Designation: G 114 – 98

Standard Practice for Aging Oxygen-Service Materials Prior to Ignitibility or Flammability Testing[1]

This standard is issued under the fixed designation G 114; 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 methods to determine the influence of time and stress on a material's oxygen compatibility.

1.2 This practice addresses both methods that have a foundation of experience and potential methods that have yet to be verified for validity. The latter are included to promote research and later elaboration in this practice as methods of the former type.

1.3 The values stated in SI units are to be regarded as the standard, however, all numerical values must also be cited in the systems in which they were actually measured.

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. Specific precautionary statements are given in Section 9.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4809 Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Intermediate Precision Method)[2]

G 72 Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment[3]

G 74 Test Method for Ignition Sensitivity of Materials to Gaseous Fluid Impact[3]

G 86 Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Pressurized Oxygen Environments[3]

G 125 Test Method for Measuring Liquid and Solid Material Fire Limits in Gaseous Oxidants[3]

2.2 *Federal Standard:*

Federal Specification BB-0-925 Oxygen, Technical, Gas and Liquid[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aging*—the exposure of a material to stress, such stress of which may include time, pressure, contact with materials or chemicals, temperature, abrasion, ionizing radiation, light, impact with gas or particles, tensile, or compressive force (either static or cyclic), or any other feature that may be present during a material's service life. These stressors may be present individually or in combination.

3.1.2 *artificial aging*—aging in which a stress variable is outside the domain of exposure that the material might see in a component for oxygen service or in which an alternative mechanism is used to produce an effect that simulates the results of natural aging. The degree of artificiality may vary on a large scale. An example of mild artificiality might be exposure of a material to a greater pressure than it experiences in the use conditions. An example of extreme artificiality would be the use of sand paper to increase a material's surface roughness to simulate particle-impact abrasion that occurs in the use condition. A high degree of artificiality affects the strength of conclusion that can be drawn, because it may be difficult to relate the results to the use condition. Artificial aging is preferred that accelerates natural aging but does not alter it.

3.1.3 *natural aging*—aging in which the material is exposed to conditions replicating those that are present in actual service in a component for oxygen service.

4. Summary of Practice

4.1 This practice allows a systematic evaluation of the influence of age and use on a material's oxygen compatibility. To apply its principle, the user first characterizes the material of interest, then subjects the material to an aging stressor, and recharacterizes the material. The effect of the aging is then reported as positive or negative depending upon whether the material's compatibility is improved or degraded, and the measure of the influence is reported as the degree to which the measured properties changed. In incident studies, in which initial characterization data are not available, then historical or average-property data may be used to draw somewhat coarser conclusions.

4.2 This practice describes a rationale for selecting aging procedures and relating them to key fire properties. Most of the

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[2] *Annual Book of ASTM Standards,* Vol 05.03.

[3] *Annual Book of ASTM Standards,* Vol 14.03.

[4] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111.

possible studies have not been verified for significance. However, one general procedure that involves aging at pressure and temperature has been used and found to yield meaningful results, and it is described in specific detail as an example. As data become available to validate other specific meaningful aging procedures, they too will be included as examples.

5. Significance and Use

5.1 A material's fire resistance is a crucial material selection property. However, after a period of service, the material may metamorphosize into something quite different than that which it was when new. For example, all materials for oxygen service should have good mechanical properties, because mechanical failure often leads to ignition and fire damage. Therefore, any mechanism that may compromise a material's mechanical properties can invalidate it for use.

5.2 This practice allows the user to gain insight into the effects of aging on the materials in a system.

6. Apparatus

6.1 The apparatus used to age materials can vary greatly. This practice will focus on small-scale aging methods involving only a few specimens at most. The scale of the aging procedure can be increased in numerous ways, provided care is taken to ensure safety. When possible, the apparatus used to perform the fire test may also serve as a vehicle for the aging step, and that is the premise that will be discussed here.

6.2 An example of an aging procedure might be to insert a specimen into the autogenous ignition test vessel of Test Method G 72 and to both pressurize it and warm it to preselected soak levels for an aging cycle. In this case, the apparatus is the same as in Test Method G 72.

