



The Resistance of Elastomers to Flex Fuel (Ethanol and Methanol) Dilution in Engine Oil

1 Introduction

Note: Nothing in this standard supercedes applicable laws and regulations.

Note: In the event of conflict between the English and domestic language, the English language shall take precedence.

1.1 Purpose.

1.1.1 Establish a test protocol for cured elastomeric compounds from molded sheet (slab), cylinder (button) or O-ring, exposed to ethanol or methanol oil mixtures, to the requester specified conditions.

1.1.2 Determine the effects of fuel dilution on elastomers by comparing specified property values before and after exposure. These effects may be by absorption, extraction, sealing pressure loss and/or chemical reaction.

1.1.3 To determine the resistance to flex fuel dilution in engine oil the following properties are used (see 1.1.3.1 thru 1.1.3.5).

1.1.3.1 Volume change (see 3.5.1.5).

1.1.3.2 Compressive Stress Relaxation (see 3.5.1.10).

1.1.3.3 Elongation (see 3.5.1.3).

1.1.3.4 Hardness (see 3.5.1.4).

1.1.3.5 Additional specified properties in Relative Drawings or Specifications.

1.2 Applicability. These procedures are intended for GM Powertrain material suppliers and gasket fabricators for the purpose of measuring the static gasket or dynamic seal heat cured elastomer resistance characteristics used in Flex Fuel Vehicle (FFV) engine oil systems.

1.2.1 This document applies to gas engines. Flex fuel dilution in engine oil resistance with Room Temperature Vulcanized (RTV) sealant and anaerobic is covered in a separate document.

1.3 Remarks. Resistance as measured using these procedures is not an indicator of durability. Attempts to run traditional accelerated development or durability conditions for elastomers, created abnormal run conditions far beyond customer experience. Given that the test fuel constituents have low boiling points (65 °C to 110 °C), replenishment became a challenge with safety a necessary consideration.

1.3.1 Elastomer resistance to flex fuel dilution in engine oil is expressed, as a change in specified properties (see 1.1.3), from test specimens, after exposure to specified test conditions in this document, and stated on the drawing or standard.

1.3.2 Functional noun name conventions used on prints have no logical foundation. Any circular shaped, press-in-place gasket, even though it is not an O-ring (toroid), may be identified as such. Actual O-rings may not have that name in the drawing title box. Regardless of the naming convention, the term "O-ring" refers to a rubber seal of homogeneous composition molded in one piece to the configuration of a torus with circular cross section.

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2 References

Note: Only the latest approved standards are applicable unless otherwise specified.

2.1 External Standards/Specifications.

ANSI B1.20.1	ASTM D1415	DIN 3535	ISO 1817
ANSI B1.20.3	ASTM D1460	DIN 3536	ISO 3601
ASTM D395	ASTM D1566	DIN 53504	ISO 17025
ASTM D412	ASTM D2240	ISO 37	SAE ARP3050
ASTM D471	ASTM D3182	ISO 48	SAE AS568
ASTM D572	ASTM D4968	ISO 815-1	SAE J1748
ASTM D1414	ASTM E515	ISO 1101	

2.2 GM Standards/Specifications.

GMW3149	GMW14914	GMW17113	GMW17332
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2.3 Additional References.

McMaster Carr Part Number (P/N) 3671K56

McMaster Carr P/N 3845K17

McMaster Carr P/N 4704K111

McMaster Carr P/N 5556K39

McMaster Omega P/N 39095K61

McMaster Omega P/N CN740

Relative Drawings or Specifications

www.corning.com/lifesciences

2.4 Test Standards Equivalency. For reasons of cost and convenience, this document lists equivalent industry standards. Equivalency as it applies to this document is stated in ASTM D4968 (Appendix A2). Documents may have some minor form differences, but otherwise will give "identical" results to those actions performed as a result of following the Organization for International Standards (ISO) standard and "vice versa". Use only the co-listed standards listed in 3.5 Test Required Information and Section 4 Procedure.

3 Resources

3.1 Facilities. Conform to the relevant and referenced test procedures. Lab accreditation to ISO 17025 for the referenced industry procedures is strongly recommended.

3.1.1 Calibration. The test facilities and equipment shall be in good working order and shall have a valid calibration label.

3.1.2 Alternatives. Alternative test facilities and equipment may also be used. However, all measuring variables as specified in this standard shall be determined correctly with respect to their physical definition.

