These smaller sub-samples shall be re-heated only once prior to testing.

4. APPARATUS and MATERIALS

The following apparatus and materials are required:

a. Toluene - technical grade (for cleaning bitumen from apparatus).

b. Stirrer - overhead, electric set to 600 rpm.

c. Stirrer/rotor conforming to the equipment specified in AG:PT/T101 (see Note 2).

d. Thermometer; mercury and glass, 76 mm partial immersion, range -10 to +250°C, for independent check of electronic temperature controller

e. Electronic timer-stopwatch

f. Heating block - Aluminium block, approximately 150 mm diameter, 100 mm high, with a cavity able to accept 400 mL beakers with a clearance up to 1 mm

g. Beaker(s) - 400 mL to fit heating block (or equivalent container), with suitable lid.

h. Thermoset hotplate - able to maintain a temperature in the range 135 - 200°C, with a setpoint accuracy of ± 5°C.

i. 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.

j. 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.

k. Forced convection oven - able to maintain a temperature in the range 60°C to 200°C, with a set point accuracy better than ± 5°C.

l. BHP EHL sampler cassette (or alternative design when available)

m. Sampler adapter providing a gas tight fit to the inlet side of the sampler cassette.

n. Fume cupboard conforming to AS2243.8.

o. Gas source with flow meter capable of delivering 2 L/min ± 0.1 L/min for 1 hour.

p. Flow meter capable of measuring 2 L/min ± 0.1 L/min.

q. Top loading electronic balances, capacity 6000 g; capacity 160 g.

5. PROCEDURE

5.1 Gas Flow Calibration

Connect the gas source to the final flow meter and calibrate the source flow meter against the final flow meter. A check of overall calibration can be achieved by passing the gas into an inverted, water filled 1 L standard flask. The time taken to displace 1 L of water provides an adequate calibration for the system.

When the system is connected with the reaction vessel in series (Figure A1), the initial and final flow meters should show their calibrated values. Where a gas loss occurs, the final meter will show a reduced flow compared with the initial meter. The loss should not exceed 25 percent. Apply the correction to the flow calculation (section 7).

5.2 Fume Collection

a. Heat a sample of the test binder to135°C using the forced convection oven.

b. Mix the sample with a broad spatula (including required additives) to ensure homogeneous and transfer 500 ± 10 g into the reaction vessel.

c. Place the reaction vessel into the heating mantle and switch on the heater controller. Insert the temperature probe and stirrer; ensure binder is fluid before turning on stirrer, then stir at a speed just below the formation of the incipient vortex. Set the controller to 165°C to provide an initially rapid heat up rate, but to control overshoot. Readjust and stabilise the temperature at 180 ± 2°C. Figure 1 presents the schematic layout for the fume collection system.

d. Connect the BHP sampler to the system for the selected timing period. Where more than one sampler is used, ensure a record of the sampler numbers and timing periods is maintained. Five minute sample periods are appropriate when 5 percent cutter oil is present.

e. On completion of the test, pour the bulk of the hot spent mixture into a suitable waste container before it solidifies. Allow the reaction vessel to cool sufficiently before handling. Clean residual material from the vessel, stirrer and thermocouple with toluene. Thoroughly dry the equipment before commencing the next test.

7. CALCULATIONS

7.1 Gas Sample

Volume (m^3) = R x T x Initial/Final x 0.001 (m^3/L)

where

R is the flow rate through the sampler (Litre/minute)

T is the sampling period (Minute)

Initial/Final is the ratio of the initial flow rate to the final flow rate with all measurements made on the final flow meter.

7.2 Analyte

Concentration (mg/m^3) = Analyte mass (μg) x 0.001 (mg/μg) / Sample Volume (m^3)

8. PRECISION AND ACCURACY

a. Sampling accuracy is not applicable due to the empirical nature of the test.

b. Chemical analysis precision and accuracy information provided by BHP EHL.

9. TEST REPORT

The final test report shall include the following:-

a. Copy of the test method, including any variations during the conduct of the test.

b. Photos of the laboratory rig and key components.

c. Equipment calibrations, data and observations.

d. Sample handling and storage details.

e. Results, including all raw and calculated data.

Notes

1. Samples taken from bulk storage, transport tankers or during transfer from or into these vessels shall be treated as original samples.

2. Since the stirrer design can influence the efficiency of the mixing process the stirrer specified in AG:PT/T102 has been selected. When selecting speed range for this task, the speed should be such that the all regions of the container are well mixed without causing a vortex to drag air into the sample. This should be checked on a dummy sample before actual sample preparation is commenced..

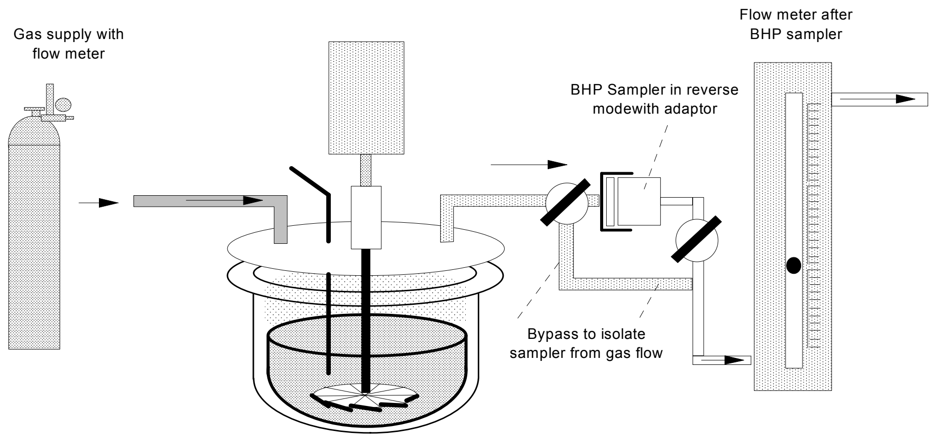

Figure 1. Test Assembly - gas management and fume collection

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 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.

