Standard Practice for

Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)

AASHTO Designation: R 28-12

AASHTO

1. SCOPE

1.1. This standard practice covers the accelerated aging (oxidation) of asphalt binders by means of pressurized air and elevated temperature. The practice is intended to simulate in-service oxidative aging of asphalt binders and is intended for use with residue from T 240 (RTFOT).

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

1.3. *This standard practice may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:
- M 231, Weighing Devices Used in the Testing of Materials
- M 320, Performance-Graded Asphalt Binder
- T 240, Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)

2.2. *ASTM Standard*:
- E220, Standard Test Method for Calibration of Thermocouples By Comparison Techniques

2.3. *Deutsche Industrie Norm (DIN) Standard*:
- 43760, Standard for Calibrating Thermocouples

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. *asphalt binder*—an asphalt-based cement that is produced from petroleum residue either with or without the addition of nonparticulate organic modifiers.

3.1.2. *in-service*—refers to aging of the asphalt binder that occurs in the pavement as a result of the combined effects of time, traffic, and the environment.

4. SUMMARY OF PRACTICE

4.1. Asphalt binder is first aged using T 240 (RTFOT). A specified thickness of residue, from the RTFOT, is then placed in stainless steel pans and aged at the specified aging temperature for 20 h in a vessel pressurized with air to 2.10 MPa. The aging temperature is selected according to the grade of the asphalt binder. At the completion of the PAV process, the asphalt binder residue is then vacuum degassed.

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 practice may be used to estimate the physical or chemical properties of asphalt binders after 5 to 10 years of in-service aging in the field.

5.2. Asphalt binders aged using R 28 are used to determine specification properties in accordance with M 320. The asphalt binder is aged with the RTFO test prior to this conditioning step. Tank asphalt binders, as well as RTFOT and residue from this practice, are used to determine specification properties in accordance with M 320.

5.3. For asphalt binders of different grades or from different sources, there is no unique correlation between the aging time and temperature in this 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 aging time and temperature that will predict the properties of all asphalt binders after a specific set of in-service exposure conditions.

5.4. The relative degree of hardening of different asphalt binders varies at different aging temperatures in the PAV. Therefore, two asphalt binders may age similarly at one temperature, but age differently at another temperature.

6. APPARATUS

6.1. A test system consists of a pressure vessel, pressure-controlling devices, temperature-controlling devices, pressure- and temperature-measuring devices, and a temperature-recording device (Figure 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 stainless steel pans and a pan holder. The pressure vessel shall contain a pan holder capable of holding ten stainless steel pans in a horizontal (level) position such that the asphalt binder film thickness in the bottom of the pans does not vary by more than 0.5 mm across any diameter of the pan. The holder shall be designed for easy insertion and removal from the vessel when the holder, pans, and asphalt binder are at the test temperature. A schematic showing the vessel, pan holder and pans, and specifying the dimensional requirements is shown in Figure 2.

Note 1—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).

