

Designation: D6521 – 22

# Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)<sup>1</sup>

This standard is issued under the fixed designation D6521; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the conditioning of asphalt binders to simulate accelerated aging (oxidation) by means of pressurized air and elevated temperature. This is intended to simulate the changes in rheology which occur in asphalt binders during in-service oxidative aging, but may not accurately simulate the relative rates of aging. It is normally intended for use with residue from Test Method D2872 (RTFOT), which is designed to simulate plant aging.

Note 1—PAV conditioning has not been validated for materials containing particulate materials.

1.2 The aging of asphalt binders during service is affected by ambient temperature and by mixture-associated variables, such as the volumetric proportions of the mix, the permeability of the mix, properties of the aggregates, and possibly other factors. This conditioning process is intended to provide an evaluation of the relative resistance of different asphalt binders to oxidative aging at selected elevated aging temperatures and pressures, but cannot account for mixture variables or provide the relative resistance to aging at in-service conditions.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard

Note 2—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a

means of evaluating and controlling some of those factors.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- D8 Terminology Relating to Materials for Roads and Pavements
- D2872 Test Method for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)
- D3666 Specification for Minimum Requirements for Agen-22cies Testing and Inspecting Road and Paving Materials
- D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing
- D6373 Specification for Performance-Graded Asphalt Binder
- D8239 Specification for Performance-Graded Asphalt Binder Using the Multiple Stress Creep and Recovery (MSCR) Test
- E1137/E1137M Specification for Industrial Platinum Resistance Thermometers
- 2.2 AASHTO Standards:<sup>3</sup>
- M 320 Specification for Performance-Graded Asphalt Binder
- M 332 Specification for Performance-Graded Asphalt

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<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability and Distillation Tests.

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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

Binder Using Multiple Stress Creep Recovery (MSCR) Test

2.3 CGA Standard:<sup>4</sup>

CGA G-7.1–1997 Commodity Specification for Air, Fourth Edition

# 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 Definitions of terms used in this practice may be found in Terminology D8, determined from common English usage, or combinations of both.

3.1.2 Terms related to standardization, calibration, and verification are defined in Specification D3666.

## 4. Summary of Practice

4.1 Asphalt binder is normally first conditioned using Test Method D2872 (RTFOT). Residue from the RTFOT is then placed in standard stainless steel pans and conditioned at the temperature and pressure specified in Section 10. The residue may then be vacuum degassed. The applied high pressure is intended to improve diffusion of air into the asphalt binder with a focus on oxygen as the reactive component of interest.

Note 3—For more information on selecting the conditioning temperature, see the applicable specification on performance-graded asphalt binder. Examples include Specifications D6373 and D8239, AASHTO M 320, and AASHTO M 332.

## 5. Significance and Use

5.1 This practice is designed to simulate the in-service oxidative aging that occurs in asphalt binders during pavement service. Residue from this conditioning practice may be used to estimate the physical or chemical properties of asphalt binders after several years of in-service aging in the field.

5.2 Binders conditioned using this practice are normally used to determine specification properties in accordance with Specification D6373 or D8239, or AASHTO M 320.

5.3 For asphalt binders of different grades or from different sources, there is no unique correlation between the time and temperature in this conditioning practice and in-service pavement age and temperature. Therefore, for a given set of in-service climatic conditions, it is not possible to select a single PAV conditioning time, temperature, and pressure that will predict the properties or the relative rankings of the properties of asphalt binders after a specific set of in-service exposure conditions.

5.4 The relative degree of hardening of different asphalt binders varies with conditioning temperatures and pressures in the PAV. Therefore, two asphalt binders may age at a similar rate at one condition of temperature and pressure, but age differently at another condition. Hence, the relative rates of aging for a set of asphalts at PAV conditions may differ significantly from the actual in-service relative rates at lower pavement temperatures and ambient pressures.

## 6. Apparatus

6.1 An equipment system consisting of a pressure vessel, ovens, pressure controlling devices, temperature controlling devices, pressure and temperature measuring devices, and a temperature and pressure recording system (see Fig. 1).

6.1.1 *Pressure Vessel*—A stainless steel pressure vessel designed to operate at  $2.1 \pm 0.1$  MPa between 90 and 110 °C, with interior dimensions adequate to hold ten PAV pans and a pan holder. The pan holder shall be capable of holding ten PAV stainless steel pans in a horizontal (level) position, such that the asphalt binder film thickness is reasonably uniform. The holder shall be designed for easy insertion and removal from the vessel when the holder, pans, and asphalt binder are at the conditioning temperature. A schematic showing a possible configuration of the vessel, pan holder, and pans, and specifying dimensional requirements is shown in Fig. 2.

