Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the accelerated aging (oxidation) of asphalt binders 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—Modified asphalt binders may phase separate or form skins during oven conditioning in Test Method D2872 (RTFOT); the results from subsequent testing of this residue may not be representative of modified asphalts short-term aged under field conditions. Phase separation, or formation of skins, or both can also occur during PAV conditioning. Therefore, the practice may not be suitable for some modified asphalts.

NOTE 2—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 the standard. Values in parentheses in inch-pound units are provided for informational purposes only.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D8 Terminology Relating to Materials for Roads and Pavements

D2872 Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)

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

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

2.2 *AASHTO Standards:*[3]

M 320 Specification for Performance-Graded Asphalt Binder

2.3 *CGA Standards:*[4]

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

3. Terminology

3.1 *Definitions:*

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.

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 aged at the specified conditioning temperature for 20 h in a vessel pressurized with air to 2.10 MPa. The conditioning temperature is selected according to the grade of asphalt binder. The residue is then vacuum degassed.

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[1] 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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[2] 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.

[3] 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.

[4] Available from Compressed Gas Association (CGA), 4221 Walney Rd., 5th Floor, Chantilly, VA 20151-2923, http://www.cganet.com.

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 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 ± 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 3—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.

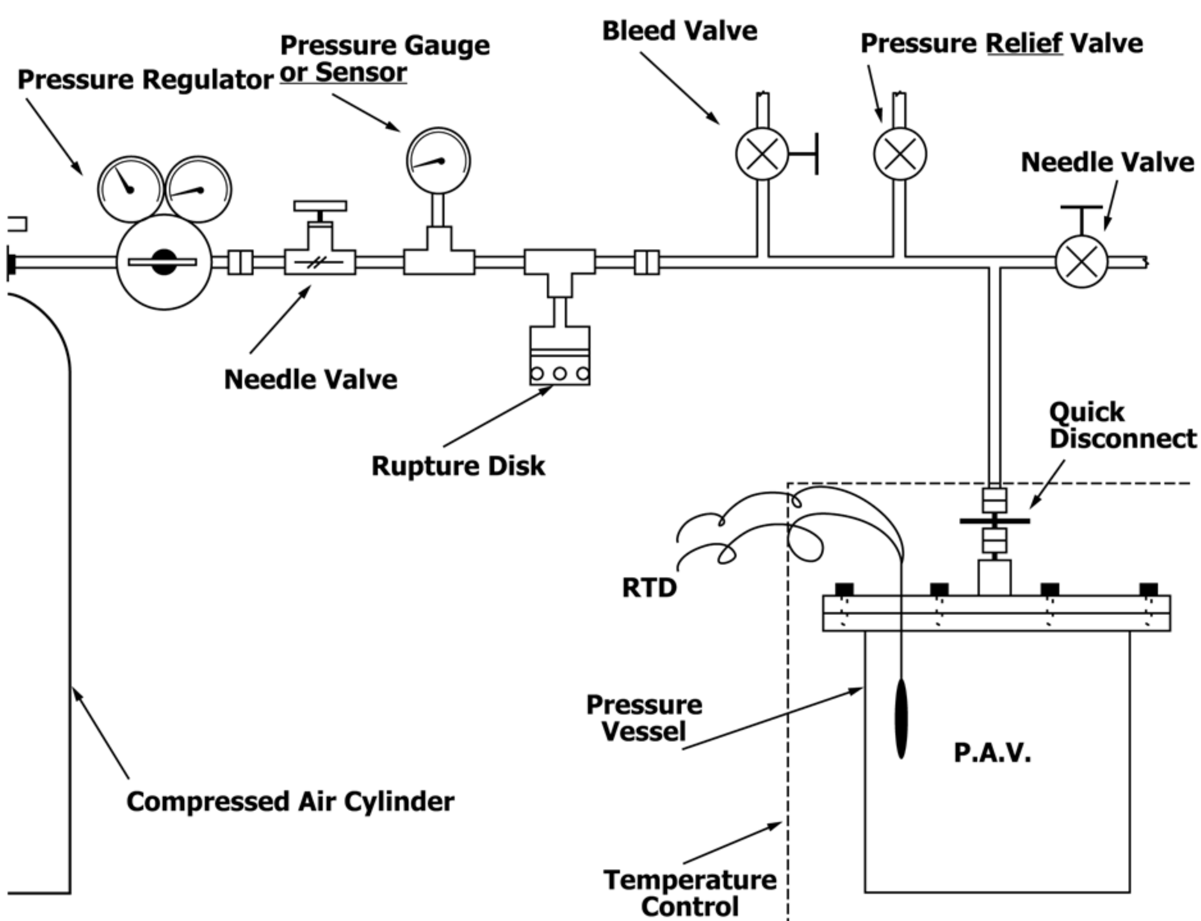
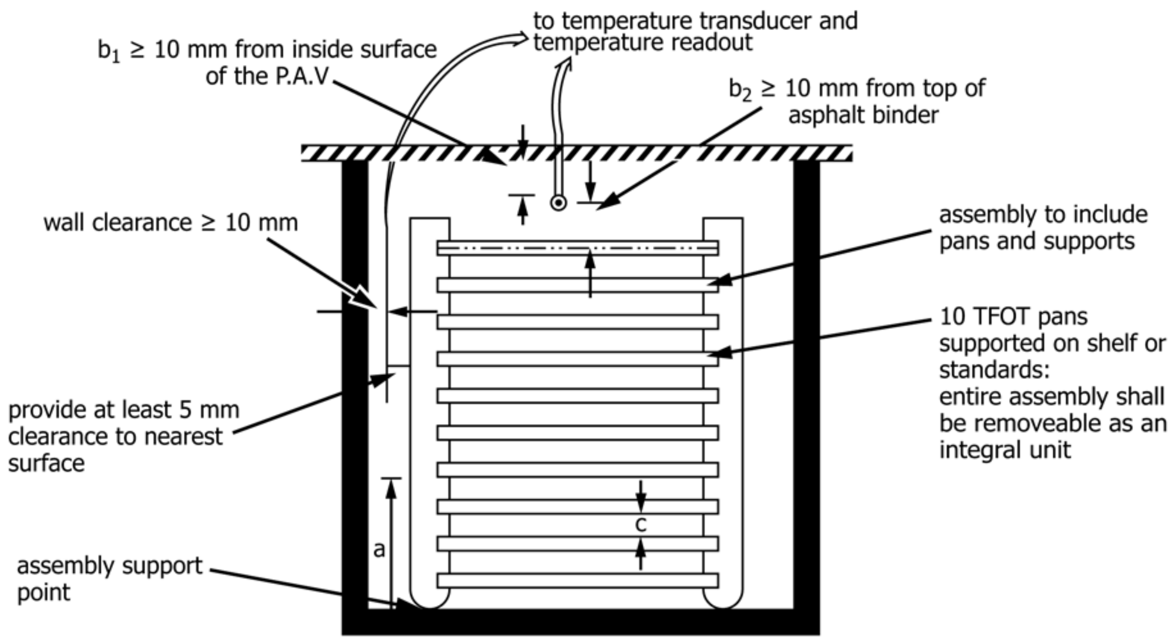


