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Practice for Accelerated Aging of Leather[1]

This standard is issued under the fixed designation D8137; 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 is based on studies relating the rate of deterioration of leathers having known durabilities from long-term storage of samples, to the deterioration experienced by laboratory exposure of specimens to known contents of acid gases in air or oxygen. This is accomplished by measuring the deterioration (reduction in tensile strength) of leather specimens when they are subjected to exposure to a mixture of air, moisture and sulfur dioxide at a given temperature and pressure and for a given exposure time. The loss of tensile strength of the specimens resulting from this exposure is compared to that experienced by a variety of leathers having various tannages and having historically long and short service lifetimes. The initial tensile strength and the degree of loss of tensile strength is related to what service life can be anticipated from a given leather.

1.2 *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.*

1.3 *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:*[2]

D1517 Terminology Relating to Leather

D1610 Practice for Conditioning Leather and Leather Products for Testing

D1813 Test Method for Measuring Thickness of Leather Test Specimens

D2209 Test Method for Tensile Strength of Leather

3. Terminology

3.1 For definitions of leather terms used in this Practice refer to Terminology D1517.

4. Reagents

4.1 *Sulfur Dioxide (SO_2),* Anhydrous, 99.9 % pure.

4.2 *Air,* From compressor, filtered, stored at ~21°C (saturated water vapor conditions) 90 psi to 100 psi delivery pressure.

5. Summary of Practice

5.1 Specimens are prepared and placed in a pressure chamber containing known or constant quantities of air, moisture and SO_2. They are held in the chamber at a constant temperature for a given time period. After that processing, the tensile strengths of the specimens are measured along with the tensile strengths of unprocessed specimens of the same leather. The loss of tensile strength resulting from the processing relative to that of the unprocessed specimens (%) is determined and may be directly compared to that of other leathers considered for an application. Where applicable, the results may also be considered as the basis for projected service lifetimes as shown in Section 11.

6. Significance and Use

6.1 A substantial difficulty in using leather in applications requiring very long service lives is estimating how well a particular leather will actually hold up in service. Such applications may include use in musical instruments such as pipe organs, bookbinding leathers, etc. Use of leather in pipe organs in the past (prior to approximately 1930) demonstrated service lives frequently over 100 years, and the consequences of short service lives can result in extremely costly repairs. Many post-1930 leathers have had short service lives (as little as 15 years) due to the use of various more modern tannages and processing methods. Identifying exactly what tannage and processing was used in a particular leather and how successful this tanning and processing was can be very difficult. Failure of bookbinding and upholstery leathers formed the impetus for extensive work by leather chemists in the 1940s to identify tests that could be used to verify the durability of leather

[1] This practice is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.05 on Upholstery.

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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.

samples. Early studies by Cheshire[3] and Frey & Beebe[4] resulted in tests relating the rate of deterioration of leathers having known durabilities from long-term storage of samples, to the deterioration experienced by laboratory exposure of specimens to known contents of acid gases in air or oxygen. They were considered to be applicable to leathers having a wide range of tannages and processing. Later work published by Piltingsrud & Tancous[5] described their modifications to those tests. Further work directed towards verifying the durability of leathers used in pipe organs resulted in the practice described in this document. The appropriateness of its use for any given leather samples must be determined by the leather chemists utilizing the practice. This practice may not be applicable for leathers having unusual tannages or treatments. Estimates of service lives made using this practice are speculative, as it would take many decades of natural aging to verify the results (see comments in Section 11).

7. Apparatus

7.1 The reaction pressure vessel (RPV) is made up of 316 Alloy Stainless Steel, Schedule 10, 3 in. pipe components welded together. These consist of two end caps (one end cap has a 1 in. hole in it), a 12 in. section of pipe, and a 1 in. National Pipe Thread (NPT) union (cut in half). These are welded together using inert gas welding and alloy 316 stainless steel filler rod.

7.2 *The RPV Valve*—The valve used has ⅜ in. NPT threads and is made of alloy 316 stainless steel. It is a positive shutoff needle valve rated for pressures up to 1000 psi. It is used with a 1- to ⅜-in. NPT 316 stainless steel reducer bushing.

7.3 *RPV Pressure Gauge*—The gauge used for measurements of pressure in the RPV and for filling measurements must have a range up to 125 psi, and an accuracy of ±2.0 psi.

7.4 *Die*—Leather cutting punch type for test strip ¼ in. wide by 2 in. long.