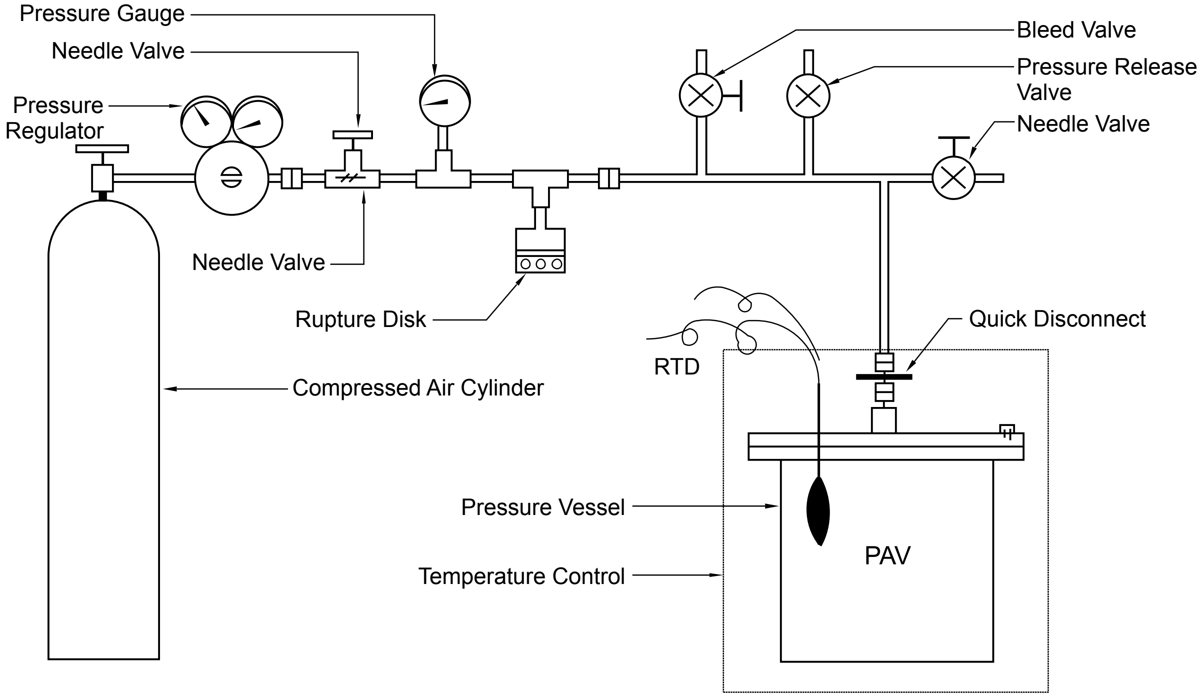
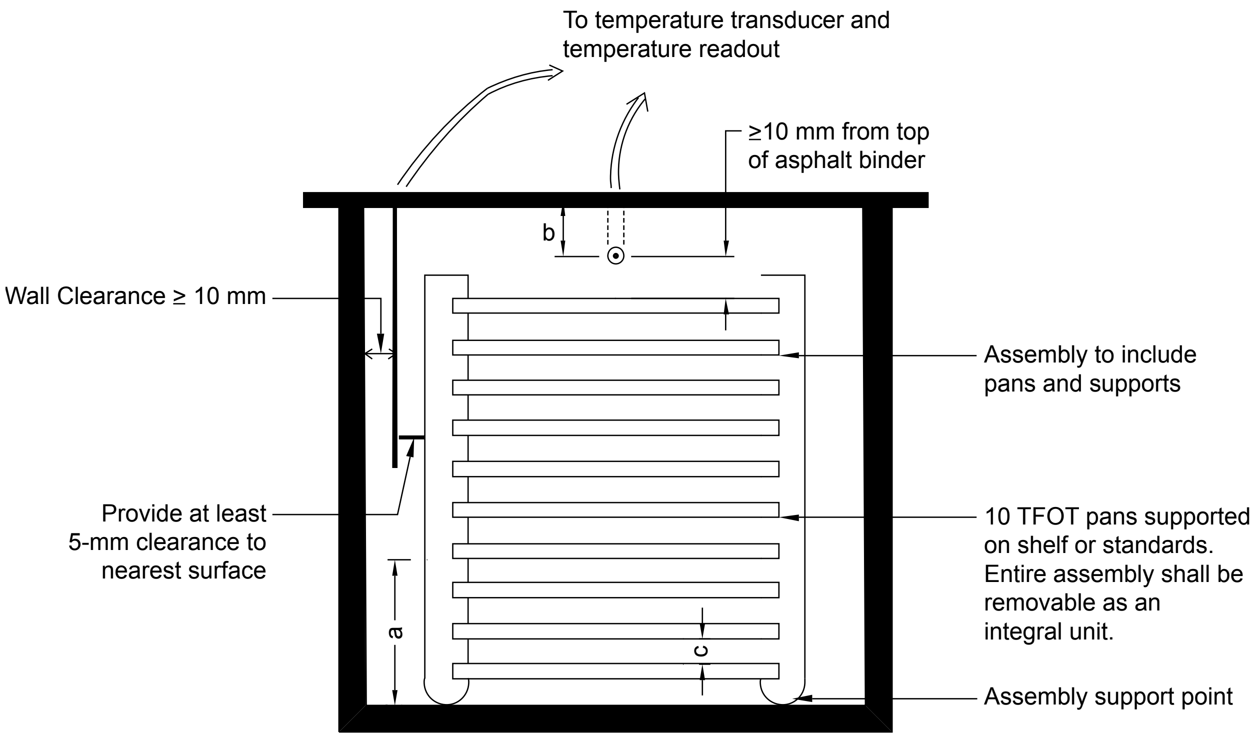