FIG. 1 Schematic of PAV Test System



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 ±0.05 mm.

NOTE 2—Distances b_1 and b_2 shall be such that any active portion of the temperature transducer is ≥10 mm from any adjacent surface.

NOTE 3—Distance "c" shall be ≥12 mm.

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

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 ±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 ± 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 ±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 ±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 vessel within 2 hours of placing the loaded vessel in the heating device, and maintaining the temperature within the pressure vessel at the conditioning temperature ±0.5°C.

NOTE 4—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 ±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 ±0.1°C) may be used.

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

6.2 *Stainless Steel Pans*—Cylindrical pans, each 140 ± 1 mm (5.5 ± 0.04 in.) in inside diameter and 9.5 ± 1.5 mm (3/8 ± 1/16 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 5—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 6—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 ±5°C and 15 ± 1.0 kPa absolute pressure shall be used (see Note 7).

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 ±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 ±0.5 kPa (±1.0 in. Hg).

6.5 *Vacuum System*—A vacuum system capable of generating and maintaining pressure below 15 kPa absolute. Suitable vacuum systems include a vacuum pump, an air aspirator, or a house vacuum system.

NOTE 7—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 temperature-corrected 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 ± 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 8—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 19.5–23.5 % oxygen, balance being predominantly nitrogen. Carbon dioxide (CO_2) is limited to 1000 ppm (v/v), carbon monoxide is limited to 10 ppm and oil (condensed) to 5 mg/m^3 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 ±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 9—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 ± 0.1°C, *4)* after equilibrium is reached, simultaneously record the temperature of the two thermometric devices at one minute 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 10—If the sensor in the PAV is oriented in a horizontal direction, a block of brass with dimensions approximately 25 mm × 25 mm × 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 within ±1°C at least every six months using a calibrated thermometric device traceable to a national standard. Verification shall be performed at a temperature that is within 10°C of the use temperature.

9.2 *Pressure and Vacuum Gauges*—

9.2.1 *PAV Pressure Gauge*—Verify the calibration of the pressure gauge or digital pressure measurement system to within ±0.02 MPa at least every six months using a calibrated pressure indicator traceable to a national standard. Verification shall be performed near the pressure of use within a range of 2.00 to 2.10 MPa.