A2 PROCEDURE

The procedure shall be as follows:

a. Insert a temperature probe (see Note A1) into the sample as follows:

b. Punch a hole through the lid of the container See Note A2).

c. Fill the container with the sample and secure on the lid (see Note A3).

d. Allow the sample to cool until it is semi-solid.

e. 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.

f. Cool the sample to room temperature.

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

h. 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).

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

j. Remove the container, cool to room temperature and repeat Steps (c) and (d).

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 sub-sample 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 Glass beakers and metal containers with cut-off rims will require lids.

A3 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.

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.

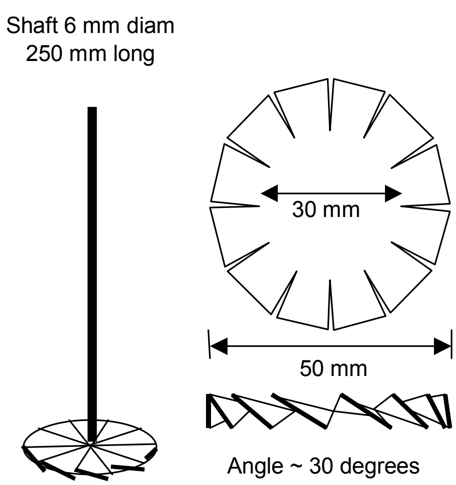

Figure B1. Impeller assembly (Informative)

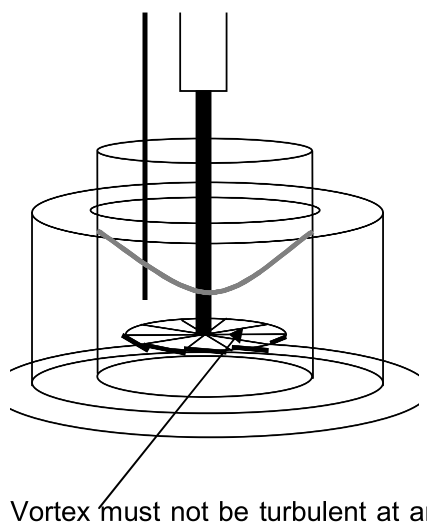

Figure B2. Incipient Vortex

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 using the control function.

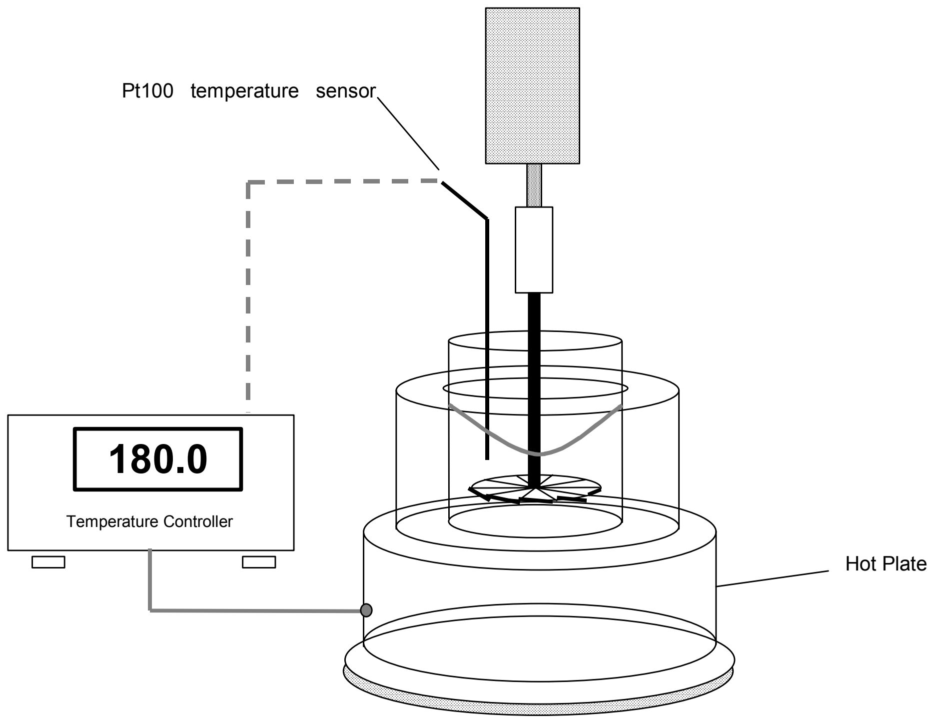

Figure C1. Temperature Control Module and Hotplate Assembly

AMENDMENT RECORD

Amendment No.	Clauses amended	Action	Date
1	This version of the method requires the "mechanical stirrer" from AG:PT/T130 (MBT-02) to be used	New	June 2002
2	Commentary Page	New	Oct 2004
	Footer and header	Format	
	Applied revised test method number	Format	
	Applied new styles	Format	
3	Applied revised test method number	Substitution	March 2006
	Moved notes to end of methods	Format	
4	Corrected references	Substitution	June 2006

Key

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