Designation: D6546 – 15

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 practices and determine the applicability of regulatory limitations prior to use.*

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

3.1.2 *compound*—a fully formulated elastomer material containing all fillers and cross-linking agents.

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

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

3.1.4.1 *Discussion*—O-rings are used as both dynamic and static seals. The size of the O-ring is normally designated by a

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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 Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

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

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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

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.

3.1.5 *ultimate elongation*—the amount of stretch that the O-ring is exposed to before breaking.

3.1.6 *work function*—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 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:

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

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:

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

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:

$$\Delta H = H_2 - H_1 \tag{3}$$

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:

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

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.

12.6 *Original Thickness Measurement*—Measure the original thickness of the specimen to the nearest 0.02 mm (0.001 in.). Place the specimen on the anvil of the micrometer so that the presser foot will indicate the thickness at the central portion of the top and bottom faces.

12.7 *Application of Compressive Force*—Place the O-ring test specimen between the plates of the compression device with the spacers on each side, allowing sufficient clearance for the bulging of the O-ring when compressed. Where a lubricant is applied, it shall consist of a thin coating of the O-ring, with a lubricant having substantially no action on the rubber. For most purposes, a silicone or fluorosilicone fluid is suitable. Tighten the bolts so that the plates are drawn together uniformly until they are in contact with the spacers. The amount of compression employed shall be approximately 25 %. A suitable mechanical or hydraulic device may be used to facilitate assembling and disassembling the test fixture.

12.8 *Test Temperature and Time*—The test temperature shall be the maximum anticipated service temperature. The test times shall be 72 h, 100 h, 250 h, 500 h, and 1000 h. In comparative tests, use identical temperature and test periods.

12.9 *Test Specimen*—The test specimens should be O-rings molded from the same compound 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. A minimum of two test specimens should be tested at one time. It is important that only O-rings of one size and one material be placed in the compression fixture.

12.10 *Test Fluid*—For accurate compatibility assessment, it is desirable to use the fluid with which the elastomer will come into contact when in service. For comparative tests, samples of fluid from the same drum or shipment shall be used.

12.11 *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.

12.12 *Test Procedure*—The test specimen shall be at room temperature when inserted in the compression device. Place the assembled compression device, immersed in the test fluid, in the oven within 2 h after completion of the assembly, and allow it to remain there for the required test period at the test temperature. At the end of the test period, release the plates immediately and allow the specimens to cool to room temperature for 45 min in a fresh portion of the test liquid.

12.13 *Final Thickness Measurement* —After the rest period, measure the final thickness at the center of the test specimen in accordance with Practice D3767, Method A1.

12.14 Calculate the compression set expressed as a percentage of the original deflection as follows:

$$C = [(t_o - t_f)/(t_o - t_n)] \times 100 \quad (10)$$

where:

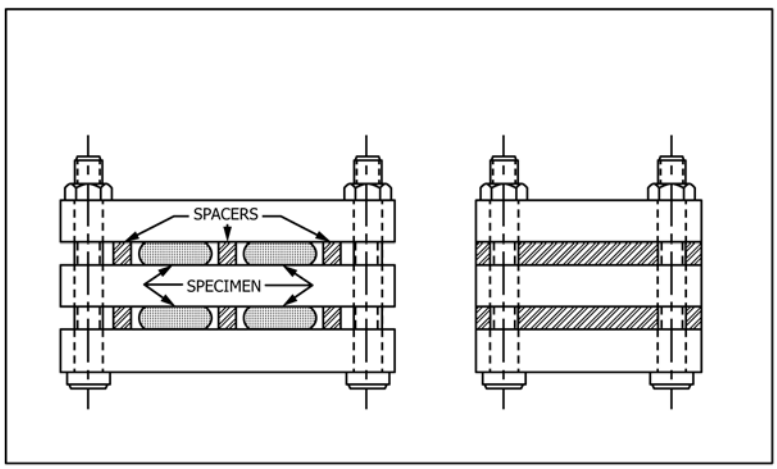

FIG. 1 Compression Device for O-ring Specimens

C = compression set expressed as percentage of the original deflection,
t_o = original thickness of specimen,
t_f = final thickness of specimen, and
t_n = thickness of spacer bar used.

Record the value for C.