3.2 Equipment. Conforming to the relevant and referenced test procedures.

3.2.1 Open Test System Equipment (see Appendix C, Table C1). The open test system (see Appendix A, Figure A1 and/or Figure A2) consists of a hot plate stirrer unit as a platform for providing heat to an insulated pressure vessel. Supplementary heat can be provided using a band heater or mantle surrounding the pressure vessel circumference. Insulation provides protection against hot surfaces and aids in maintaining uniform test solution temperature. An auxiliary tank for each pressure vessel is necessary to handle any fluid and/or vapor discharge during the test. The entire setup may be placed inside an industrial hood (see 3.2.8 Fume Hood or Vent).

3.2.2 Pressure Vessel. Use a pressure vessel similar to the description in ASTM D572, capable of maintaining the required test pressure and elevated temperature for the duration of the test or change interval hours, whatever comes first. The internal volume shall be of sufficient capacity (Libra vessel unit internal volume is approximately 5463 cm³), made of a material that is inert to the elastomer test specimens and test medium. To prevent discharge of test fluid from potentially contacting hot surfaces an auxiliary collection tank of sufficient

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volume is required (see Appendix A). The pressure vessel seal ring shall be replaced at the end of a completed test (see 3.4).

3.2.3 Pressure Vessel Support Ring. Due to the weight limits of the hot plate top (approximately 11.3 kg maximum (25 lb; maximum)) a support ring under the pressure vessel flange is highly recommended to bear the bulk weight but permit sufficient base contact to allow proper stirring of the internal test solution.

3.2.4 Hot plate and stirrer units are available with protected electronics and over temperature shutoff features. See Appendix C, Table C1, Parts and Components Specific to the Open Test System.

3.2.5 Vessel Volume. Pressure vessel capacities vary by manufacturer so the internal volume must allow the ASTM D471 required number of test specimens (see 4.1.1) and volume of test fluid with proper immersion coverage (see 4.3.4 Full Immersion Condition). Multiple specimen shapes can be combined in the same vessel provided the fluid to specimen ratio is maintained (see 4.3.4 Full Immersion Condition). Different compounds must not be run in the same vessel due to cross contamination effects. Combining Compression Stress (CS) or Compression Stress Relaxation (CSR) fixtures with tensile or volume change specimens is discouraged as the need to remove the CS/CSR fixtures for measurement will require moving specimens and suspending the test time till measurements are completed. The number and orientation of specimen loading must not disrupt mixing (see 4.3.6.1). The Libra vessel can hold three (3) CSR fixtures in a triangular pattern on one platform level.

3.2.6 Auxiliary Monitoring Equipment. A temperature probe or thermocouple (see Appendix C, Table C3) is required to monitor internal fluid temperature (ASTM D471 (Section 8)). Probes capable of controlling the hot plate/stirrer are required. Pressure gauge and relief valves are required for each vessel. All connections, gauges and thermocouple access points must be leak checked before and during testing. Use smoke generators, soap/water solutions or other leak detection methods at all potential leak path points. Use appropriate torque, tapes, pastes, etc., to seal these joints. See Appendix A.

3.2.7 Auxiliary Collection Tank. Located external to thermal chamber or hot plate/stirrer with sufficient volume capacity to accommodate fluid expansion. A typical capacity is 1.5 times the internal vessel volume depending on the number and size of components tested. The tank is fabricated from non-reactive material to fit the requirements. Any discharge of vapor and/or fluid is transported directly into the collection tank. Since a low pressure condition is maintained (see 4.2.2.6 Test Pressure) the vapor can be bubbled into water in the tank or vented into, as local regulation permits.

3.2.8 Fume Hood or Vent. Appropriate industry approved construction and ventilation performance to remove vapors and provide containment.

3.2.9 Temperature Controls. Whichever test system is chosen, it shall provide uniform temperature control specified for the test solution for the duration of the test. See 4.2.2 Test Conditions.

3.2.10 Closed System Test Equipment (Appendix C, Table C2). Some test locations requiring a higher level of regulatory control may use explosion proof equipment. Pressure vessels shall be placed in an explosion proof air circulating oven (closed system). The closed or chamber system (see Appendix B, Figure B1 and/or Figure B2), provides circulated heated air to pressure vessels, each nested in an air driven magnetic stirring base. A special base plate measured to fit each pressure vessel is needed to house the stirring mechanism and move the magnetic stirring bar. Stirring is required to maintain a uniform test medium (see Figure A1). No supplemental insulation or heating of pressure vessels is required. The auxiliary collection tank (see 3.2.7) is a matter of tester choice. A cone shaped top permits angle mounting of temperature, pressure monitoring and relief connections where tight clearance conditions with the top of the interior chamber exists.

