Designation: D 6521 – 03a

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

This standard is issued under the fixed designation D 6521; 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 type of changes which occur in asphalt binders during in-service oxidative aging but may not accurately simulate the relative rates of aging. It is intended for use with residue from Test Method D 2872 (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 D 2872 (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 aging. Therefore, the practice may not be suitable for some modified asphalts.

NOTE 2—PAV has not been validated for materials containing particulate materials.

1.2 The aging of asphalt binders during service is affected by ambient temperature and air pressure 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.

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]

D 8 Terminology Relating to Materials for Roads and Pavements

D 1754 Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)

D 2872 Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)

D 4753 Specification for Evaluating, Selecting and Specifying Balances and Scales for Use in Soil, Rock and Construction Materials Testing

D 6373 Specification for Performance Graded Asphalt Binder

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques

E 1137 Specification for Industrial Platinum Resistance Thermometers

2.2 *CGA Standards:*

CGA G-7.1–1997 Commodity Specification for Air, Fourth Edition[3]

3. Terminology

3.1 *Definitions*:

3.1.1 Definitions of terms used in this practice may be found in Terminology D 8 determined from common English usage, or combinations of both.

4. Summary of Practice

4.1 Asphalt binder is first aged using Test Method D 2872 (RTFOT). A specified thickness of residue from the RTFOT is then placed in standard TFOT stainless steel pans and aged at the specified aging temperature for 20 h in a vessel pressurized

[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 Test.

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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 CGA, Compressed Gas Association, Inc., Chantilly, VA 20151.

with air to 2.10 MPa. Aging temperature is selected according to the grade of this asphalt binder. The residue is then vacuum degassed.

5. Significance and Use

5.1 This method is designed to simulate the type of 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 aged using this standard practice are used to determine specification properties in accordance with Specification D 6373.

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 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 aging time and elevated temperature and pressure that will predict the properties or the relative rankings of 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 with aging 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 temperatures and pressures.

6. Apparatus

6.1 An equipment system consisting of a pressure vessel, 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 TFOT pans and a pan holder. The pressure vessel shall contain a pan holder capable of holding ten TFOT 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 aging 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