13. Material Traceability and Compound Confirmation

13.1 To provide full traceability of the compound being qualified and to ensure that the compound and the state of cure do not change during production, an FTIR spectrum of a randomly chosen qualification part shall be obtained in accordance with Test Methods D3677. This spectrum appropriately dated and recorded shall remain part of the permanent record for the qualified compound. Copies of this record should be available for reference.

13.2 In addition, a TGA curve will be obtained for the same sample in accordance with Test Method E1131. This curve, showing compositional analysis, will also be appropriately dated and recorded and shall remain part of the permanent record for the qualified compound.

13.3 A Differential Scanning Calorimetry (DSC) curve, confirming the degree of cure of the sample, will be obtained in accordance with the procedure listed in Annex A1. This DSC curve will be properly dated and recorded and will also become part of the permanent record for the qualified compound. This permanent record, documenting the qualified compound, shall be used to confirm that production parts are made from the same compound as qualified and that they are fully cured as were the qualified elastomer parts.

13.4 To compare the FTIR spectra of production parts with the qualified material, a view foil of each spectrum can be made and overlaid one atop the other. The major features of both spectra should be identical. For TGA comparisons, the weight loss over specific temperature ranges for both the qualified elastomer sample and the production sample should be the same and their curves identical in appearance. Identity can again be evaluated using the view foil technique.

14. Report

14.1 State that the test was conducted in accordance with Test Methods D6546, and report the following:

14.1.1 Date,
14.1.2 Cure date and batch number of the test specimens,
14.1.3 Dates of the various periods of exposures,
14.1.4 Immersion liquid used,
14.1.5 Temperature of exposure,
14.1.6 Exposure period,
14.1.7 All observed and recorded data, including the type of properties being reported,
14.1.8 Results calculated in accordance with the preceding procedures, and
14.1.9 Conclusions of the tests based upon the compatibility limits listed in Section 9.

14.2 The FTIR, TGA, and DSC results should be maintained on file for comparison to the results for production parts.

15. Precision and Bias

15.1 The precision and bias of these test methods for measuring physical properties of new O-rings and O-rings after exposure to industrial hydraulic fluids and thermal aging are essentially as specified in Test Methods D1414, Practice D3677, and Test Method E1131.

16. Keywords

16.1 compatibility; compression set; density; elastomer seal; elongation; fluid aging; hardness; industrial hydraulic fluid; O-ring; tensile strength; volume shrinkage; volume swell; work function

ANNEXES

(Mandatory Information)

A1. TEST PROCEDURE FOR DETERMINING DEGREE OF CURE OF ELASTOMERS BY DIFFERENTIAL SCANNING CALORIMETRY (DSC)

A1.1. Scope

A1.1.1 This test method covers the determination of the cure state of elastomers by DSC. It is based on Test Method D5028.This test method is applicable to elastomers with adequate vulcanizers. The normal operating temperature range is from room temperature to 250 °C (482 °F), but not limited to such. All elastomers should be fully cured.

A1.2. Apparatus

A1.2.1 Differential Scanning Calorimeter, capable of heating a test specimen and a reference material at a controlled rate up to at least 20 °C (68 °F) per minute and of automatically recording the differential heat flow.

A1.2.2 *Specimen Holders,* composed of clean aluminum or other high thermal conductivity material. Specimen holders may be of the open, covered, or sealed type.

A1.2.3 *Nitrogen,* or other inert purge gas supply.

A1.2.4 *Flowmeter,* for purge gas.

A1.2.5 *Recording Charts,* for temperature recording apparatus with suitable graduation for measurements of energy differential or time.

A1.3. Calibration

A1.3.1 Calibrate the apparatus in accordance with the manufacturer's instructions with appropriate standard reference materials at the same heating rate to be used for samples. For the temperature range of this procedure, indium with a melting point of 156 °C (312.8 °F) may be used.

A1.4. Procedure

A1.4.1 *Cured Elastomer Part:*

A1.4.1.1 Randomly select a cured elastomer part and randomly choose a small section of the part from its center, and weigh out 76.54 mg to 153.08 mg of the section.

A1.4.1.2 Crimp a flat metal cover against the pan with the sample sandwiched between them to ensure good heat transfer. Place the sample in the DSC cell.

A1.4.1.3 Intimate thermal contact between the sample and the thermocouple is essential for reproducible results.

A1.4.1.4 Purge the cell with nitrogen at 50 cc/min to 100 cc/min (3.06 $in.^3$/min to 6.10 $in.^3$/min) gas flow rate.