3.2.10.1 Thermal Test Chamber with Temperature Controller. Chamber internal volume sufficient (at greater than 164 L (5.8 ft³)) to house three (3) or more pressure vessels with base and capable of maintaining the specified solution test temperature to the tolerances given in 4.2.2.5 Test Fluid Temperature. Test chamber must have plumbing and wiring access hole(s). Temperature controller with programming feature must maintain ± 3 °C of set temperature. Unit (with temperature indicator) must have multiple thermocouple channels to monitor chamber and part temperature. Sensor input must accept designated temperature measuring device. When a program is interrupted by shutdowns, it must permit the operator to reset the test cycle at ambient (23 °C) and resume the cycle toward the last direction the temperature cycle was headed.

3.2.10.2 All gauging and electronic monitoring equipment shall be located outside of the closed test chamber.

3.2.10.3 Chamber Temperature Monitor. Computer controlled data analyzer capable of measuring in -40 °C to 150 °C range to ± 3 °C used for chamber temperature control.

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3.2.10.4 Air Delivery Lines; Supply and Return. Recommended: Supply lines, 12 mm (0.5 in) inside diameter, minimum and rated above the maximum test chamber temperature, system test pressures and pulses. Non-corrosive, stainless steel lines with quick connect end fittings are recommended. These lines connect the manifold to each stirrer base for the closed system test.

3.2.10.5 Air Manifold; Supply. Made of non-corrosive material with individual connections for each stirrer base. Capable of operating in the test environment.

3.2.10.6 Air Supply. Either shop air or bottled gas, to provide air to drive the stirring base unit. An inline oil and water filter is recommended with shop air supply removes water and oil vapors as well as condensed moisture from an airline.

3.2.10.7 Multistage Pressure (Gas) Regulator. Appropriate for air source and delivery pressures. A feed pressure of approximately 0.2 bar (3 psig) is recommended to operate the stirrer.

3.2.10.8 Flow Control Valve. Made of non-corrosive material(s) that withstand the air and test environment. The valve provides a constant air feed pressure to each stirrer base.

3.2.11 Pressure Vessel Temperature Control (VTC) Device. Capable of measuring to 150 °C range to ± 3 °C accuracy. These two (2) devices meet these requirements: A Resistive Temperature Differential (RTD) sensor, American National Standards Institute (ANSI) Types E, J, K, or T thermocouple, solid bare wire diameter (gauge) with wire insulation.

3.2.12 Vessel Pressure Monitoring and Measuring Pressure Equipment. Capable of measuring pressure as required per test instructions. Equipment must operate at the required temperatures and be compatible with the test fluid used. Gauge must not corrode in this test environment.

3.3 Test Vehicle/Test Piece. Not applicable.

3.4 Test Time.

Calendar time: 42 days
 Test hours: 168 hours to 1008 hours
 Coordination hours: Varies with tests required

3.5 Test Required Information. All specified properties are tested to the same time, temperature and fluid media conditions per Table 1 Flex Fuel in Oil Formulations and 4.2.2 Test Conditions.

3.5.1 Measured Properties/Methods/Conditions (Initial and After Test). Select the properties identified in see 1.1.3 and any additional specification/drawing requirements using the following standards (see 3.5.1.1 thru 3.5.1.10).

3.5.1.1 Tensile Strength. ASTM D412 Method A, Die C, ISO 37, Type 2 or DIN 53504 die S2.

3.5.1.2 Tensile Stress at 100% Elongation. ASTM D412 Method A, Die C, ISO 37, Type 2 or DIN 53504 die S2.

3.5.1.3 Elongation at Break. ASTM D412 Method A, Die C, ISO 37, Type 2 or DIN 53504 die S2.

3.5.1.4 Hardness. ASTM D2240 Shore A (slab/sheet) or ASTM D1415 and ISO 48 M, International Rubber Hardness Degree (IRHD) (parts).