Figure 1—Schematic of Typical PAV Test System



Notes:
1. Distance "a" controls the levelness of the pans. 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. Provisions shall be made to ensure that the bottom of the vessel is leveled so that the thickness of the binder in the pan varies by no more than ±0.05 mm across the diameter of any pan.
2. Distance "b" shall be such that any active portion of the temperature transducer is ≥10 mm from the top surface of the vessel.
3. Distance "c" shall be ≥12 mm.

Figure 2—Schematic Showing Location of Pans and RTD within Representative PAV

6.1.2. *Pressure-Controlling Devices*:

6.1.2.1. A pressure release valve that prevents pressure in the vessel from exceeding 2.2 MPa during the aging procedure.

6.1.2.2. A pressure regulator capable of controlling the pressure within the vessel to ±1 percent, 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 during the practice.

6.1.2.3. A slow release bleed valve that allows the pressure in the vessel at the completion of the conditioning procedure to be reduced at an approximately linear rate from 2.1 MPa to local atmospheric pressure within 9 ± 1 min.

6.1.3. *Temperature-Controlling Devices*—A temperature control device as described in Section 6.1.3.1 or Section 6.1.3.2 for maintaining the temperature during the aging procedure at all points within the pressure vessel at the aging temperature ±0.5°C and a digital proportional controller for maintaining the specified temperature control.

6.1.3.1. A forced-draft oven or fluid bath capable of (1) bringing the loaded unpressurized vessel to the desired conditioning temperature ±0.5°C, as recorded by the Resistance Thermal Detector (RTD) inside the vessel within 2 h, and (2) maintaining the temperature at all points within the pressure vessel at the aging temperature ±0.5°C. 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 (i.e., maintains the film thickness in the aging pans within the specified tolerance).

6.1.3.2. A pressure vessel with integral temperature control system that is capable of (1) bringing the loaded pressure vessel to the desired conditioning temperature ±0.5°C within 2 h, as recorded by the RTD inside the loaded pressure vessel, and (2) maintaining the temperature at all points within the pressure vessel at the aging temperature ±0.5°C.

6.1.4. *Temperature- and Pressure-Measuring Devices*:

6.1.4.1. A platinum RTD accurate to the nearest 0.1°C and meeting DIN Standard 43760 (Class A), or equal, for measuring temperature inside the pressure vessel. The RTD shall be calibrated as an integral unit with its respective meter or electronic circuitry.

Note 2—The RTD or thermistor and its meter may be calibrated by the manufacturer or a commercial vendor. Verification can be obtained by comparing the output from the RTD with an NIST traceable ASTM 94C mercury-in-glass thermometer in accordance with ASTM E220. A stirred fluid bath is suitable for calibrating the thermal detector. Select a partial immersion mercury-in-glass thermometer with an appropriate range and place the thermal detector and the thermometer in the stirred water bath. Fasten the detector to the glass thermometer with a rubber band or rubber O-ring. Allow the bath, detector, and thermometer to come to thermal equilibrium and record the temperature of the glass thermometer and the readout from the thermal detector. The temperature in the bath shall not change by more than 0.1°C/min during the calibration process.

6.1.4.2. *Temperature-Recording Device*—A strip chart recorder or other data acquisition system capable of recording temperature throughout the test to 0.1°C. As an alternative, an electronic device capable of reporting 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 ±1 percent during the test.