Note 4—The vessel may be a separate unit to be placed in a forced-draft oven for conditioning the asphalt binders or an integral part of the temperature control system (for example, by direct heating of the vessel or by surrounding the vessel with a permanently affixed heating unit, forced-air oven, or liquid bath). For practical purposes, it is recommended that the vessel have the dimensions of 250 mm in diameter and 265 mm in height.

## 6.1.2 Pressure and Temperature Controlling Devices:

6.1.2.1 A pressure relief valve that prevents pressure in the vessel from exceeding the design pressure of the vessel, but in no case exceeding 2.5 MPa during the conditioning procedure. 6.1.2.2 A pressure regulator or regulating system capable of controlling the pressure within the vessel to  $\pm 0.02$  MPa, and with a capacity adequate to reduce the pressure from the source of compressed air, so that the pressure within the loaded pressure vessel is maintained at  $2.1 \pm 0.1$  MPa gauge (relative) pressure during the conditioning process.

6.1.2.3 A slow-release bleed valve or pressure controller that allows the pressure in the vessel at the completion of the conditioning procedure to be reduced from 2.1 MPa to local atmospheric pressure within 8 to 15 min.

6.1.3 *Temperature Controlling Device*—A digital temperature control device as described in 6.1.4.1 or 6.1.4.2 for maintaining the temperature during the conditioning procedure within the pressure vessel at the conditioning temperature  $\pm 0.5$  °C.

6.1.3.1 A heating device (forced-draft oven or fluid bath) capable of restoring the conditioning temperature within the vessel after loading the pans and the pan holder and prior to pressurizing the vessel within 2 h of placing the loaded vessel in the heating device. The device shall be capable of maintaining the temperature within the pressure vessel at the conditioning temperature  $\pm 0.5$  °C. If an oven is used, the oven shall have sufficiently large interior dimensions to allow forced air to freely circulate within the oven and around the pressure vessel when the vessel is placed in the oven. The oven shall contain a stand or shelf that supports the loaded pressure vessel in a level position above the lower surface of the oven.

6.1.3.2 A pressure vessel with an integral temperature control system that is capable of restoring the pre-conditioning temperature, as determined in 9.3, within the vessel after loading the pans and the pan holder, prior to pressurizing the

<sup>&</sup>lt;sup>4</sup> Available from Compressed Gas Association (CGA), 4221 Walney Rd., 5th Floor, Chantilly, VA 20151-2923, http://www.cganet.com.

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FIG. 1 Schematic of PAV Test System

vessel within 2 h of placing the loaded vessel in the heating device, and maintaining the temperature within the pressure vessel at the conditioning temperature  $\pm 0.5$  °C.

Note 5—Preheating the pressure vessel may be necessary to achieve the conditioning temperature within the required 2-h period.

### 6.1.4 Temperature and Pressure Measuring Devices:

6.1.4.1 A platinum resistive thermometric device (RTD) accurate to the nearest 0.1 °C and manufactured in accordance with Specification E1137/E1137M (IEC 751), or equal, for measuring temperature inside the pressure vessel. The RTD shall be calibrated as an integral unit with its respective metre or electronic circuitry.

6.1.4.2 *Temperature Recording Device*—A strip chart recorder or other data acquisition system capable of recording temperature throughout the conditioning process to within  $\pm 0.1$  °C at a minimum interval of once per minute. As an alternative, an electronic device capable of reporting only maximum and minimum temperatures (accurate to  $\pm 0.1$  °C) may be used.

6.1.4.3 A pressure gauge capable of measuring the pressure in the pressure vessel to within  $\pm 0.02$  MPa during the conditioning process.

6.2 *Stainless Steel Pans*—Cylindrical pans, each 140  $\pm$  1 mm (5.5  $\pm$  0.04 in.) in inside diameter and 9.5  $\pm$  1.5 mm (<sup>3</sup>/<sub>8</sub>  $\pm$  <sup>1</sup>/<sub>16</sub> in.) deep, with a flat bottom. Pans shall be manufactured

of stainless steel and shall have a metal thickness of approximately 0.6 mm (0.024 in.).

Note 6—Stainless steel pans, rather than aluminum pans, are required for use in the PAV because they provide a safer environment for hydrocarbons under elevated temperatures and pressures and they are not as easily warped or bent.

Note 7—Pans have a tendency to become warped or bent with use. Although tests show that a slight degree of warping does not significantly affect the results, frequent inspection to eliminate warped or damaged pans is advisable. The indicated metal thickness has been found to provide adequate rigidity.

6.3 *Balance*—A balance that is in accordance with Guide D4753, Class G2.

6.4 *Vacuum Oven*—A vacuum oven capable of maintaining temperature up to 180 °C with an accuracy of  $\pm 5$  °C and 15  $\pm$  1.0 kPa absolute pressure shall be used (see Note 8).