6.3 Aging related to gaseous impact might involve placing the specimen in the Test Method G 74 apparatus and soaking it at pressure (or elevated temperatures the apparatus, or both, is able to safely contain). In this case, the apparatus is that of Test Method G 74.

6.4 Specimen preparation for larger scale experiments or unique combinations of stressors that qualify as research may utilize other hardware that allows safe aging. Safety *must* be carefully evaluated for any aging arrangement.

7. Rationale for Aging Tests

7.1 This practice addresses methods to age materials so that aging effects on fire properties may be assessed using standard fire-test methods.

7.2 The body of information for aging influences is small, and so, this practice is intended to encourage such testing as a part of its goal of proposing methods to age and analyze the materials. In principle, for an aging test to be meaningful and useful, it must demonstrate an ability to discern at least some significant changes in at least some materials as a result of aging.

7.3 Aging of specimens can potentially result in numerous changes to the material. Aging may modify the surface of a specimen; exposure to ozone, light, or mechanical wear can increase the surface area through the development of cracks (crazing) or erosion. Chemical exposure may result in etching of a surface. Aging may alter the chemistry of a material; oxides may form or molecular chains may be broken. Aging may introduce free oxygen into the structure of a material; diffusion may lead to oxygen molecules permeating the material structure and entering pores that may be present in the material.

7.4 Some changes resulting from aging may alter a material's fire properties, including its ignition and propagation tendencies. In some cases, aging may render a material less susceptible to fire, while others may make it more susceptible to fire. Hence, aging tests may be used to evaluate whether a material may become unacceptable in time or whether it can be conditioned (artificially aged) as a means to improve its long-term properties. For example, temperature aging may drive off volatile materials that can compromise ignition temperature, without necessarily destroying mechanical properties and render batch testing less necessary.

7.5 In general, aging is expected to have a greater influence on a material's ignition properties than its propagation properties. To date, the only background on aging influences is that of the Bundesanstalt Für Materialforschung und -Prüfung (BAM) which has for years assessed the effect on a material's autogenous ignition temperature of aging it at elevated pressure and temperature. BAM has used the results of the testing to establish maximum constraints on the use of materials at elevated pressure and temperature.[5]

8. Reagents and Materials

8.1 *Oxygen*—Typically oxygen conforming to Federal Specification BB-0-925, Type I or oxygen of 99.5 % minimum purity is used. Oxygen of other purities or in mixture with other materials may be necessary depending upon the intent of the study.

8.2 A wide range of reagents and materials may find application in the use of this practice. For example, solvent exposure of an elastomer may represent a stressor that requires study. It is not practical to itemize such materials in this practice, as they are associated with normal or upset use conditions. Therefore, the identification of such solvents must rest with the user of this practice.

9. Safety Precautions

9.1 *Oxygen*

NOTE 1—**Warning:** Oxygen vigorously accelerates combustion.

Keep oil and grease away. Do not use oil or grease on regulators, gages or control equipment.

Use only with equipment conditioned for oxygen service by carefully cleaning to remove oil, grease and other combustibles.

Keep combustibles away from oxygen and eliminate ignition sources.

Keep surfaces clean to prevent ignition or explosion, or both, on contact with oxygen.

Always use a pressure regulator. Release regulator tension before opening cylinder valve.

All equipment and containers used must be suitable and recommended for oxygen service.

Never attempt to transfer oxygen from cylinder in which it is received

[5] Wegener, W., Binder, C., Hengstenberg, P., Herrmann, K. P., and Weinert, P., "Tests to Evaluate the Suitability of Materials for Oxygen Service," *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986,* D. W. Schroll, Ed. ASTM, 1988, pp. 268–278.

to any other cylinder.

Do not drop cylinder. Make sure cylinder is secured at all times.
Keep cylinder valve closed when not in use.
Stand away from outlet when opening cylinder valve.
For technical use only. Do not use for inhalation purposes.
Keep cylinder out of the sun and away from heat.
Keep cylinder away from corrosive environment.
Do not use cylinder without label.
Do not use dented or damaged cylinders.