6.2. *Stainless Steel Pans*—Ten standard stainless steel pans with an inside diameter of 140 mm ($5^1/_2$ in.) and 9.5 mm ($^3/_8$ in.) deep with a flat bottom. Pans shall be made of stainless steel and should have a metal thickness of approximately 0.635 mm (0.025 in.).

6.3. *Balance*—A balance conforming to the requirements of M 231, Class G 2.

6.4. *Vacuum Oven*—A vacuum oven capable of maintaining a temperature up to 180°C with an accuracy of ±5.0°C and a vacuum of 1.0 kPa absolute.

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

7. MATERIALS

7.1. Commercial bottled air or equivalent.

8. HAZARDS

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

9. CALIBRATION AND STANDARDIZATION

9.1. *Temperature Detector*—Verify the calibration of the RTD to 0.1°C at least every 6 months using a calibrated thermometer.

9.2. *Pressure Gauge*—Standardize the pressure gauge to an accuracy of 1 percent at least every 6 months.

Note 3—The pressure gauge is usually calibrated by the manufacturer or a commercial calibration service. Verification of the continued stability of the pressure gauge within the specified requirements should be done periodically by checking against another certified pressure measurement device.

9.3. To determine the optimum temperature at which to apply pressure to the pressure vessel, several tests should be made. With the vessel loaded with pan rack and empty pans, increase the temperature to aging temperature. When the temperature inside the pressure vessel is within 10°C of the aging 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 aging procedure.

10. PROCEDURE

10.1. Condition the asphalt binder in accordance with T 240 (RTFOT).

10.2. After combining the RTFOT residue into a single container and blending as specified in T 240; (1) pour the hot residue directly into the stainless steel pans for immediate conditioning in the PAV, or (2) pour the residue into the stainless steel pans, cover and set aside for conditioning at a later time, or (3) allow the residue to cool in the single container for conditioning at a later time. If the residue is allowed to cool in the pans, reheat the pans and residue to the conditioning temperature. If the residue is stored in a single container, heat the residue, stir gently, and pour the heated residue into the pans.

10.3. Place the pan holder inside the pressure vessel. If an oven is used, place the pressure vessel inside the oven. If an integrated temperature control pressure vessel is used, turn on the heater. Select an aging temperature and preheat the pressure vessel to the aging temperature selected.

Note 4—If conditioning asphalt binders for conformance to M 320, select the appropriate aging temperature from Table 1 of M 320.

Note 5—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 6—Aging 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 aged in accelerated tests and should be avoided.

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

Note 7—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 gain in mass as a result of oxidation is masked by air absorbed by the asphalt binder as a result of the pressurization.

10.5. If the vessel is preheated to other than the desired aging temperature, reset the temperature control on the heating device to the aging temperature.

10.6. 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 test.) Place the pan holder with filled pans inside the pressure vessel, and close the pressure vessel.

10.7. If an oven is used, place the loaded and closed pressure vessel in the oven.

10.8. Connect the temperature transducer line and the air pressure supply line to the loaded pressure vessel's external connections.

10.9. Perform the operations described in Sections 10.5 to 10.8 as quickly as possible to avoid cooling of the vessel and pan holder.

10.10. Wait until the temperature inside the pressure vessel is within 20°C of the aging temperature, apply an air pressure of 2.1 ± 0.1 MPa, and then start timing the test. If the temperature inside the vessel has not reached the desired temperature for applying pressure within 2 h of loading the pan holders and pans, discontinue the procedure and discard the asphalt samples.

Note 8—Pressures in excess of 2.1 MPa do not substantially increase the rate of aging. Therefore, higher pressures are not warranted.

Note 9—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 aging time. Relatively little aging occurs at ambient pressure during the time that the vessel is being reheated to the test temperature, given that asphalt binder residue under test has been exposed to 163°C in the RTFOT.