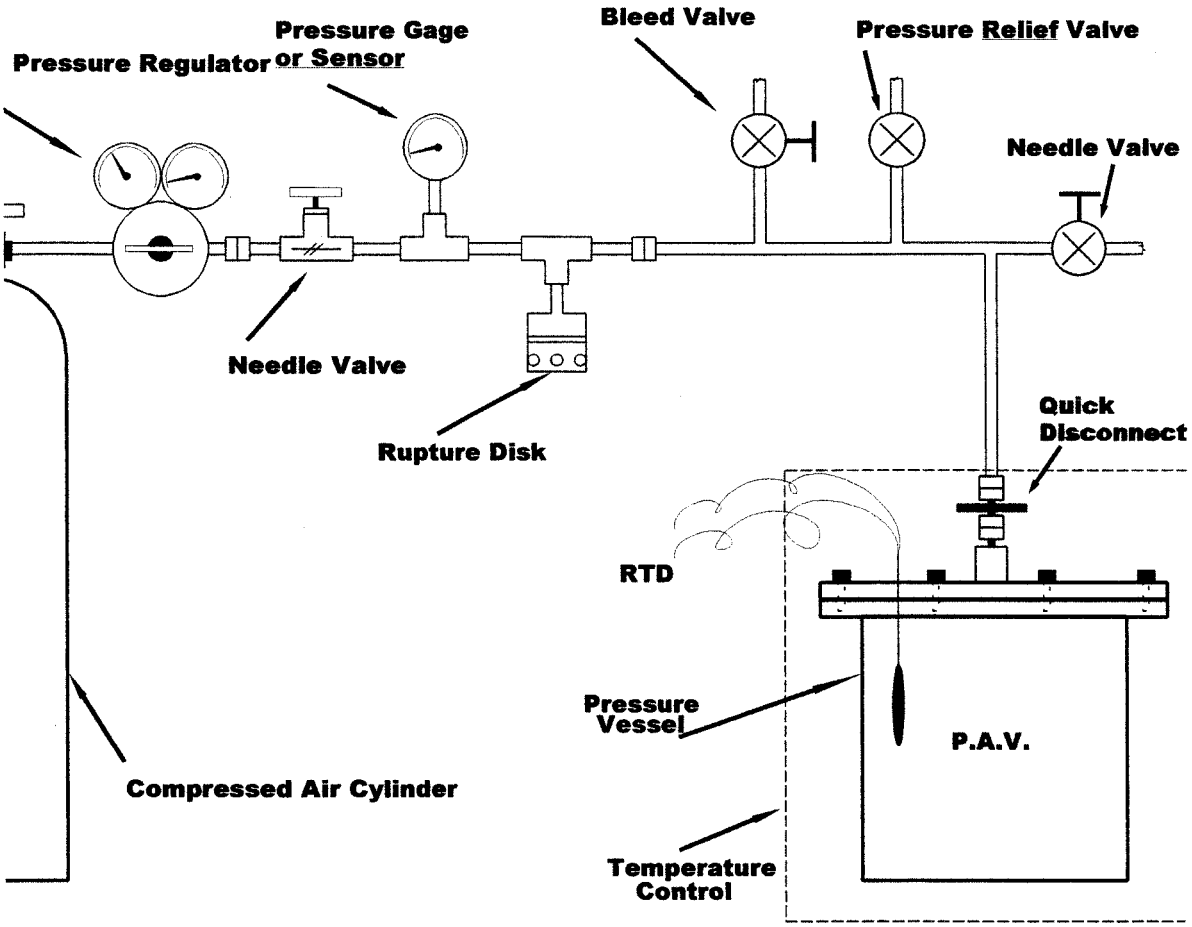
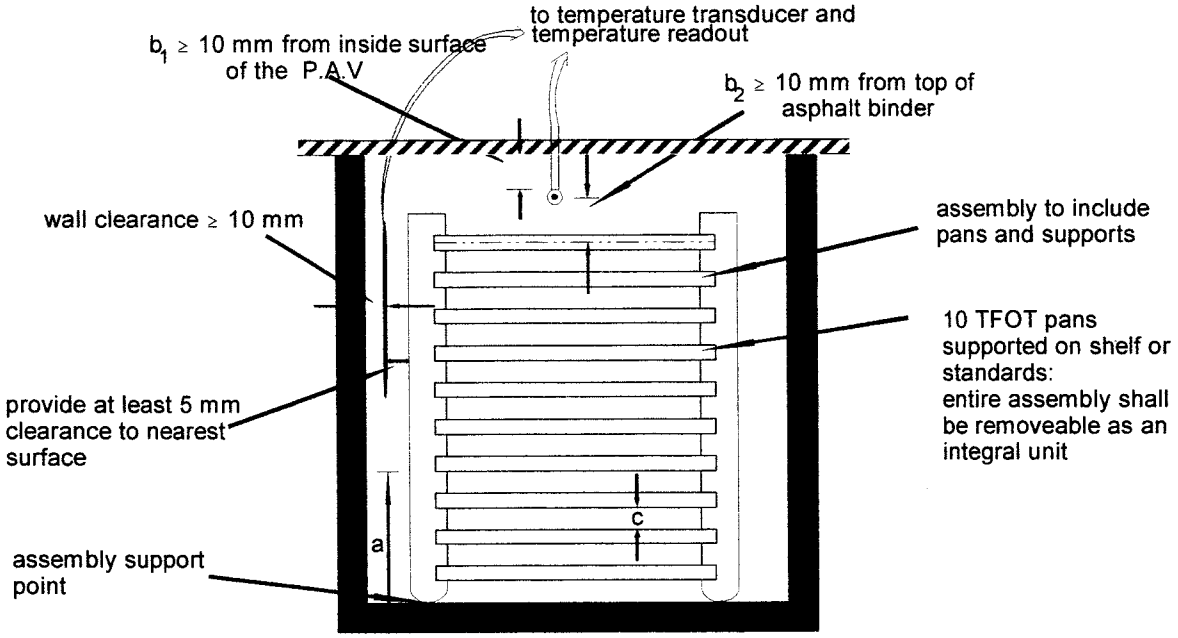


FIG. 1 Schematic of PAV Test System



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

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 has the dimensions of 250 mm in diameter and 265 mm in height. Research has shown that the volume of the vessel is not an important factor in hardening due to aging.

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 aging 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 gage (relative) pressure during the aging process.

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 from 2.1 MPa to local atmospheric pressure within 8 to 15 min.

6.1.3 *Temperature Controlling Devices*—A digital temperature control device as described in 6.1.3.1 or 6.1.3.2 for maintaining the temperature during the aging procedure within the pressure vessel at the aging temperature ± 0.5°C.

6.1.3.1 A heating device (forced-draft oven or fluid bath) capable of restoring the aging 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. Maintain the temperature within the pressure vessel at the aging 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 which supports the loaded pressure vessel in a level position above the lower surface of the oven.

6.1.3.2 A pressure vessel with integral temperature control system that is capable of restoring the pre-aging temperature, as determined in 9.3, within the vessel after loading the pans and the pan holder and 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 aging temperature ± 0.5°C.

NOTE 4—Preheating the pressure vessel may be necessary to achieve the aging temperature within the required 2 h period.

6.1.4 *Temperature and Pressure Measuring Devices*:

6.1.4.1 A platinum RTD accurate to the nearest 0.1°C and meeting Specification E 1137 (IEC 751), 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.

6.1.4.2 *Temperature Recording Device*—A strip chart recorder or other data acquisition system capable of recording temperature throughout the aging process to 0.1°C. 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 gage capable of measuring the pressure in the pressure vessel to within ±0.02 MPa during the aging process.

6.2 *Stainless Steel Pans*—Ten standard stainless steel TFOT pans meeting the requirements of Test Method D 1754.

NOTE 5—Stainless steel pans are required for use in the PAV because

they provide a safer environment for hydrocarbons under elevated temperatures and pressures.

