



Specifying Conditions for Compression Stress Relaxation Testing of Rubber and Thermoplastic Elastomers 90A or Less

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.

This document describes test procedures and conditions for determining the loss in counterforce of a standard specimen shape compressed at a constant deformation in specified media, under conditions of time and temperature. It is designed for testing of:

- a. Specimens of vulcanized rubber, and thermoplastic elastomers with instantaneous Shore A hardness from 45 to 90 (ASTM D2240), molded button (ASTM D395) and,
- b. Finished production articles of same hardness range (e.g., molded profile O-ring) per ASTM D3183.

1.1 Purpose.

1.1.1 This document is a supplement to ASTM D6147, SAE J2979 and ISO 3384 procedures for Compressive Stress Relaxation (CSR) testing.

1.1.2 Supplements. Supplemental features include:

- a. Mandatory classification of test conditions and requirements using a "line callout" designation. See 8.2 and 8.3. In 2013, the SAE International Committee on Automotive Rubber Specifications added a SR suffix to the J200M classification standard allowing compression stress relaxation to be specified using this industry classification standard. Unfortunately, the use of alpha-numeric codes to identify all the test parameters is limited. GM will continue to use this standard for identifying all relevant test conditions.
- b. Authorized Fixtures. See 3.2.3.
- c. For referee purposes, a designated system with data acquisition to provide continuous counterforce measurement for the specified test duration. See 3.2.2.
- d. Deviations from these industry procedures. See 1.3 through 1.3.3 and Appendix A, Table A1.

1.2 Applicability.

1.2.1 Specifying retained force for your compound will only reveal if it is capable of contributing to a sealing solution. It cannot evaluate sealing contribution in relation to design, construction/dimensional variation, and assembly. These contributions are evaluated in GMW3155 testing.

1.2.2 Under constant load (strain), rubber forms new crosslinks resulting in permanent physical deformation (change in shape). The intensity (stress) of the internal forces at constant deformation decreases over time and temperature. Stress relaxation is that measure of loss in force (stress) or the amount retained as a percent of the initial stress. This information is of practical value for studying materials for gaskets, hose clamped or with end fittings and other compressed applications.

1.2.3 The button and O-ring deflection is 15%, or 15% and 35% for manual and continuous measurement. Lower specimen deflection produces higher percent compression load loss. See 5.2.1.

1.2.4 Continuous CSR (Method B) runs the GMW3155 thermal cycle on elastomer compounds with a low temperature TR₁₀ of -25 °C or colder to define their low temperature performance.

1.3 Remarks. This document uses two (2) methods to measure counterforce; discontinuous (manual) or continuous at the specified media and test temperature. The two (2) methods differ significantly from Method A and Method B of ASTM D6147 and ISO 3384. Both GM methods load and start at the same temperature, but the end temperature measurements differ.

1.3.1 Method A (Manual – (M) at 23 °C). The test specimen compression and the initial measurement of the counterforce take place at $23\text{ °C} \pm 2\text{ °C}$. The test specimen is then stored in a chamber controlled at the test temperature, but it is removed from the chamber (or lowered to temperature) for each of the subsequent force measurements, which are made at $23\text{ °C} \pm 2\text{ °C}$. This is the only method for manual measurement of counterforce subjected to isothermal or cyclic temperatures. It is the most common method used for generating CSR data.

1.3.2 Method B (Continuous at Temperature). The test specimen compression and the initial measurement of the counterforce take place at $23\text{ °C} \pm 2\text{ °C}$. The test specimen is then stored in a chamber controlled to the test temperature or cycle. Measurements are made on a continuous basis at the designated temperatures. When designating this method, continuous measurement equipment is required. Make sure the source has access to this equipment.

1.3.3 SAE J2979 describes counterforce measurement by the intermittent (manual) method and only applies to Method A. Follow this procedure for manual measurement with the following exceptions:

1.3.3.1 Subsequent Test Time Intervals. Fixture cool down time (2 h) and measurement time (1 h) are not to be included in the test time. The only exception is when a single end of test measurement is specified in Table 3, as a sequence 5.

1.3.3.2 Test Pressure Vessel. See 3.2.5 for each media.

1.3.3.3 Test Specimens. O-ring provisions are not part of SAE J2979. See 3.3.6.

1.3.3.4 Duration of Test. Use the appropriate Table 3 code.

1.3.3.5 Specimen Molding to Testing. The minimum time permitted from compound finish mixing, molding (includes post-cure if appropriate) of test specimens is 72 h, not 24 h. See 3.3.