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

10.12. At the end of the 20-h test period, slowly begin reducing the internal pressure of the PAV, using the air pressure bleed valve. Adjust the bleed valve to an opening that requires 9 ± 1 min to equalize the internal and external pressures on the PAV, thus avoiding excessive bubbling and foaming of the asphalt binder. During this process it may be necessary to adjust the setting of the needle valve as the pressure drops in order to maintain an approximate linear rate of pressure decrease. Do not include the pressure release and equalization time as part of the 20-h aging period.

10.13. If the temperature indicated by the temperature-recording device falls above or below the target aging temperature ±0.5°C for more than 60 min during the 20-h aging period, declare the test invalid and discard the material.

10.14. Remove the pan holder and pans from the PAV, and place the stainless steel pans in an oven set at a minimum temperature for a minimum time until sufficiently fluid to pour. Stir the residue in the pan gently to assist in the removal of air bubbles. If the binder is conditioned in multiple pans, pour the hot residue into a single container. Scrape residue remaining in the pans into the container, followed by gentle stirring to blend the residue. If the sample must be heated to temperatures greater than 175°C to facilitate pouring, note the temperature and heating time in the report.

Note 10—A temperature of 163°C has been found to be suitable for many binder grades.

10.15. *Vacuum Degassing (Optional)*—Preheat the vacuum oven until it stabilizes at 170 ± 5°C. Follow the instructions in Section 10.14, except select a container of dimensions such that the depth of the residue in the container is between 15 and 40 mm.

10.15.1. After the binder has been combined into a single container, immediately transfer the container to the vacuum oven and maintain the temperature in the vacuum oven at 170 ± 5°C for 15 ± 1 min, without a vacuum applied. After the 15 ± 1 min of equilibration, open the vacuum valve as rapidly as possible to reduce the pressure in the oven to 15 ± 2.5 kPa absolute. Maintain the absolute pressure in the oven at 15 ± 2.5 kPa for 30 ± 1 min. At the end of the 30 min, release the vacuum and remove the container. If any bubbles are visible on the surface of the residue, remove them by flashing the surface of the residue with a torch or hot knife.

10.16. *Correction to Gauge Reading for Elevation*—A vacuum gauge attached to the vessel indicates the difference between atmospheric pressure and the pressure in the vessel. If the vessel is located above sea level, the gauge reading must be corrected for altitude to indicate the correct absolute pressure within the vessel. This correction is not used if the vessel is fitted with an absolute pressure gauge. Do not correct the reading for temperature or for the barometric pressure reported by a weather station as this pressure is typically already corrected for elevation.

10.16.1. Correct the vacuum gauge reading to sea level using a correction factor in accordance with Table 1. If an absolute pressure gauge is used, no correction is needed.

Note 11—At sea level, standard atmospheric pressure is 29.92 in.Hg, 760 mmHg, 1.013 kPa, or 14.7 psi.

Note 12—If the material foams over the lip of the container during the degassing, reduce the rate at which the vacuum is released until the foaming ceases.

Table 1—Gauge Readings Corrected for Elevation

Elevation (m)	Corrected Gauge Readings (mmHg)		Corrected Gauge Readings (kPa)	
	(min)	(max)	(min)	(max)
0	26.3	24.9	83.8	88.8
50	26.1	24.7	83.2	88.2
100	26.0	24.6	82.6	87.6
150	25.8	24.4	82.0	87.0
200	25.6	24.2	81.4	86.4
250	25.4	24.0	80.8	85.8
300	25.3	23.9	80.2	85.2
350	25.1	23.7	79.7	84.7
400	24.9	23.5	79.1	84.1
450	24.8	23.4	78.5	83.5
500	24.6	23.2	77.9	82.9
550	24.4	23.0	77.4	82.4
600	24.3	22.9	76.8	81.8
650	24.1	22.7	76.2	81.2
700	23.9	22.5	75.7	80.7
750	23.8	22.4	75.1	80.1
800	23.6	22.2	74.6	79.6
850	23.4	22.0	74.0	79.0
900	23.3	21.9	73.4	78.4
950	23.1	21.7	72.9	77.9
1000	22.9	21.5	72.4	77.4
1050	22.8	21.4	71.8	76.8
1100	22.6	21.2	71.3	76.3
1150	22.5	21.1	70.7	75.7
1200	22.3	20.9	70.2	75.2
1250	22.1	20.7	69.7	74.7
1300	22.0	20.6	69.1	74.1
1350	21.8	20.4	68.6	73.6
1400	21.7	20.3	68.1	73.1
1450	21.5	20.1	67.6	72.6
1500	21.4	20.0	67.0	72.0
1550	21.2	19.8	66.5	71.5
1600	21.1	19.7	66.0	71.0
1650	20.9	19.5	65.5	70.5
1700	20.8	19.4	65.0	70.0
1750	20.6	19.2	64.5	69.5

