Designation: ~~D6546 – 15~~ D6546 – 23

Standard Test Methods for and Suggested Limits for Determining Compatibility of Elastomer Seals for Industrial Hydraulic Fluid Applications[1]

This standard is issued under the fixed designation D6546; 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 These test methods cover the procedure for measuring physical properties of elastomer seals in the form of O-rings after exposure to industrial hydraulic fluids and thermal aging. The measured properties are then compared to the physical properties of elastomer seals that have not been exposed to the industrial hydraulic fluids and thermal aging. The changes in these properties form a basis for assessing compatibility when these changes are compared against the suggested limits in Table 1.

1.2 While these test methods involve the use of O-rings, they can also be used to evaluate the compatibility of the elastomeric compounds of specialty seals with industrial hydraulic fluids and their resistance to thermal aging. The compounds can be molded into O-rings for evaluation purposes.

1.3 These test methods provide procedures for exposing O-ring test specimens to industrial hydraulic fluids under definite conditions of temperature and time. The resulting deterioration of the O-ring material is determined by comparing the changes in work function, hardness, physical properties, compression set, and seal volume after immersion in the test fluid to the pre-immersion values.

1.4 The values stated in SI units are to be regarded as the standard.

1.4.1 *Exception*—The values given in parentheses are for information only.

1.5 *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~~safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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]

D395 Test Methods for Rubber Property—Compression Set

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.N0 on Hydraulic Fluids.

Current edition approved ~~Dec. 1, 2015~~July 1, 2023. Published ~~February 2016~~July 2023. Originally approved in 2000. Last previous edition approved in ~~2010~~2015 as ~~D6546 – 00 (2010).~~D6546 – 15. DOI: ~~10.1520/D6546-15.~~10.1520/D6546-23.

[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.

***A Summary of Changes section appears at the end of this standard**

TABLE 1 Property Change Limits

Time, h	Maximum Volume Swell, %	Maximum Volume Shrinkage, %	Hardness Change, Shore A Points	Maximum Tensile Strength Change, %	Maximum Elongation Change, %	Maximum Work Function Change, %	Maximum Compression Set, %
24	15	−3	±7	−20	−20	±12	. . .
70	15	−3	±7	−20	−20	±12	20
100	15	−3	±8	−20	−20	±12	20
250	15	−4	±8	−20	−20	±12	25
500	20	−4	±10	−25	−25	±17	30
1000	20	−5	±10	−30	−30	±20	35

D471 Test Method for Rubber Property—Effect of Liquids
D1414 Test Methods for Rubber O-Rings
D2000 Classification System for Rubber Products in Automotive Applications
D2240 Test Method for Rubber Property—Durometer Hardness
D3677 Test Methods for Rubber—Identification by Infrared Spectrophotometry
D3767 Practice for Rubber—Measurement of Dimensions
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D5028 Test Method for Curing Properties of Pultrusion Resins by Thermal Analysis
E1131 Test Method for Compositional Analysis by Thermogravimetry

2.2 *SAE Standard:*[3]
AS568A O-ring Sizes

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 ~~*Definitions:*~~*Definitions of Terms Specific to This Standard:*

3.2.1 ~~*batch—*~~*batch, n—*all the O-rings molded from the same lot of material and presented for inspection at one time.

3.2.2 ~~*compound—*~~*compound, n—*a fully formulated elastomer material containing all fillers and cross-linking agents.

3.2.3 *fluid saturation* ~~*effect—*~~*effect, n—*the absorption of fluid by the elastomer until an equilibrium swell value is reached at a particular temperature.

3.2.4 ~~*O-ring—*~~*O-ring, n—*a rubber seal of homogeneous composition molded in one piece to the configuration of a torus with circular cross section.

3.2.4.1 *Discussion—*
O-rings are used as both dynamic and static seals. The size of the O-ring is normally designated by a dash number corresponding to the size tables listed in AS568A. The dimensions for the O-rings used in these test methods are listed in Annex A2.

3.2.5 *ultimate* ~~*elongation—*~~*elongation, n—*the amount of stretch that the O-ring is exposed to before breaking.

3.2.6 *work* ~~*function—*~~*function, n—*work done on a test specimen to cause 20 % deformation.

4. Significance and Use

4.1 When more than one elastomer seal material is tested, the test methods yield comparative data on which to base judgements as to expected service quality. Suggested in-service property change limits are provided. Property changes beyond these limits will indicate limited service life of the elastomer seal.

4.2 These test methods attempt to simulate service conditions through controlled aging and evaluation of property changes but may not give any direct correlations with actual part performance since actual service conditions vary widely. These test methods

[3] Available from Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

yield comparative data and indications of property changes of the elastomeric seal material under ideal service conditions. These test methods can be used for quality control purposes, for engineering assessments, for service evaluation, and for manufacturing control. The information from these test methods can be used to anticipate expected service quality.