6.4.1 Temperature and Vacuum Measuring Devices:

6.4.1.1 *Temperature Measuring Device*—A temperature sensor capable of measuring the vacuum oven chamber temperature to within  $\pm 5$  °C.

6.4.1.2 Vacuum Measuring Device—A vacuum gauge, absolute pressure gauge, or digital vacuum measuring system capable of measuring the absolute pressure in the chamber to within  $\pm 0.5$  kPa ( $\pm 0.2$  in. Hg).

6.5 *Vacuum System*—A vacuum system capable of generating and maintaining pressure below 15 kPa absolute. Suitable



NOTE 1—Distance "a" controls the levelness of the pan. The assembly shall be supported at three or more support points. The distance "a," measured from each assembly support point to the bottom of the pan (top of shelf or pan support point), shall be controlled to  $\pm 0.05$  mm. NOTE 2—Distances "b<sub>1</sub>" and "b<sub>2</sub>" shall be such that any active portion of the temperature transducer is  $\geq 10$  mm from any adjacent surface. NOTE 3—Distance "c" shall be  $\geq 12$  mm.

#### FIG. 2 Schematic Showing Location of Pans and RTD Within PAV

vacuum systems include a vacuum pump, an air aspirator, or a house vacuum system.

Note 8—A vacuum gauge provides the difference in pressure between ambient atmospheric pressure and the absolute pressure within the vacuum oven. At sea level, where the atmospheric pressure is equal to 101.3 kPa (29.9 in. Hg), and with an absolute pressure inside the oven equal to 15.0 kPa (4.4 in. Hg), the vacuum gauge will read 86.3 kPa (25.5 in. Hg). At an altitude of 1000 m (3281 ft) where the ambient atmospheric pressure is 89.7 kPa (26.5 in. Hg), the vacuum gauge reading will be 26.5 in. Hg minus 4.4 in. Hg or 22.1 in. Hg. A temperaturecorrected altitude conversion for relative pressure gauge indication is to subtract 0.85 in. Hg for each 250 m of altitude (subtract 0.52 in. Hg for each 500 ft of altitude).

6.6 *Oven*—An oven capable of maintaining a temperature of  $168 \pm 5$  °C, readable to 1 °C.

#### 7. Materials

7.1 Commercial bottled air meeting at least the minimum requirements of the CGA for Grade D air, and having a maximum dew point to -40 °C.

Note 9—In North America, CGA Grade D air is commonly referred to as *OSHA breathing air*. CGA Publication G-7.1–1997 defines Grade D air as containing 19.5 to 23.5 % oxygen, balance being predominantly nitrogen. Carbon dioxide (CO<sub>2</sub>) is limited to 1000 ppm (v/v), carbon monoxide is limited to 10 ppm, and oil (condensed) to 5 mg/m<sup>3</sup> at NTP.

#### 8. Hazards

8.1 Use standard laboratory safety procedures in handling the hot asphalt binder when preparing and conditioning specimens and removing the residue from the pressure vessel. Use special precaution when lifting the pressure vessel.

### 9. Calibration and Standardization

#### 9.1 Temperature Sensors:

9.1.1 *PAV Thermometric Device*—Verify the calibration of the thermometric device to within  $\pm 0.1$  °C at least every six months using a calibrated thermometric device traceable to a national standard. Verification shall be performed near the temperature of use within a range of 90.0 to 110.0 °C.

NOTE 10-The sensors for the thermometric devices in commercially manufactured PAV vessels cannot, or to avoid damage to the sensors and fittings, should not be removed for verification. A suitable verification technique is to: (1) bring the probe of the calibrated thermometric device into intimate contact with the sensor mounted in the PAV vessel, (2) place the cover over the vessel, allowing the leads for the thermometric device to exit the vessel under the cover without securing the cover, (3) allow the vessel to come to thermal equilibrium so that the temperature is constant to  $\pm 0.1$  °C, (4) after equilibrium is reached, simultaneously record the temperature of the two thermometric devices at 1-min intervals until the temperature differential readings differ by no more than 0.1 °C for three consecutive readings, and (5) record the average of the three readings for each device. The difference in the average readings for the two devices is the correction factor to be applied when selecting the conditioning temperature. Post and date a notice on the PAV apparatus that contains the correction to be applied when setting conditioning temperature. Alternatively, if the PAV thermoelectric device so allows, adjust the calibration of the device so that the indicator and the calibrated temperature measuring device indicate the same temperature.

Note 11—If the sensor in the PAV is oriented in a horizontal direction, a block of brass with dimensions approximately 25 mm by 25 mm by 25 mm, with holes drilled in two adjacent faces to accommodate the sensors, may be used to thermally couple the two sensors.

9.1.2 Vacuum Oven Thermometric Device—Verify the calibration of the thermometric device used in the vacuum oven to