2 References

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

2.1 External Standards/Specifications.

ASTM D395	ASTM D3183	ISO 3384	SAE J2332
ASTM D471	ASTM D5423	ISO 17025	SAE J2979
ASTM D572	ASTM D6147	ISO 23529	SAE J200M
ASTM D883	IEEE/ASTM SI 10	ISO/IEC 811	
ASTM D1418	ISO 188	SAE AS568-214	
ASTM D2240	ISO 1629	SAE J2236	

2.2 GM Standards/Specifications.

9985809	GMW3155	GMW14914	GMW17332
9986153	GMW3356	GMW16444	GMW17918
9986333	GMW3420	GMW16678	

2.3 Additional References.

- Elastocon AB Tvinnargatan 25 SE-507 30 Bramhult, Sweden, website: www.elastocon.se.
- Grace Technology and Development, 8925 Highland Bay, Woodbury, MN 55125. Phone: (651) 731-6909 at <http://www.gracetdc.com/>.
- Parr Instrument Company, 211 Fifty Third Street, Moline, Illinois 61265-9984. Phone: 1-800-872-7720/(309) 762-7716, Fax (309) 762-9453, website: parr@parrinst.com.
- Tirreno Industry Company de Produtos Quimicos LTDA., Bandeirantes, 610 - Conceição, Diadema - SP, 09912-230, Brazil phone: +55 11 4053-3339. **Fax:** (11) 4056-5061 **E-mail:** tirreno@tirreno.com.br.

3 Resources

3.1 Facilities. A suitable area or room for conducting testing with a stable and known environment. See 4.2.1.

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. Use only the SAE J2979 automotive standard fixture for discontinuous testing in any media. This fixture meets ISO 3384 and ASTM D6147 requirements. For the 13 mm disk (D0), the standard jig includes base, cap and cap screws. Centering cup, three (3) wedges, shims, and indenter pin are extra. Also available is a fixture to accommodate the SAE AS568-214 O-ring including base, cap and cap screws. Larger centering cup, three (3) wedges, shims, and indenter pin are extra. See SAE J2979 Appendix A – Test Equipment.

Note: Fixture available from Classic Manufacturing Incorporated, 2980 Granada Lane North, Oakdale, MN 55128, Phone: (651) 770-1212, and Grace Technology and Development (see 2.3), 8925 Highland Bay, Woodbury, MN 55125. Phone: (651) 731-6909.

3.2.1 See SAE J2979 apparatus section. Each fixture has a means to center the *D0* specimen before, during, and after loading is complete.

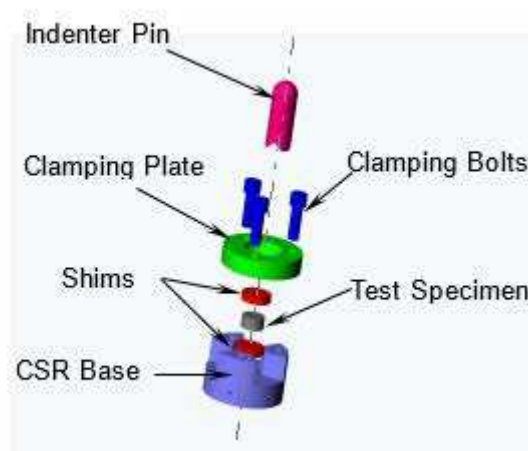


Figure 1: CSR Fixture Components

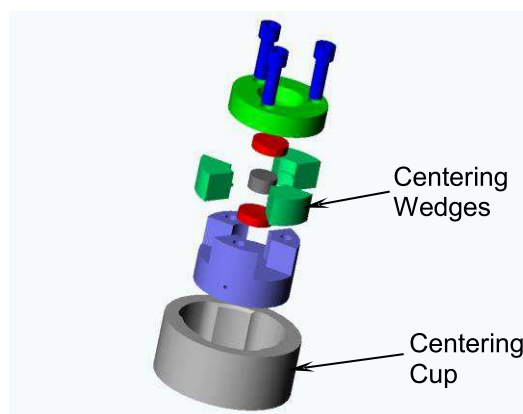


Figure 2: CSR Centering Elements

3.2.2 For referee purposes use Method B and stress relaxation and-creep/relaxation-ovens and-boxes ONLY from Elastocon AB (see 2.3) with temperature ranges from -40 °C to +200 °C for thermal cycle testing and +20 °C to +200 °C/300 °C for high temperature isothermal testing.