3.5.1.5 Volume/Swell. ASTM D471 or ISO 1817.

3.5.1.6 Weight/Mass. ASTM D471 or ISO 1817.

3.5.1.7 Change in Length. ASTM D1460.

3.5.1.8 Appearance. See 4.2.2.8.3.

3.5.1.9 Compression Set. ASTM D395, Method B, Type 2 Specimen. Maintain load during cooling for 30 minutes to 2 h. Remove load and measure in 10 minutes or less. Optional use ISO 815-1, type B using the same post-test conditions.

3.5.1.10 Compressive Stress Relaxation. Report retained sealing force per GMW17113.

3.5.2 O-ring Properties. See 4.1.3. Use ASTM D1414 (section 5) on 9 mm Inside Diameter (ID) or larger provided they fit into pressure vessel to the following conditions (see 3.5.2.1 thru 3.5.2.7) and properties.

3.5.2.1 Tensile Strength, Elongation and Modulus.

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3.5.2.2 Test Spool Diameters. SAE ARP3050 provides recommended sizes for each ID range. Select the spool size that results in zero or minor stretch to the O-ring ID as it is positioned on the spools. See ASTM D1414 (Section 8) regarding stretch on spools.

3.5.2.3 Test Pull Rate. Is 500 mm $\pm$ 50 mm per minute (all sizes).

3.5.2.4 Micro-hardness. ASTM D1415, IRHD. Optional use ISO 48-M. See 4.1.3.1 Micro-hardness of Cross Sections and 4.1.6.5 Micro-hardness.

3.5.2.5 Compression Set, Low Temperature, Immersion in Fluids.

3.5.2.6 Compressive Stress Relaxation. See 4.1.5 Compression Stress Relaxation. SAE AS568-214 size rings may be used, but the button specimen is preferred.

3.5.2.7 All remaining properties are measured using ASTM D3182 slabs or buttons molded from production part conditions.

3.6 Personnel/Skills. Conform to company and/or laboratory requirements. Technician training records must be available upon request.

4 Procedure

4.1 Preparation.

4.1.1 Prepare five (5) specimens of each material being tested, for each test fuel being used, and for each property being examined. **Note:** An extra set of five (5) tensile specimens of each material will be needed to determine non-immersion properties. (ASTM D412 (Section 11 and Section 13.))

4.1.2 Specimens are to be cut from ASTM D3182 molded slabs (ASTM D412 (Section 11)). ASTM D3182 slab cure conditions must be the same as the production compound. Die cut specimens will be made on new or recently sharpened dies. Mark each sheet with Identification number (ASTM D412 (Section 11)).

4.1.3 O-Rings. Use actual production parts specimens with an inside diameter of 9 mm to 100 mm (SAE AS568-110 to -242 or ISO 3601). All other sizes are to be SAE AS568-214 molded to actual production part conditions. Follow ASTM D1414.

4.1.3.1 Micro-hardness of Cross Sections. The upper and lower surfaces of the cut cross section test piece shall be flat, smooth and parallel.

- Tolerance of Flatness: $t = 0.1$ mm according to ISO 1101
- Tolerance of Parallelism: $t = 0.2$ mm related to a diameter of 150 mm according to ISO 1101

4.1.4 Compression Set. The ASTM D395 Method B compression plate test fixture (no dimensions) must fit into the pressure vessel and allow proper positioning of test specimens for accurate measurement. To avoid interference with the magnetic stirrer, place the fixture on a platform whose height still permits full fluid immersion of the fixture/specimen.

4.1.5 Compression Stress Relaxation. Test conditions are designated as manual or automated (Elastocon) using GMW17113. Since automated systems are expensive, the majority of testing is with manual test fixtures. Follow Section 8 protocol and define the test conditions from GMW17113, Table 3 thru Table 7. The end of test time is always 1008 h and select any number of intervals between 0 h and 1008 h.

4.1.6 Specimen Sizes.

4.1.6.1 Tensile, tensile stress and elongation use Die C dimensions (ASTM D412 (Figure 2)) or ISO 37, type 2 or DIN 53504 die S2 dimensions.

4.1.6.2 Volume swell, mass, and appearance change use 25 mm x 50 mm x 2 mm specimen (ASTM D471 (Section 9) or ISO 1817 (Section 5)).