A1.4.1.5 Heat the sample at a rate of 10 °C/min (50 °F/min) under nitrogen atmosphere from ambient to a temperature high enough to achieve the entire exothermic curing curve. Record the thermogram.

A1.4.1.6 Measure the heat of reaction (ΔH_R), the shaded area of Fig. A1.1 (see Figs. A1.1 and A1.2).

A1.5. Calculation

A1.5.1 Use the resulting thermogram to determine the exothermic heat of the curing reaction (ΔH_R) of the cross-linking reaction. This is used to indicate the extent of cure. A fully cured material shall exhibit a ΔH_R of 0 cal/g (0 Btu/lb). Fig. A1.2 depicts a fully cured material.

A1.5.2 If the sample exhibits a ΔH_R of 0 cal/g (0 Btu/lb), the sample is fully cured. If the sample exhibits a ΔH_R of greater than 0 cal/g (0 Btu/lb) (see Fig. A1.1), run a duplicate sample. If the duplicate sample yields a ΔH_R of 0 cal/g (0 Btu/lb), run a third sample such that two independent runs are in agreement. If the duplicate sample yields a ΔH_R greater than 0 cal/g (0 Btu/lb), the elastomer part is not fully cured and processing is not consistent.

A1.6. Report

A1.6.1 Report the following information:

A1.6.1.1 Sample identification including compound and batch inside diameter and mass,

A1.6.1.2 Heating rate and purge gas flowrate,

A1.6.1.3 Value of ΔH_R,

A1.6.1.4 Date of testing, and

A1.6.1.5 Copies of the actual DSC curves.

A1.7. Precision and Bias

A1.7.1 Since this test method is designed to determine whether a sample is fully cured or not, the heat of reaction (ΔH_R) is the value of interest. If the value of ΔH_R is positive, then the sample is not fully cured. Calorimetric precision is normally ±1 % and calorimetric accuracy is also ±1 %, but fully cured materials have no exotherm. What is important in determining if a material is fully cured is the stability of the baseline because any positive value above baseline can be interpreted as an exotherm. Baseline noise for these types of