3.2.3 The automotive standard test jig (Figure 1 and Figure 2) and Elastocon test fixtures accept button and O-ring test specimens. Whether compressed in a circular groove or between circular plates, each specimen holder must have a through hole to permit fluid or balanced air pressure on the inside and outside the intact O-ring.

3.2.4 Ovens. Precise cabinet temperature control obtained through air exchange and circulation uniformity (in time and space) is critical to measurement repeatability and reproducibility at isothermal temperatures. Small units capable of obtaining ± 1.0 °C around a set temperature are best suited for manual CSR measurement. Select ovens with air exchange rates of three (3) changes per hour to twenty (20) changes per hour meeting ISO 188 and ISO/IEC 811 or ASTM D5423 Type I requirements.

3.2.5 Pressure Vessel. Use the unit described in GMW16678 for CSR measurement with fuel dilution in engine oil test solutions. Coolant testing shall be run in a pressure vessel. Oil testing can use any of the SAE J2979 equipment options. See 4.1.4.4 and Figure 3. For test mixtures requiring stirring to maintain uniformity, use the equipment shown in Figure 4. Provisions for a pressure gauge and relief valve are required. Use of a thermocouple attached to a stirring hot plate with temperature control (see 4.1.4.5). Use of a band heater is optional. Pressure vessel size/volume and number of CSR fixtures permitted is defined in GMW16678 for fuel dilution in engine oil and coolant and SAE J2979 for oil only.

Note: Available from Parr Instrument Company (see 2.3).



Figure 3: Pressure Vessel and Basket

3.2.5.1 Basket for Test Jigs (optional and info). In a suitable sized Parr Pressure Vessel, the 2 L basket holds eighteen(18) jigs.

3.2.6 Stirring Hot Plate with Temperature Control. The Corning 620D model is acceptable (see Figure 4).

3.2.7 Extensometer and Accessories. Measurement of counterforce on CSR manual test fixtures requires a power driven machine equipped to compress at a uniform rate of 1% per second in the test specimen. The machine must also have a suitable dynamometer, an indicator and recording system for measuring applied force. A 1 kN load cell and a captive guided spring displacement Linear Variable Differential Transformer (LVDT), stroke range 0.25 mm to 12.5 mm (0.01 in to 0.5 in) provide the proper load range and detect indenter pin movement respectively.

3.2.8 Thermocouple. Digital and capable of measuring for measuring fixture and test fluid temperatures within $\pm 2^\circ\text{C}$ of the specified temperatures. See SAE J2979.

3.2.9 All remaining items for manual equipment, see SAE J2979.

3.2.10 Equipment for fuel dilution in engine oil testing is described in GMW16678. The apparatus is shown in Figure 4 and Figure 5.

3.2.11 Manual Automotive Standard Device (ASD) Calibration Springs. Use different/appropriate load ranges and make repetitive measurements on test jig fixtures to determine if the measurement “system” (e.g., load frame stiffness and stability, load cell, supporting platform flatness) is robust, and independent of elastomer specimen influence. Report the sealing force Newtons (N), system stiffness Newton per millimeters (N/mm), spring (specimen) stiffness (N/mm) and stiffness ratio. Run a sufficient number to report results statistically. Standard Deviation (σ) for 20N to 200N spring load range is about 0.01 to 0.12.