6.3 *Balance*—A balance conforming to the requirements of Specification D 4753, Class G2.

6.4 *Vacuum Oven*—A vacuum oven capable of maintaining temperature up to 180°C with an accuracy of ± 5°C and vacuum of 15 ± 1.0 kPa absolute shall be used.

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.

7. Materials

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

NOTE 6—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 is predominantly nitrogen. Carbon dioxide (CO_2) is limited to 1 000 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 aging 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 and its respective meter or electronic circuitry to 0.1°C at least every six months using a calibrated temperature indicator traceable to a national standard.

NOTE 7—The RTD or thermistor and its meter may be calibrated by the manufacturer or a commercial vendor. One suggested method is to perform the following steps: (*1*) 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; (*2*) Fasten the detector to the glass thermometer; (*3*) Allow the bath, detector, and thermometer to come to thermal equilibrium; (*4*) 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 per minute during the calibration process); (*5*) If necessary, adjust the calibration of the digital indicator so that the indicator and the thermometer indicate the same temperature, or post a correction notice to be used in adjusting the selected aging temperature to compensate for any error.

9.2 *Pressure Gage*—Calibrate the pressure gauge to an accuracy of 0.02 MPa at least every six months.

NOTE 8—The pressure gage is usually calibrated by the manufacturer or a commercial calibration service. Verification of the continued stability of the pressure gage within the specified requirements should be done periodically by checking against another certified pressure measurement device.

9.3 For those vessels or PAV systems where pressurization is operator-controlled, determine the optimum temperature at which to apply pressure to the pressure vessel. Several aging runs should be conducted. With the vessel loaded with pan rack and empty pans, increase the temperature inside the vessel to the aging temperature. When the temperature inside the 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. This information will be useful in 10.9.

10. Procedure

10.1 Condition the asphalt binder in accordance with Test Method D 2872 (RTFOT).

10.2 Combine the hot residue from the RTFOT bottles into a single container, stir to blend, then transfer to TFOT 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 TFOT pans.

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

NOTE 9—If conditioning asphalt binders for conformance to AASHTO MP1, select the appropriate aging temperature from Table 1 of MP1.

NOTE 10—For vessels placed in an oven, preheating the vessel 10 to 15°C above the aging 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 11—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 using an accelerated aging practice and should be avoided.

10.4 Place the TFOT 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 12—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 binder as a result of the pressurization.

10.5 If an oven-heated vessel is preheated to other than the desired aging temperature, reset the temperature control on the oven to the aging 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 aging 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 9.3, apply an air pressure of 2.10 ± 0.1 MPa and then start timing the aging 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 aging 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 13—Pressures in excess of 2.1 MPa do not substantially increase the rate of aging. Therefore, higher pressures are not warranted.

NOTE 14—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 preheated to the aging temperature, given that the asphalt binder residue being aged has 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 aging temperature ± 0.5°C for more than a total of 60 min during the 20 h aging period, declare the aging process invalid and discard the material. If the pressure at the end of the aging period is outside the range designated in 10.9, declare the aging 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 aging temperature, declare the aging process invalid and discard the material.

10.12 At the end of the 20-h aging 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. Do not include the pressure release and equalization time as part of the 20-h aging period.

10.13 Vacuum de-gas the aged samples

10.13.1 Remove the pan holder and pans from the PAV, and place the pans in an oven set to 163°C for 15 ± 1 min.)

NOTE 15—PAV residue may be heated in the TFOT pans to 163°C and stirred to remove air bubbles. These precautions will help avoid oxidative hardening and volatile loss that will harden the sample. During the heating process, the sample should be covered and stirred occasionally scraping the bottom of the pans to ensure homogeneity.

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

10.13.3 Remove the pans from the oven and pour the hot residue from the pans containing a single 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 is scraped, transfer the container to the vacuum oven within one minute. Maintain the temperature vacuum oven at 170 ± 5°C for 10 ± 1 min.

10.13.4 Open the vacuum valve as rapidly as possible to reduce the pressure to 15 ± –2.5 kPa absolute. 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.

NOTE 16—A pressure of 15 ± 2.5 kPa absolute is equal to a relative pressure gage indication of –25.5 ± 0.75 in. Hg at standard sea level temperature and pressure. This value changes with altitude, that is, at an altitude of 1 000 m (3 281 ft) the relative pressure gage indication will be approximately —22.3 ± 0.75 in. Hg at an absolute pressure of 15 ± 2.5 kPa. A temperature-corrected altitude conversion for relative pressure gage indication is to subtract 0.80 in. Hg for each 250 m of altitude [subtract 0.67 in. Hg for each 500 ft of altitude].

10.14 If tests to determine the properties of the degassed PAV residue are not performed immediately, it is permissible cover and store the samples in their containers at room temperature for future testing.

11. Report

11.1 Report the following information:

11.1.1 Sample identification,

11.1.2 Aging 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 Since this is a practice and material is not tested, precision and bias statement are not needed.

13. Keywords

13.1 accelerated aging; elevated temperature; inservice aging; PAV; pressure aging; pressure aging vessel; vacuum degassing