Elevation (ft)	Corrected Gauge Readings (mmHg)		Corrected Gauge Readings (kPa)	
	(min)	(max)	(min)	(max)
0	26.3	24.9	83.8	88.8
200	26.1	24.7	83.1	88.1
400	25.9	24.5	82.3	87.3
600	25.7	24.3	81.6	86.6
800	25.5	24.1	80.9	85.9
1000	25.3	23.9	80.2	85.2
1200	25.0	23.6	79.5	84.5
1400	24.8	23.4	78.8	83.8
1600	24.6	23.2	78.1	83.1
1800	24.4	23.0	77.4	82.4
2000	24.2	22.8	76.7	81.7
2200	24.0	22.6	76.0	81.0
2400	23.8	22.4	75.3	80.3
2600	23.6	22.2	74.6	79.6
2800	23.4	22.0	74.0	79.0
3000	23.2	21.8	73.3	78.3
3200	23.0	21.6	72.6	77.6
3400	22.8	21.4	72.0	77.0
3600	22.6	21.2	71.3	76.3
3800	22.4	21.0	70.6	75.6
4000	22.2	20.8	70.0	75.0
4200	22.0	20.6	69.3	74.3
4400	21.9	20.5	68.7	73.7
4600	21.7	20.3	68.1	73.1
4800	21.5	20.1	67.4	72.4
5000	21.3	19.9	66.8	71.8
5200	21.1	19.7	66.2	71.2
5400	20.9	19.5	65.5	70.5
5600	20.7	19.3	64.9	69.9
5800	20.6	19.2	64.3	69.3
6000	20.4	19.0	63.7	68.7
6200	20.2	18.8	63.1	68.1
6400	20.0	18.6	62.5	67.5
6600	19.8	18.4	61.9	66.9
6800	19.7	18.3	61.3	66.3
7000	19.5	18.1	60.7	65.7

Note: Based on an absolute pressure equal to 15 ± 2.5 kPa or 4.7 ± 0.7 in.Hg. Calculations based on the following equation:

$pa = po\ \{1 - [(1 - (H)(0.0065/288.16)]^{5.2561}\}$, where pa = pressure at altitude, po = pressure at sea level, H = altitude in meters.

Reference: *Manual of Barometry* (WBAN), Vol. 1, First Edition, U.S. Dept. of Commerce, Weather Bureau, Washington, DC, 1963.

10.17. From the residue generated in Sections 10.14 or 10.15, prepare test specimens directly from the residue in the container, pour the residue from the container (Section 10.14) to subdivide into smaller containers for future testing, or set the container aside for future testing.

11. REPORT

11.1. *Report the following information*:

11.1.1. Sample identification;

11.1.2. Aging test temperature, nearest 0.5°C;

11.1.3. Maximum and minimum aging temperature recorded, nearest 0.1°C;

11.1.4. Total time during aging that temperature was outside the specified range, nearest minute;

11.1.5. Total aging 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. PRECISION AND BIAS

12.1. *Precision*—The research required to develop precision estimates for tests performed on PAV residue has not been conducted.

12.2. *Bias*—The research required to establish the bias of tests performed on PAV residue has not been conducted.

13. KEYWORDS

13.1. Accelerated aging; elevated temperature; in-service aging; PAV; pressure aging; pressure aging vessel.