9.1.1 See Compressed Gas Association Pamphlets G-4 and G-4.1[6] for details on the safe use of oxygen.

9.2 Refer to the safety precautions sections of referenced standards for further safety information applicable to the use of each standard and therefore applicable to this practice when used in conjunction with it.

10. Procedure

10.1 *Choosing Aging Criteria and Test Methods*:

10.1.1 The user must first identify the factors most likely to contribute to aging of the material, as well as the test method that is most likely to measure the change.

10.1.1.1 *Time*—Time may be the most elemental aging factor. Time alone may alter a material. Aging through time alone cannot be accelerated. As a result, it may be desirable in some instances to test materials that have been in service or in storage and to compare the results with historical data or generic averages for the particular material. Time may affect any of the properties, and hence characterization with any of the test procedures may be worthwhile.

10.1.1.2 *Elevated Temperature*—Elevated temperature may cause aging. Elevated temperature may be of most interest when a material is going to be used at elevated temperature. In this case it is most important to study whether aging at elevated temperature leads to a degradation of the autoignition temperature using Test Method G 72. At this writing, this is the only tactic Committee G-4 knows to be in use. In addition, temperature may lead to crazing and an increase in specific surface area, that can produce easily ignitable edges, hence gaseous impact may also give insight. Evaporation of volatiles or incorporation of oxygen into the material may yield a reduction in the heat of combustion, see Test Method D 4809, or may yield a change in oxygen index, see Test Method G 125. Any other property may be affected and other test methods may also be of interest.

10.1.1.3 *Pressure*—Pressure may cause absorption of increased amounts of oxygen in the structure or in the pores. To explore absorbed oxygen that may be mixed with volatile material or particulates in the pores, the gaseous impact test, Test Method G 74 may be most desirable. However, Test Methods G 86 and G 72 may also yield insight. However, the user might be surprised to see a change in the heat of combustion, Test Method D 4809, or oxygen index, Test Method G 125.

10.1.1.4 *Friction/Erosion*—Friction erosion are mechanisms that tend to increase the specific surface area of smooth surfaces and to decrease the specific surface area of rough surfaces. Increased surface area suggests autoignition tests, see Test Method G 72, gaseous impact tests, see Test Method G 74 and perhaps mechanical impact tests, see Test Method G 86, might all detect changes. Unless the surface was of different composition than the interior of the specimens, the user would not expect to see great changes in the heat of combustion, see Test Method D 4809, or oxygen index, see Test Method G 125.

10.1.1.5 *Chemical Exposure*—Chemical exposure can produce several changes in a material. Solvents can extract materials or become dissolved in the material themselves. They can attack the material surface and alter its specific surface area. And they can change a material's mechanical properties, turning it hard, gummy or otherwise. This wide assortment of prospects suggests any of the test methods may reveal aging changes.

10.2 *Characterizing the Original Material*:

10.2.1 The material should be in the exact condition for use prior to aging. Any cleaning should be consistent with cleaning required for the application of interest.

10.2.2 Test the material as specified in the test method(s) chosen: Test Methods G 72, G 74, G 86, G 125, D 4809, or other. If time is suspected to be a key aging parameter, retain some of the material in its original condition for later testing in concert with the aged material.

10.2.3 If desired to increase the data base obtained, the material may be further characterized prior to aging by weighing it, checking its physical properties, (hardness, flexibility, tensile strength, etc.) either qualitatively or quantitatively. These observations allow additional insight into whether aging occurred and whether it is similar from batch to batch.

10.3 *Aging the Specimens*:

10.3.1 Aging is most meaningful if done at conditions replicating those of service. In this case, use the same cleaning methods as intended for service. Lubricants that would be used with the material should be applied in similar amounts. If the material is used in intimate contact with other materials, then it is preferable to age the material in contact with these same materials.

10.3.2 Subject the material to the selected stressor(s). For example, to effect time/pressure/temperature aging, place the material in the vessel of Test Method G 72, pressurize it as specified in Test Method G 72, raise the temperature to the level of interest, and allow it to soak for the chosen time. By using elements of Test Method G 72 for this procedure, the safety measures of Test Method G 72 and the historical experience adds confidence in the margin of safety present, provided the amount of material involved is not in excess of the amount the vessel of Test Method G 72 is capable of containing in an inadvertent ignition.