7.5 *Oven*—The RPV must be kept at a constant temperature throughout the exposure period of the test. This is best accomplished by placing the RPV in an oven having mechanical air circulation, and with an accurate electronic temperature control with the capability of maintaining a temperature of approximately ±1.0 °C, and a uniformity of ±4.0 °C. The uniformity isn't quite as important as the stability, as the thermal conductivity and thermal capacity of the RPV aids in providing thermal uniformity inside the RPV. The temperature calibration of the oven should be checked using a thermocouple thermometer. Many commercial laboratory ovens are capable of this performance.

7.6 *Compressor-air Storage Tank System*—A typical compressor-air storage tank system having a tank volume of at least 100 L is quite sufficient. Make sure that there is some water in the bottom of the tank (normal for most compressor-air storage tanks that are operated frequently) so that air stored in the tank at room temperature comes to equilibrium near 100 % relative humidity at laboratory room temperature. This helps to ensure that the initial humidity in the RPV during runs is consistent run to run. The day before the test is accomplished, the air source should be checked to ensure that there is an adequate pressure to be able fill the RPV to 100 psi and that there is sufficient time before the run (at least 12 h) for the air in the tank to reach room temperature and humidity equilibrium. The tank pressure is normally set to 120 psi allowing sufficient pressure to regulate down to the desired 100 psi in the RPV.

8. Specimens

8.1 Cut the specimens using a ¼ by 2 in. cutting die. Cut 15 samples for aging test if possible (see Fig. 1). Cut another 15 specimens for control reference. Specimens need to be cut with the same orientation parallel or perpendicular to the backbone of the hide and in a similar location such that the specimens have similar properties. Leather should be conditioned in accordance with Test Method D1610.

8.2 Process only one type of leather in the RPV at a time.

9. Procedure

9.1 *Assembly of the Reaction Pressure Vessel (RPV):*

9.1.1 Make sure that no foreign materials are present in the interior of the RPV. If there are any residues present or you are not sure if the vessel is clean, use cleaning methods you would ordinarily use for stainless steel laboratory ware used for analytical chemistry purposes.

9.1.2 Make sure the interior of the RPV is dry by placing it in the oven (at 60 °C) for 15 min, without the valve installed. Cool the RPV to room temperature before placing specimens in it.

9.1.3 Place the specimens to be treated into the RPV.

9.1.4 Using polytetrafluoroethylene (PTFE) tape sealant, prepare the valve head threads for assembly into the RPV. Make sure all old sealant is removed using a stainless steel brush before applying the PTFE tape. The PTFE tape is wrapped around the threads, several layers thick, depending on the thickness of the tape (see Fig. 2). Methods used should result in leak free joints (see section on leak checking).

FIG. 1 Specimens for Aging Test

[3] Chesire, A., "The Aging of Leather," *Journal of the International Leather Trades Chemists*, June 1946, Vol. 30, No. 6, pp. 134-166.

[4] Frey, R. W., and Beebe, C. W., "A Proposed Standard Gas Chamber for Accelerated Aging of Leather," *The Journal of the American Leather Chemists Association*, 1940, Vol. 35, pp. 180-192.

[5] Piltingsrud, H. V., and Tancous, J., "The Development of a Standard Accelerated Aging Test for Measuring the Durability of Leathers Used in Musical Instruments," *The Journal of the American Leather Chemists Association*, 1987, Vol. 82 (9), pp. 277-310.

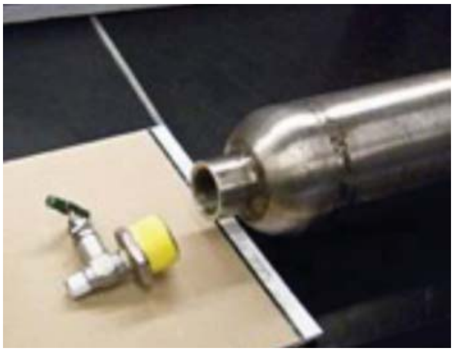

FIG. 2 Preparing the Valve Head Threads

9.1.4.1 Insert valve head into the RPV. Threads must be tightened sufficiently so that there is no leakage when tested later by the soap bubble method. Normally tightening is accomplished by applying a locking chain clamp around the body of the RPV and a 12 in. crescent wrench on the 1 ⅜ in. hex head of the valve head assembly (see Fig. 3). Normally, the assembly is placed on the floor and tightened as needed (as tight as an average person can tighten).

9.1.5 Attach the pressure gauge and the filling port (see Fig. 4) to the top of the RPV valve using PTFE tape as a sealant (see Fig. 5)

FIG. 4 The Pressure Gauge and Filling Port

FIG. 5 Attaching the Pressure Gauge and Filling Port to the RPV

9.2 *Filling the RPV with SO_2:*

9.2.1 Remove the ¼ in. NPT plug from the SO_2 tank. Insert the mating connector into the threaded port on the SO_2 tank using PTFE tape as a sealant. Insert purging connector (a fitting that mates with the connector on the hose, and has a through passage allowing gas to escape) into the opposite end of the SO_2 filling hose.