5. General Test Methods

5.1 Except as otherwise specified, the test methods for rubber O-rings referred to in 5.1.1 – 5.1.6, which are applicable in general to vulcanized rubber, shall be complied with as required and are hereby made a part of these test methods.

5.1.1 *Tension Test*—Test Methods D412 and D1414.

5.1.2 *Compression Set*—Test Methods D395 and D1414.

5.1.3 *Fluid Aging*—Test Method D471 and Test Methods D1414.

5.1.4 *Hardness*—Test Method D2240.

5.1.5 *Compositional Analysis*—Test Methods D3677 and Test Method E1131.

5.1.6 *Degree of Cure*—Test Method D5028.

5.2 In case of conflict between the provisions of the ASTM test methods referenced in 5.1.1 – 5.1.6 and the detailed provisions of the test methods in Test Methods D6546, the latter shall take precedence.

6. Test Conditions

6.1 *Temperature*—The test temperature shall be the maximum sustained temperature anticipated in service.

6.2 *Immersion Periods*—The following immersion periods are recommended: 24 h, 72 h, 100 h, 250 h, 500 h, and 1000 h. The final immersion period will depend upon the results of the previous immersion period. If the changes in the physical properties have deteriorated beyond the suggested limits, then further testing is not required. The tolerance for any immersion period shall be ±1 % of the immersion period.

7. Test Fluids

7.1 For reliable compatibility assessments, it is desirable to use the fluid with which the elastomer will come in contact in actual service. For comparative tests, samples of fluid from the same drum or shipment shall be used.

8. Test Specimen

8.1 The test specimens shall be O-rings molded from the same compound batch from which the actual seals will be molded. The test samples should approximate the cross section of the actual seal to be used so that the fluid saturation effect is properly considered. The test samples should be either -021, -120, -214, or -320 O-rings, in accordance with AS568A. These have an approximate inside diameter of 25.4 mm (1 in.) and represent the most popular cross sections of seals used in industrial systems. The actual dimensions of each O-ring size are listed in Annex A2.

8.2 Test specimens shall be wiped clean of external contaminants prior to testing by using a clean dry wipe.

9. Suggested Compatibility Test Limits

9.1 For a critical seal application, property change limits, as described in Table 1, should be observed.

9.2 All values are in reference to soak time in the operational fluid at the operating temperature of the application. Values reflect changes from the determined pre-immersion original physical property values of the test specimens.

9.3 If the changes are within these limits, the elastomer should be considered compatible. Once a seal material is found to be compatible, all seals for that system should be ordered by specific compound or specification and not by Classification D2000 call out number or generic polymer designation.

10. Procedure for Change in Volume

10.1 *Apparatus:*

10.1.1 *Test Container,* a Mason jar (quart size) fitted with a lid to prevent liquid and vapor from escaping. The lid shall not contaminate the test liquid. Cover the lid with aluminum foil.

10.1.2 *Heating Device,* a forced air oven, aluminum block heater, or oil bath heater. Maintain the temperature within ±1 °C (1.8 °F).

10.1.3 *Test Specimen*—The test specimen shall consist of an entire O-ring. The same specimen may be used for all tests with hardness and volume determinations made prior to stress-strain tests. Place the test specimen in the test liquid so that it is not distorted or in contact with the sides of the test container or with the other test specimens. Test a minimum of three test specimens at one time. It is also important that only O-rings of one size and one material compound be placed in the test container.

10.1.4 *Analytical Balance,* an analytical balance capable of allowing a test specimen to be weighed whether in air or while submerged in water.

10.2 *Volume Change*—Test three specimens.

10.2.1 Weigh each test specimen in air, M_1, to the nearest 1 mg, and then weigh each specimen immersed in water, M_2, at room temperature. It is important that all air bubbles clinging to the test specimen be removed before reading the weight in water. Blot the specimen dry.

10.2.2 Suspend the specimens in the glass jar by the use of corrosion-resistant wire. Separate the specimens by bending small loops in the wire or by locating them in different locations so that they do not contact each other.

10.2.3 Suspend the specimen vertically so that 25.4 mm (1 in.) of test fluid is between the lower extremity of the specimen and the bottom of the apparatus. Add enough test fluid to cover the specimen to a depth of 25.4 mm (1 in.) over the upper extremity of the specimen.

10.2.4 Place the test apparatus in the heating device adjusted to maintain the sample at the test temperature for the required length of time. At the end of the required immersion period, remove the specimen from the apparatus. Cool the specimen to room temperature by immersing it in a cool, fresh amount of the test fluid for 45 min.