4.1.6.3 Dimension change use 100 mm x 1.6 mm x 2 mm (ASTM D1460 Apparatus A (Section 7)).

4.1.6.4 Compression set and compression stress relaxation, ASTM D395 Method B, Type 2 specimen or ISO 815-1, Type B.

4.1.6.5 Micro-hardness. Test pieces taken from elastomeric components shall have a test area of at least 4 mm x 6 mm, if possible, and a thickness of at least 1.5 mm. Generally the test area shall be 4 mm x 20 mm. Laminated test pieces are not permitted. Test pieces (cross sections) taken from elastomeric rings or profiles shall have a diameter of at least 2.0 mm and a thickness of 1.5 mm to 2.5 mm. The cut area shall be such that the distance between the points of test of the test piece to an edge shall be at least 0.75 mm. See 4.1.3.1.

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4.2 Conditions. All test specimens will be conditioned in accordance with test procedures used.

4.2.1 Environmental Conditions. Meeting the intent of the appropriate referenced test standards.

4.2.2 Test Conditions. Deviations from the requirements of this standard shall have been agreed upon. Such requirements shall be specified on component drawings, test certificates, reports, etc.

4.2.2.1 Test Fluid. The test fluid constituent information for gas engines is in GMW14914.

Table 1: Flex Fuel in Oil Formulations

Media	Test Fluid	Formula Constituents
Solution E: Ethanol Diluted E.O.	Ethanol/Oil: 919 mL GMW17332, 55 mL Reference Fuel C (RFC), 25 mL Ethanol, 1 mL Corrosive Water.	GMW14914
Solution M: Methanol Diluted E.O.	Methanol/Oil: 919.5 mL GMW17332, 55 mL Reference Fuel C, 25 mL Methanol, 0.5 mL Corrosive Water, 0.01 mg Cu ⁺ ion.	GMW14914

Note: E.O. = Engine Oil.

4.2.2.2 Test solution purity shall be at least American Chemical Society (ACS) reagent grade (denatured if appropriate).

4.2.2.2.1 Copper Ion Source. Use copper (II) sources from Copper Napthenate, Chemical Abstracts Service (CAS) 1338-02-9 available at Huls or Veba Chemie; Copper Cyclohexanecarboxylate, CAS 2218-80-6 from Eastman Kodak Number 10389; or Cupric Acetate, 1766 J. T. Baker, CAS 6046-93-1 (monohydrate, crystal).

4.2.2.2.2 Copper Ion Concentration. The amount of copper compound required to give 0.01 g copper per liter is calculated where the weight percent copper is obtained from the suppliers specification for the copper compound or it is determined by testing analysis; $[(0.01 \text{ mg Cu}) \div (\text{wt\% Cu in copper compound}) \times 0.01] = \text{mg Cu compound to be used}$. For cupric acetate, mix 10 mg/L of methanol, and for each liter of this solution, add 1 mL to every liter of diluted test fluid.

4.2.2.3 During the test, replace the test solution every 70 h for Condition A and every two (2) weeks for test periods beyond 336 h. Test fluids shall not be reused but can be analyzed for extraction information.

4.2.2.4 Test Period. If no condition is specified, Condition B is the default. Test times represent the total time at temperature. Test times are not to include down time to make test fluid changes.

4.2.2.4.1 Condition A. Is 336 h.

4.2.2.4.2 Condition B. Continue exposure to 1008 h (SAE J1748 (Section 5)).

4.2.2.4.3 Condition Z. As defined, but no less than 168 h.

4.2.2.5 Test Fluid Temperature. Is $120^\circ\text{C} \pm 3^\circ\text{C}$.

4.2.2.6 Test Pressure. Internal pressure during testing must not exceed 34.47 kPa (5 psig). Provisions in test hardware shall be made to control and monitor this required pressure.

4.2.3 Handling After Testing.

4.2.3.1 Dimension Change Specimens. Test to ASTM D1460 at $23^\circ\text{C} \pm 2^\circ\text{C}$. Remove the specimens from the test fuel and place in the measurement fixture. All dimensional measurements are taken while the specimen is wet and within 30 s of removing it from the fuel.

4.2.3.2 Change in Mass, Volume and Appearance (ASTM D471/ISO 1817).

4.2.3.2.1 After equilibrium or a predetermined test time is reached, remove the specimens from the pressure vessel and allow them to cool in the same test fluid at room temperature. The size and shape of the container must keep the specimens immersed. Specimens may be kept in the pressure vessel until test data is complete if that is desired (ASTM D471 (Section 10)).

4.2.3.2.2 Blot dry the specimen being careful not to disturb the surface and place in a tarred weighing bottle within 10 s to minimize evaporative weight loss. Weigh each specimen to the same level of precision as the initial weighing (ASTM D471 (Section 10)).

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