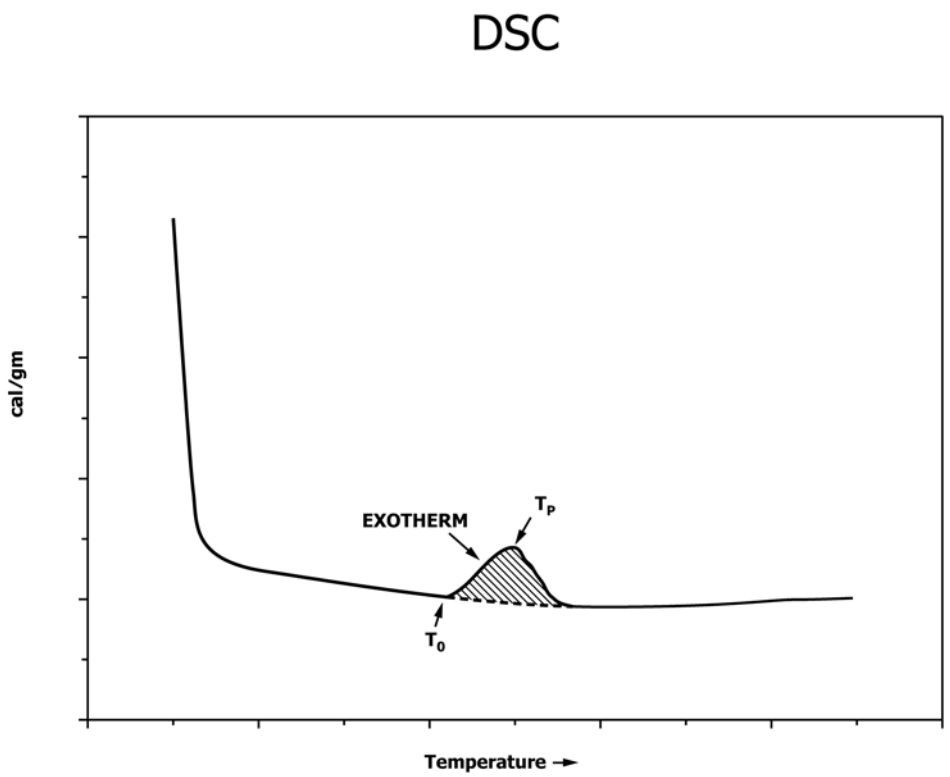

FIG. A1.1 Thermogram Representative of an Uncured Elastomer

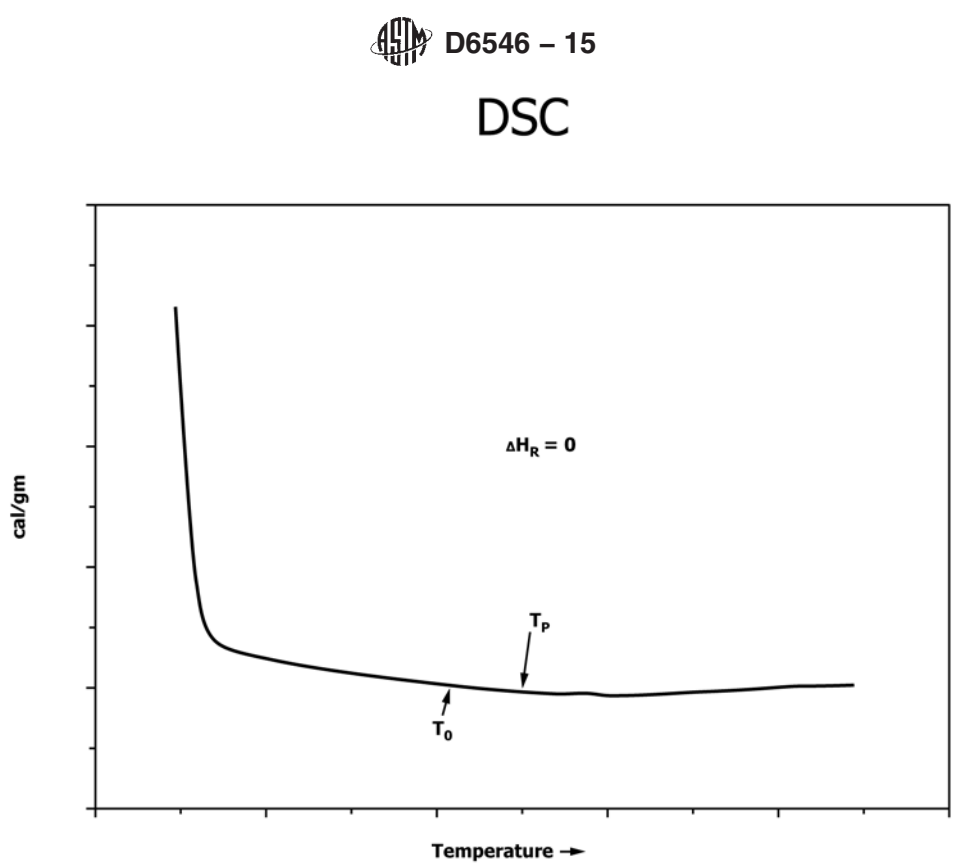

FIG. A1.2 Thermogram Representative of a Fully Cured Elastomer

instruments is 0.00431 cal (0.0000171 Btu). Baseline noise during a run can in effect create a positive indication. If the maximum baseline noise value is divided by the minimum sample mass, 76.54 mg, then a baseline maximum deviation of 0.0563 cal/g can be expected. Therefore, any test value that ranged from 0 cal/g to +0.0563 cal/g would be equivalent to fully cured material.

A2. SIZES OF O-RINGS USED IN TEST PROCEDURES

A2.1 Table A2.1 lists the actual sizes of the O-rings used in the test methods. The sizes are in accordance with AS568A.

TABLE A2.1 O-ring Sizes

AS568A Designation	Inside Diameter (in.)	Cross Section, in.	Inside Diameter (mm)	Cross Section, mm
-021	0.926 ± 0.009	0.070 ± 0.003	23.52 ± 0.23	1.78 ± 0.08
-120	0.987 ± 0.010	0.103 ± 0.003	25.07 ± 0.25	2.62 ± 0.08
-214	0.984 ± 0.010	0.139 ± 0.004	24.99 ± 0.25	3.53 ± 0.10
-320	1.100 ± 0.012	0.210 ± 0.005	27.94 ± 0.30	5.33 ± 0.13

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SUMMARY OF CHANGES

Subcommittee D02.N0 has identified the location of selected changes to this standard since the last issue (D6456 – 00 (2010)) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Revised SI unit formatting throughout.

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