9.2.2 *Vacuum Oven Vacuum or Absolute Pressure Gauge*—Verify the calibration of the chamber vacuum or absolute pressure gauge or digital vacuum measurement system to equate to a reading within ±0.5 kPa (±0.2 in Hg) absolute pressure at least every six months using a calibrated vacuum or pressure indicator traceable to a national standard. Verification shall be performed near the absolute pressure of use within a range of 12.5 to 17.5 kPa. (See Note 7).

9.3 *Levelness*—The relative levelness of samples is important to the outcome of the conditioning process.

9.3.1 Verify the levelness of the sample rack itself by placing it on a measured level surface and measuring the levelness of the top shelf of the rack. A spirit level – circular with a "bulls-eye" is preferred. However, a small (approximately 150 mm or 6 in.) machinist's level may be used, but levelness must be verified in more than one plane (that is, at right angles).

9.3.2 Verify the levelness of the sample rack as it rests in the pressure vessel. The pressure vessel and rack should both be heated to normal operating temperature before performing this verification. Verification is similar to that described in 9.3.1, except that the PAV oven supports or PAV system leveling supports must be adjusted according to the manufacturer's recommendations to provide levelness.

9.4 *Standardization*—For those vessels or PAV systems where pressurization is operator-controlled, use the following procedure to determine the optimum temperature at which to apply pressure to the pressure vessel. Several conditioning runs should be conducted. With the vessel loaded with pan rack and empty pans, increase the temperature inside the vessel to the conditioning temperature. When the temperature inside the vessel is within 10°C of the conditioning temperature, apply an air pressure of 2.1 ± 0.1 MPa. Record the temperature increase when the pressure is applied. Perform the procedure at least three times and use the average temperature increase to establish the temperature at which to apply pressure to the vessel for performing the conditioning procedure. This information will be useful in 10.9.

10. Procedure

10.1 Place the pan holder inside the pressure vessel. If an oven is used, place the pressure vessel inside the oven, select a conditioning temperature, and preheat the pressure vessel to the conditioning temperature selected. If an integrated temperature control pressure vessel is used, one should select a conditioning temperature and follow the manufacturer's instructions for preheating the pressure vessel.

NOTE 11—If conditioning asphalt binders for conformance to Specification D6373 or AASHTO M 320, select the appropriate conditioning temperature from Table 1 or 2 of Specification D6373 or Table 1 or 2 of AASHTO M 320.

NOTE 12—For vessels placed in an oven, preheating the vessel 10 to 15°C above the conditioning temperature can be used to reduce the drop in PAV temperature during the loading process and minimize the time required to stabilize the system, after loading, to attain the required temperature.

NOTE 13—Conditioning temperature in the PAV is selected to account for different climatic regions. Temperatures in excess of approximately 115°C can change the chemistry of asphalt binders using an accelerated aging practice and should be avoided.

10.2 If required, condition the asphalt binder in accordance with Test Method D2872 (RTFOT).

10.3 Combine the hot residue from the RTFOT bottles into a single container, stir to blend, and then transfer to PAV pans in accordance with 10.4 for PAV conditioning, or allow the hot residue in the container to cool to room temperature and cover and store at room temperature for PAV conditioning at a later date. If conditioned asphalt binder is allowed to cool to room temperature, heat it until it is sufficiently fluid to pour and stir it before pouring it into the PAV pans.

10.4 Place each PAV pan on a balance and add 50 ± 0.5 g mass of asphalt binder to the pan. This will yield approximately a 3.2-mm thick film of asphalt binder.

NOTE 14—The mass change is not measured as part of this procedure. Mass change is not meaningful because the asphalt binder absorbs air as a result of pressurization. Any loss in mass as a result of volatilization is masked by air absorbed by the binder as a result of the pressurization.

10.5 If an oven-heated vessel is preheated to other than the desired conditioning temperature, reset the temperature control on the oven to the conditioning temperature.

10.6 Perform the operations described in 10.7 and 10.8 as quickly as possible to avoid cooling of the vessel and pan holder.

10.7 Place the filled pans in the pan holder. Pans containing asphalt binders from different sources and grades may be placed in the pressure vessel during a single conditioning run. Place the panholder with filled pans inside the pressure vessel and close the pressure vessel. Unused slots in the pan holder need not be filled with empty pans.

10.8 If an oven is used, place the loaded and closed pressure vessel in the oven. Connect the temperature transducer line and the air pressure supply line to the loaded pressure vessel's external connections as required by the vessel design and oven configuration.

