

# DURABILITY TEST METHOD FOR BITUMEN

## 1. INTRODUCTION

### 1.1 SCOPE

The following test method has been developed to assess the long-term field durability (resistance to oxidative hardening) of NZTA M1 penetration grade bitumens.

*Note: this test method involves the use of hazardous operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 1.2 PRINCIPLE

Films of the test bitumen (1.0 mm thick) are oxidised at 60°C under 2,069 kPa (300 psi) of air for 80 hours. The samples are removed after 80 hours and the dynamic shear modulus is measured at 5°C and 9 Hz and compared to standard values based on the behaviour of Safaniya bitumen of the same NZTA M1 penetration grade.

## 2. TEST METHOD

### 2.1 REFERENCED DOCUMENTS

The following documents are referred to in this method:

NZTA M1:2010 Specification for Roding Bitumens.

### 2.2 APPARATUS AND MATERIALS

2.2.1 Cylinder of industrial-grade compressed dry air, fittings and hose for connection to pressure vessel.

2.2.2 Pressure vessel system comprising:

- Pressure vessel certified for operation at 2069 kPa (300 psi) and 60°C (see Figure 1).
- Thermocouple, thermistor or platinum resistance thermometer, and temperature logging device accurate to  $\pm 0.1^\circ\text{C}$ , to measure the temperature inside the vessel.
- Pressure release valve that prevents the pressure inside the vessel exceeding the maximum design pressure.
- Pressure regulator capable of controlling the pressure inside the vessel at  $2,069 \pm 34$  kPa ( $300 \pm 5$  psi).

- Pressure gauge readable to 10 psi and calibrated at 2069 kPa (300 psi).
- Slow release bleed valve to allow depressurisation of the vessel from the test pressure to atmospheric pressure over five to 10 minutes.



Figure 1: Example Pressure Vessel

- 2.2.3 Stainless steel or aluminium sample holders capable of holding at least 1.0 g of bitumen as a uniform, 1.0mm thick, horizontal film.
- 2.2.4 A metal rack or holder capable of supporting at least three sample holders in a horizontal position so that the bitumen film thickness within the sample holders remains uniform.
- 2.2.5 A stirred temperature-controlled fluid bath or forced-draft oven capable of maintaining the temperature inside the pressure vessel at  $60.0 \pm 0.1^\circ\text{C}$ .
- If a water bath is used it must be fitted with a water inlet and level control device so that a constant water level is automatically maintained over long periods.
  - Both the bath or oven must be sufficiently large to allow air or the bath fluid to freely circulate around the vessel, and contain a shelf or stand to allow the vessel to held in a horizontal (level) position.

- It is recommended is that the position of the vessel relative to the sides of the bath or oven is kept fixed.

2.2.6 A vacuum oven and pumping system capable of maintaining  $100 \pm 5^\circ\text{C}$  and a vacuum of  $<6.9$  kPa.

2.2.7 Dynamic shear rheometer or similar instrument capable of applying sinusoidal loading and measuring the dynamic shear modulus of bitumen at 9 Hz and  $5^\circ\text{C}$ .

2.2.8 Hotplate.

2.2.9 Balance readable and accurate to  $\pm 0.001$  g.

## 2.3 PROCEDURE

### 2.3.1 Initial Setup of Pressure Vessel

- Place the pressure vessel in the  $60^\circ\text{C}$  water bath or oven to be used in the test, and pressurise to  $2,069 \pm$  kPa ( $300 \pm 10$  psi).
- The thermocouple should be positioned near the vertical axis of the vessel and at approximately half height.
- After four hours, log the temperature inside the vessel at 15 minute intervals for at least 24 hours and adjust if necessary so that a temperature of  $60.0 \pm 0.1^\circ\text{C}$  is maintained.

### 2.3.2 Preparation of Sample Bitumen Films

- Weigh a sufficient quantity of bitumen onto the centre of the sample holder to achieve a  $1.0 \pm 0.1\%$  mm film at  $60^\circ\text{C}$ . If the density of the bitumen is not known assume a density of  $1.0$  g  $\text{cm}^{-3}$  at  $60.0^\circ\text{C}$ .
- Heat the sample holder on a hot plate at  $90$ - $110^\circ\text{C}$  for three to four minutes to achieve an even film, and allow to cool to room temperature on a level surface.

### 2.3.3 Ageing the Bitumen Films

- Before the test is begun, ensure that the pressure vessel (containing the sample holder rack) has been maintained at  $60.0^\circ\text{C}$  for at least four hours.
- Remove the pressure vessel from the bath or oven and place the sample holders in the vessel.
- Return the vessel to the bath and pressurise the vessel to  $2069 \pm 34$  kPa ( $300 \pm 5$  psi). The sample loading operation must be completed within five minutes and preferably as rapidly as possible to avoid cooling of the vessel.

- After 15-20 minutes, check the pressure in the vessel and re-adjust if necessary.

### 2.3.4 Removing the Bitumen Films from the Pressure Vessel

- After 80 hours ( $\pm 15$  minutes) allow the pressure vessel to depressurise slowly over about five minutes (to avoid excessive bubbling of the samples), and before removing the vessel from the bath.
- Remove a sample holder and scrape the oxidised film into a small vial. Heat the sample under vacuum ( $< 6.9\text{kPa}$ ) at  $100 \pm 5^\circ\text{C}$  for 30 minutes. Quickly release the vacuum, remove the vial and stir the sample. Store in a freezer.

### 2.3.5 Modulus Measurement

Exact analysis details will vary according to the type of rheometer used but analysis must be consistent from sample to sample.

- The modulus measurement is carried out using an 8mm parallel geometry with a 1.0mm gap. Larger diameter plates can be used if it can be demonstrated that the compliance of the instrument is not significantly affecting the measured modulus.
- A preliminary stress sweep may be necessary to ensure that the strain used lies within the linear viscoelastic region.
- The bitumen is annealed at  $120^\circ\text{C}$  for 10 minutes before testing
- The sample is brought to the test temperature of  $5.0 \pm 0.1^\circ\text{C}$  and the modulus measured at  $9 \pm 0.1\text{Hz}$ .

## 2.4 Reporting

The following information is to be reported:

- Sample identification and testing date.
- Modulus (to the nearest MPa) of the untreated bitumen.
- Modulus (to the nearest MPa) of the oxidised bitumen (the Durability value)

## 2.5 Precision

As a guide, based on measurements made on four different bitumens (ranging from 64 to 186 penetration at  $25^\circ\text{C}$ ), the single operator repeatability of the measured modulus is  $\pm 7\%$ .

## 3. REFERENCES

NZ Transport Agency (2010). *NZTA M1: 2010* (Specification for Roading Bitumens). Wellington: Transit New Zealand.