9.2.2 Place the RPV, SO_2 tank, and filling apparatus in a fume hood that has been determined to be suitable for working with the SO_2 used in this test. Open the SO_2 tank valve slightly allowing the SO_2 to purge the air from the interior of the SO_2 filling hose (see Fig. 6). Close the SO_2 tank valve and remove the purging connector from the SO_2 filling hose.

9.2.3 Connect the SO_2 filling hose to the filling port on the RPV, making sure the gauge reads zero before connecting to the SO_2 filling hose (See Fig. 7).

9.2.4 Open the RPV valve completely (wide open) and slowly open the SO_2 tank valve to the point resulting in a pressure increase on the pressure gauge of approximately 1 psi every 2 s. Allow the pressure in the RPV to increase to 14.7 psi ± 0.2 psi. Then rapidly close the valve on the tank and the RPV.

9.2.5 Disconnect the RPV from the SO_2 filling hose.

9.3 *Filling the RPV with Compressed Air:*

9.3.1 Briefly place a purging port in the compressed air tank outlet port to allow a small amount of air to bleed out of the

FIG. 3 Tightening of the Valve Head

FIG. 6 Purging the Air from the Interior of the SO_2 Filling Hose

FIG. 7 Connecting to the SO_2 Filling Hose

tank. This will allow you to see where the pressure regulator is actually set, in case there has been leakage through the regulator building up pressure on the lower pressure side. Make sure that the pressure regulator is set several psi below the 100 psi desired setting so the pressure can then be raised up to the correct 100 psi setting once the pressure gauge and RPV are connected.

9.3.2 Zero the pressure gauge.

9.3.3 Insert the filling port into the pressure tank outlet port and observe the pressure on the RPV pressure gauge (see Fig. 8).

9.3.4 Slowly increase the pressure regulator pressure setting so that the pressure reading on the RPV pressure gauge is 100 psi.

9.3.5 Open the valve to the RPV allowing the RPV to be fully filled to 100 psi.

9.3.6 Close the valve to the RPV and remove the RPV from the outlet port of the air tank.

9.3.7 Remove the pressure gauge assembly from the RPV valve.

9.4 *Leak Checking:*

9.4.1 Use a soap water solution for leak checking. Apply soap water solution to the exposed threads of the RPV and the valve assembly (see Fig. 9). Use a liquid dishwashing detergent diluted to approximately a 10 % solution with water.

9.4.2 Carefully examine the bubble size for growth, which could indicate a leak.

9.4.3 With a drop of soap solution on your finger tip attempt to lace a soap water film over the exposed port of the valve. Any increase in the size of the film would indicate an internal leak of the valve. Any detected leaks should be corrected. Possible causes could be insufficient tread sealant, insufficient tightening of the joints, a defective valve, etc.

9.4.4 Apply new PTFE tape to the exposed threads on the valve and place the stainless steel cap on the valve outlet (see Fig. 10).

9.5 *Placing the RPV in the Oven:*

9.5.1 Preheat the oven to the desired operating temperature (60 °C) before placing the RPV in the oven. The oven can be any oven with forced air circulation that can maintain the RPV at 60 °C ± 1 °C. Shown in Fig. 11 and Fig. 12 is a special purpose oven made for the task. Many laboratory ovens with forced air circulation should be sufficient.

9.5.2 Place the RPV in the oven as shown in Fig. 11.

9.6 *Aging Process:*

9.6.1 The RPV is left at the desired operating temperature for 168 h (1 week).

9.6.2 The RPV should be rotated 180° every 24 h.

9.6.3 At the end of one week the RPV is removed from the oven and allowed to cool to room temperature. The stainless steel cap is removed from the valve assembly. Zero the pressure gauge and connect it to the valve outlet.

9.6.4 Open the valve and record the pressure reading. Pressure readings are taken before the RPV is placed in the oven and when it is removed, as a check to ensure that the RPV has not leaked during the time in the oven. It is normal to see some drop in pressure due to the consumption of the reagent gasses in the RPV (oxygen and SO_2) reacting with the leather specimens in the RPV. Pressure drops up to 5 % have been observed, depending on the mass of leather and the amount of chemically induced deterioration experienced by the leather.

9.6.5 Close the valve and remove the pressure gauge.

FIG. 8 Filling the RPV with Compressed Air

FIG. 9 Soap Water Solution Leak Testing