10.2.5 At the end of the cooling period, remove the specimen from the fluid, wipe with a cloth dipped in acetone, and blot dry. Weigh each test specimen in air, M_3, and then weigh each specimen immersed in water, M_4.

10.2.6 Some oils can be very viscous and may be difficult to remove with an acetone wipe. Since these oils do not readily volatize, specimens exposed to these oils can be cooled by suspending them for 45 min in air at room temperature shielded from draft. This will allow the majority of the oil to drip off the surface of the specimen. Then proceed with the acetone wipe and weighing process described in 10.2.5. Report when this alternate method of specimen cooling is used.

10.2.7 The change in volume is calculated as follows:

$$\Delta V, \% = \frac{(M_3 - M_4) - (M_1 - M_2)}{(M_1 - M_2)} \times 100 \quad (1)$$

where:

M_1 = initial mass of specimen in air, g,
M_2 = initial mass of specimen in water, g,
M_3 = mass of specimen in air after immersion, g, and
M_4 = mass of specimen in water after immersion, g.

10.3 *Volume Shrinkage-Simulated Dry Out (Optional Test Method)*—Test three specimens.

10.3.1 In some situations when long downtimes are expected, the O-ring should not shrink beyond 5 % of its previous volume change value since this can affect its ability to be an effective seal when the system is restarted. In those cases in which a positive volume change was obtained in 10.2 and long system down times are anticipated, it is recommended that volume shrinkage be determined. To perform this optional test method, additional O-rings will have to be tested in accordance with 10.2 and then tested in accordance with 10.3 since the normal test for volume change is immediately followed by the destructive tensile test.

10.3.2 The test specimen shall consist on an entire O-ring. The specimen must first be submitted for the volume swell test. This specimen is only to be used for this test sequence and not for any other testing.

10.3.3 Place the test specimen from the volume swell test in a forced-air oven that allows air circulation around the test specimen, and maintain the oven at a test temperature of 23 °C ± 1 °C (73.4 °F ± 1.8 °F) for 22 h ± 0.25 h. At the end of the required period, remove the specimen from the oven and allow it to air cool.

10.3.4 Weigh each test specimen in air, M_5, and then weigh each specimen immersed in water, M_6.

10.3.5 The change in volume or shrinkage is calculated as follows:

$$\Delta V, \% = \frac{(M_5 - M_6) - (M_3 - M_4)}{(M_3 - M_4)} \times 100 \quad (2)$$

where:

M_3 = initial mass of volume swell specimen in air after immersion, g,
M_4 = initial mass of volume swell specimen in water after immersion, g,
M_5 = mass of volume swell specimen in air after dry out, g, and
M_6 = mass of volume swell specimen in water after dry out, g.

11. Changes in Tensile Strength, Work Function, Elongation, and Hardness

11.1 *Original Properties*—The original tensile strength, work function, ultimate elongation, and hardness shall be determined using a duplicate set of specimens of O-rings of the same cross section as those that are to be immersed in the test fluid. The O-rings shall be from the same batch as those that are to be immersed in the test fluid.

11.2 *Properties After Exposure to the Test Fluid,* for determining the tensile strength, work function, ultimate elongation, and hardness of specimens after immersion in the test fluid at the test temperature. At the end of the required immersion time, remove the specimens, and if necessary, cool them to room temperature in a fresh sample of the same fluid for 45 min. At the end of the cooling period, remove the specimen from the fluid, wipe it with a cloth dipped in acetone, and blot dry. Immediately determine the hardness, tensile strength, work function, and ultimate elongation in accordance with the following test methods, using the original cross-sectional area of the untreated specimens.

11.2.1 Three specimens shall be tested. The test specimen shall consist of the entire O-ring. These specimens must first be submitted to the volume swell test and cannot be used for any other testing since physical property tests are destructive.

11.3 *Hardness Change*—Measure the hardness in accordance with Test Methods D1414, Section 16, using a microhardness tester. Select the mean value from the multiple readings taken on each O-ring, and then select the mean value for all the O-rings. (The mean value for hardness measurements is the numerical mean value; thus if five readings are obtained, for example, 70A, 69A, 69A, 72A, and 71A, the numerical mean would be 70.2 or 70A since Shore hardness is always reported in whole numbers.) The mean value for all O-rings shall be recorded. Measurements are to be taken before and after exposure to fluid.

11.3.1 The hardness change is calculated as follows:

$$\Delta H = H_2 - H_1 \quad (3)$$

where:

ΔH = hardness change,

H_1 = hardness before fluid exposure, and
H_2 = hardness after fluid exposure.