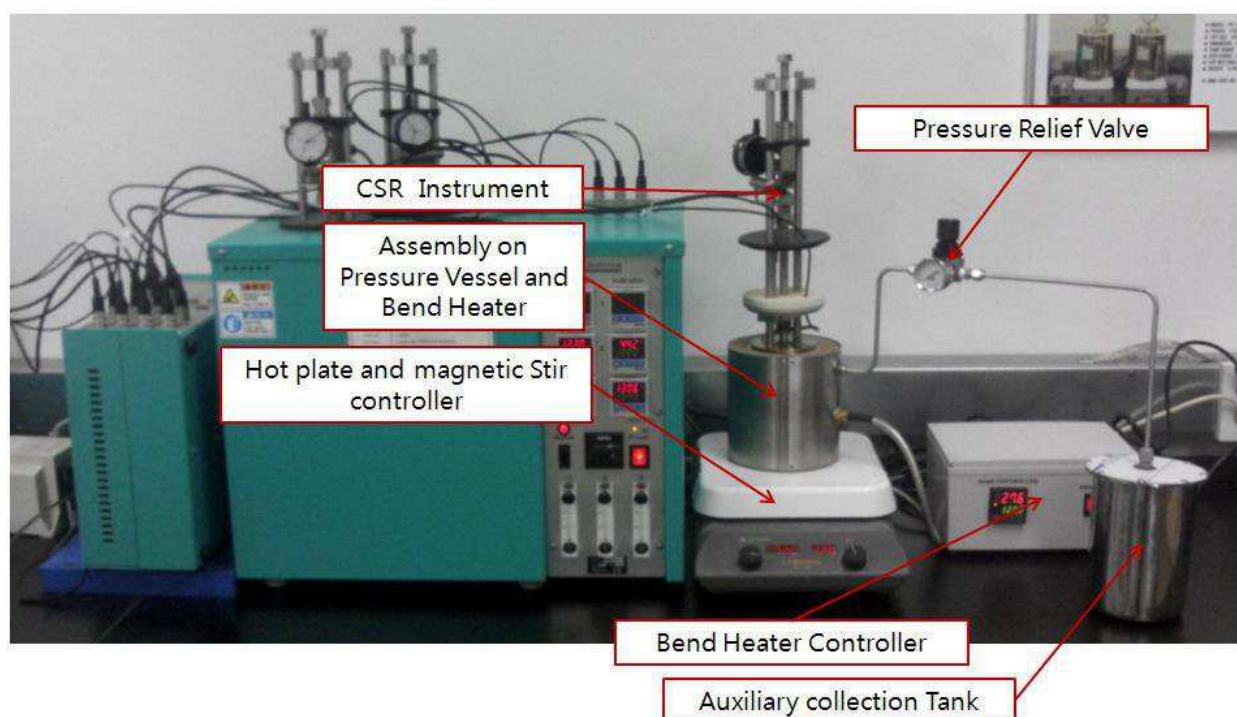
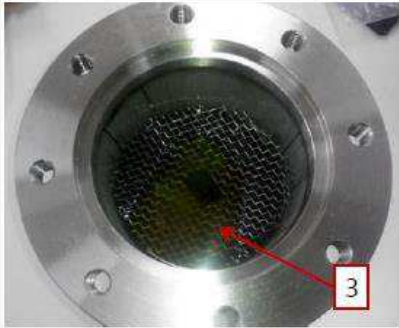


Figure 4: GMW16678 Test Setup
(Example from Dong-Ah Manufacturing Company)

	Picture	Description
Pressure Vessel		1. Material : Stainless Steel 2. Fluid volume / Vessel volume : 1100cc / 1600cc 3. hung test slab at mesh platform 4. Thermocouple : 1 place at top wall
Bend Heater and Insulation assembly		1. Bend Heater : 1 ea 2. Insulation : 2.1 max 400°C 2.2 Fiber glass material with metal barrier 3. Insulation cover : max 200°C

Note: ea = each, max = maximum.

Figure 5: Band Heater and Insulation Cover
(Example from Dong-Ah Manufacturing Company)

3.3 Test Vehicle/Test Piece. The minimum time permitted from compound finish mixing, molding (including post-cure) and the initial compression shall be 72 h. Longer times (up to 120 h) may be required to enable the test pieces to attain substantial equilibrium in accordance with ISO 23529.

3.3.1 Follow the test specimen section of SAE J2979 for the *D0* button only, including the remaining items in this section. Use three (3) specimens per test (see 3.2.3 referee device). Specimen dimensions are given in Table 7.

3.3.2 Mold *D0* specimens to the dimensions in Table 7. Make four (4) thickness measurements approximately 90 degrees apart. Discard the specimen if any one (1) of the four (4) measurements vary more than 0.02 mm. Measure using a metric dial gauge in a central area to avoid edge effects.

3.3.3 Do not use metal spacers to maintain a set sample compression; use the average of the four (1) specimen thickness readings to calculate each spacer height. The shims must give a $15\% \pm 1\%$ or $35\% \pm 1\%$ deflection.

3.3.4 The *D0* specimen shape factor (Table 7) is 0.50 to provide consistent results. Sample type (shape factor) will affect test results. Compare data only from the *D0* specimen size and shape tested in identical fixtures (see 3.2).

3.3.5 Lubricate test specimen before compression. A silicone fluid is recommended. Do not use grease as it may interfere with fixture function or accuracy. In some cases, the actual test fluid is appropriate as a lubricant.

Note: Dow Corning Corporation 200 silicone fluid meets the requirements.

3.3.6 Ring Profile Specimens (see Table 7) – For O-ring Specimens Only. A test fixture base can be modified to accommodate production part profiles, provided it allows the required compression level and does not affect test results. Indicate any modification in test reporting section.

3.3.7 O-ring Specimens. Use the SAE AS568-214 size O-ring.

3.3.7.1 Use 15% compression for O-ring specimens.