10.3.3 Cool the vessel or proceed with testing as desired. If the specimens are cooled, they may be removed and any physical property testing may be repeated as in 10.2.3.

10.4 *Recharacterizing the Material*:

10.4.1 Following return to normal (or other chosen) conditions perform the same characterization tests of 10.2. Test the aged material using the same methods used in 10.2.2. If the selected method is Test Method G 72, begin the temperature

[6] Available from Compressed Gas Association, 1235 Jefferson Davis Highway, Arlington, VA.

ramp immediately after the aging soak at temperature is complete.

10.4.2 To do related mechanical property tests, it may be preferable to do them on different specimens than used for flammability tests, since the mechanical testing may abrade disrupt, crack or otherwise change the specimens in ways that would not occur in actual service.

11. Report

11.1 In reporting the aging process, include the following data:

11.1.1 Type of material, manufacturer, composition, and batch/lot number, if known,

11.1.2 Material preparation and cure information, if known,

11.1.3 Sample dimensions and condition,

11.1.4 Pre-aging mechanical properties,

11.1.5 A cross reference to any original-condition flammability test reports that may be available,

11.1.6 All applicable aging parameters: time, temperature, pressure, abrasion, chemical exposure, friction,

11.1.7 Post-aging mechanical properties (if determined),

11.1.8 A cross reference to any final-condition flammability test reports that may be available, and

11.1.9 The original flammability parameters, the final flammability parameters and the change.

11.2 In reporting the change in flammability properties, use the following formats to cite the aging influence:

11.2.1 For use of Test Method G 72, the change in the autoignition temperature (AIT) should be reported, and a decrease in AIT shall be called a degradation, an increase is called an enhancement.

11.2.2 For use of Test Method G 74, the change in reactive pressure should be reported, and a decrease in reactive pressure should be called a degradation, an increase an enhancement.

11.2.3 For use of Test Method G 86, the change in reactive threshold energy should be reported and a decrease in threshold should be called a degradation, and an increase should be called an enhancement.

11.2.4 For use of Test Method G 125, the change in oxygen index should be reported and a decrease should be called a degradation, and an increase should be called an enhancement.

11.2.5 For use of Test Method D 4809, the change in heat of combustion should be reported and an increase should be called a degradation and a decrease should be called an enhancement.

12. Precision and Bias

12.1 *Precision*—It is not practical to specify the precision of this practice since the result will depend on the conditions selected and the test method(s) chosen for evaluation.

12.2 *Bias*—The procedure in this practice has no bias because the test conditions are defined only in terms of this practice.

13. Keywords

13.1 aging; flammability; oxygen compatibility

ANNEX

(Mandatory Information)

A1. EXAMPLE PROCEDURE FOR TIME/PRESSURE AGING

A1.1 Prepare specimens in as-used cleanliness.

A1.2 Weigh, and examine specimens for appearance flexibility.

A1.3 Test the specimens using Test Method G 72.

A1.4 Place in vessel of Test Method G 72, pressurize, warm, and soak for 100 h, using the procedures and safety precautions of Test Method G 72.

A1.4.1 The initial soak temperature should be selected as 100°C below the autoignition temperature. If testing demonstrates material degradation, then the test should be repeated at progressively lower temperatures in increments of 25°C until degradation is no longer observed, and the material should be reported as having a degradation threshold equal to the highest temperature tested at which degradation did not occur.

A1.5 At the end of the aging cycle, the vessel should be vented, cooled, and the specimens should again be examined for qualitative changes in appearance, flexibility, etc.

A1.6 Re-test the specimens in the aged condition.

A1.7 Report the difference, in weight, physical changes, and alteration of AIT.

A1.8 This example procedure is based upon the method used at BAM.[5]

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL LITERATURE

X1.1 Waller, J. M., Hornung, S. D., and Beeson, H. D., "Fuel Cell Elastomeric Materials Oxygen Compatibility Testing: Effect of 450 and 6200 kPa Oxygen", *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres, ASTM STP 1319*, 1997.