10.9 For pressure vessels placed in an oven, wait until the temperature inside the pressure vessel reaches the specified temperature minus the value determined in 9.4, apply an air pressure of 2.10 ± 0.1 MPa and then start timing the conditioning run. If an integrated temperature control pressure vessel is used, follow the manufacturer's instructions regarding the desired preheating temperature to pressurize the vessel to 2.1± 0.1 MPa and start timing the conditioning run. If the temperature inside the vessel has not reached the desired temperature for applying pressure within 2 h of loading the pan holder and pans, discontinue the procedure and discard the asphalt samples.

NOTE 15—Pressures in excess of 2.1 MPa do not substantially increase the rate of aging. Therefore, higher pressures are not warranted.

NOTE 16—Once pressurized, the temperature inside the pressure vessel will equilibrate rapidly. The time under pressure, not to include any preheating time at ambient pressure, is the conditioning time. Relatively little aging occurs at ambient pressure during the time that the vessel is being preheated to the conditioning temperature, given that the asphalt binder residue being aged has normally already been exposed to 163°C in the RTFOT.

10.10 Maintain the temperature and air pressure inside the pressure vessel for 20 h ± 10 min.

10.11 If the temperature indicated by the temperature recording device rises above or falls below the target conditioning temperature ±0.5°C for more than a total of 60 min during the 20-h conditioning period, declare the conditioning process invalid and discard the material. Further, if the temperature indicated by the temperature recording device varies from the target aging temperature by more than 5°C for a total of 10 min, declare the conditioning process invalid and discard the material. If the pressure at the end of the conditioning period is outside the range designated in 10.9, declare the conditioning process invalid and discard the material. If a device capable of recording only minimum and maximum temperatures was used and if either the maximum or the minimum temperature recorded during the 20-h period varies by more than ±0.5°C from the conditioning temperature, declare the conditioning process invalid and discard the material.

10.12 At the end of the 20-h conditioning period, begin the slow reduction of the internal pressure of the PAV, using the air pressure bleed valve. The bleed valve should be preset to an opening that requires 8 to 15 min to equalize the internal and external pressures on the PAV, thus avoiding excessive bubbling and foaming of the asphalt binder. Alternatively, the pressure controller can be programmed to release the pressure slowly over 15 min. Do not include the pressure release and equalization time as part of the 20-h conditioning period.

10.13 Vacuum degas the aged samples.

10.13.1 Remove the pan holder and pans from the PAV, and place the pans in an oven set to 168 ± 5°C for 15 ± 1 min.

10.13.2 Preheat the vacuum oven to 170 ± 5°C.

10.13.3 Remove the pans from the oven and scrape the hot residue from all pans containing the same sample into a single container. Select a container of dimensions such that the depth of the residue in the container is between 15 and 40 mm. After the last pan of a sample has been scraped, and if additional containers are to be prepared, transfer the container to the 168°C warming oven. After all of the containers have been prepared, transfer them to the vacuum oven within one min. Once the last container has been placed in the vacuum degassing oven, maintain the temperature at 170 ± 5°C for 15 ± 1 min.

NOTE 17—It is imperative that the binder be heated before it is subjected to a vacuum. Once the vacuum is applied, there will be little heat transferred to the sample.

10.13.4 Open the valve to the vacuum system as rapidly as possible to reduce the pressure to 15 ± 2.5 kPa absolute (see Note 7). Maintain the absolute pressure at 15 ± 2.5 kPa for 30 ± 1 min. Release the vacuum and remove the container. If any bubbles remain on the surface, remove them by flashing the surface of the PAV residue with a torch or hot knife.

10.14 If tests to determine the properties of the degassed PAV residue are not performed immediately, it is permissible to cover and store the samples in their containers at room temperature for future testing. No studies have been performed to determine the best point in the practice to pause if not all of the conditioning and subsequent testing is to be completed in one continuous sequence. Therefore, it is also acceptable to scrape the aged residue from the PAV pans into the containers to be used for degassing, and then (*1*) allow the sample to cool, and degas on another day, or (*2*) degas the aged material and then allow the sample to cool. The critical steps before testing the conditioned samples are: (*1*) reheating the aged asphalt to 168 ± 5°C for a maximum of 30 min prior to using the sample for subsequent tests, and (*2*) stirring the sample to ensure homogeneity.

11. Report

11.1 Report the following information:

11.1.1 Sample identification,

11.1.2 Conditioning temperature, nearest 0.5°C,

11.1.3 Maximum and minimum conditioning temperature recorded, nearest 0.1°C,

11.1.4 Total time during conditioning that the temperature was outside the specified range, nearest minute,

11.1.5 Total conditioning time, hours and minutes, and

11.1.6 The heating temperature and heating time if temperatures greater than 163°C are required at any time during the handling of the material.

12. Keywords

12.1 accelerated aging; elevated temperature; in-service aging; PAV; pressure aging; pressure aging vessel; vacuum degassing