The units are given as Shore A points and a plus or minus sign should be included. A negative sign would indicate that the O-ring is softening after exposure and its hardness value would be less than the hardness value before exposure. A positive sign would indicate that the O-ring is hardening after exposure and its hardness value would be greater than the hardness value before exposure.

11.4 *Tensile Strength Change:*

11.4.1 *Testing Machine*—The testing machine shall conform to the requirements specified in Section 3 of Test Methods D412 with the exception of grips. Grips for testing O-rings shall consist of ball-bearing spools at least 8.89 mm (0.35 in.) in diameter and be capable of being brought within 19.05 mm (0.75 in.) center-to-center distance at closest approach. Stresses within the specimen shall be minimized by rotating one spool or by lubricating the contact surface of the spools with castor oil.

11.4.2 *Test Specimen*—The test specimen shall consist of an entire O-ring.

11.4.3 *Procedure*—Bring the grips close enough together so that the specimen can be installed without stretching. Separate the grips to remove any slack in the specimen. Exercise care that no load is placed on the specimen. Pull the specimen at a rate of 50.8 cm⁄min (20 in.⁄min). Record the breaking force value, F, at the time of rupture.

11.4.4 *Calculations:*

11.4.4.1 Tensile strength is calculated as follows:

$$T = F/A \tag{4}$$

where:

T = tensile strength, MPa (psi),
F = breaking force, N (lb), and
A = twice the cross-sectional area calculated from axial thickness, W, as follows:

$$A = \pi W^2/2 = 1.57\ W^2\ \text{mm}^2\ (\text{in.}^2) \tag{5}$$

11.4.4.2 Tensile strength change is calculated as follows:

$$\Delta T = \frac{T_2 - T_1}{T_1} \times 100 \tag{6}$$

where:

ΔT = tensile strength change (%),
T_2 = tensile strength after immersion, and
T_1 = tensile strength prior to immersion.

11.5 *Elongation Change:*

11.5.1 *Testing Machine*—Same as for tensile strength change.

11.5.2 *Test Specimen*—Same as for tensile strength change.

11.5.3 *Procedure*—Same as for tensile strength change, except record the center-to-center distance (D) between the spools at rupture to the nearest 2.54 mm (0.1 in.).

11.5.4 *Calculations:*

11.5.4.1 Ultimate elongation is calculated as follows:

$$E, \% = \frac{(2D + G - C)}{C} \times 100 \tag{7}$$

where:

D = distance between centers of the spool grips at the time of rupture of the specimen,
G = circumference of one spool (spool diameter × 3.14), and
C = inside circumference of the specimen (inside diameter × 3.14)

11.5.4.2 Change in elongation is calculated as follows:

$$\Delta E, \% = \frac{E_2 - E_1}{E_1} \times 100 \tag{8}$$

where:

E_2 = elongation after immersion,
E_1 = elongation prior to immersion.

11.6 *Work Function (WF) Modulus Change:*

11.6.1 *Testing Machine*—Same as for tensile strength change.

11.6.2 *Procedure*—Same as for tensile strength change.

11.6.3 *Calculations*—Calculate the work function (*WF*) as the energy per unit volume at 20 % elongation. This value is determined as the area under the stress-strain curve from 0 % to 20 % strain, and the tensile tester should be programmed to determine this value.

11.6.3.1 Change in work function is calculated as follows:

$$\Delta WF = \frac{WF_2 - WF_1}{WF_1} \times 100 \tag{9}$$

where:

ΔWF = change in work function,%,
WF_2 = work function after immersion, MPa (psi), and
WF_1 = work function prior to immersion, MPa (psi).

12. Compression Set

12.1 *Micrometer,* for measuring the specimen thickness, in accordance with Practice D3767, Method A1.

12.2 *Spacer Bars,* to maintain the constant deflection. Spacer bars for O-ring samples shall have a thickness of 9.5 mm ± 0.02 mm (0.375 in. ± 0.001 in.).

12.3 *Compression Device,* consisting of two or more flat steel plates between the parallel faces of which the O-ring specimens may be compressed, as shown in Fig. 1. Steel spacers for the required 25 % of compression shall be placed on each side of the O-ring specimens to control their thickness while compressed. The steel surfaces contacting the rubber specimens shall be ground to a maximum roughness of 250 µm (10 µin.) and then chromium plated and polished.

12.4 *Oven,* a forced air oven capable of maintaining the test temperature within ±1 °C (1.8 °F).

12.5 *Plates*—The plates between which the O-ring test specimen is compressed shall be made of steel of sufficient thickness (at least 9.5 mm ± 0.02 mm (0.375 in. ± 0.001 in.) or thicker) to withstand the compressive stresses without bending. The surfaces against which the O-ring specimen is held shall have a highly polished chromium-plated finish and shall be cleaned thoroughly and wiped dry before each test.