



State of Cure for Elastomeric Parts

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.

Note: The words **must**, **shall** or **will** as used in this document mean a mandatory requirement.

1.1 Purpose. A Design Release Engineer (DRE) has the option to specify proper cure on; the drawing, Statement of Requirements (SOR) or Component Technical Specification (CTS), using Dynamic Mechanical Analyzer (DMA)/Dynamic Mechanical Thermal Analyzer (DMTA), Nuclear Magnetic Resonance (NMR) or solvent extraction techniques, to establish a maximum Relative Cure Index (RCI) T2 (1) value for designated parts. This is recommended for; critical internal parts, compliance to motor vehicle safety standards, or "high cost to repair" situations.

1.1.1 Method A. This Method A is relevant if the DMA/DMTA data produced can discriminate between reference specimen cure conditions and provide accurate and a fast measure for the degree of cure using thermal mechanical analysis, intended for, but not exclusive of; Ethylene Acrylic (AEM), Fluorocarbon (FKM/FPM), Hydrogenated Nitrile Butadiene Rubber (HNBR), Polyacrylate (ACM), and Silicone (MVQ) elastomers. This Method A accommodates small to moderate size specimens with equipment available in most plant or independent test laboratories. As cure state increases, hysteresis decreases. Tan delta (δ) is a measure of hysteresis. As cure state increases, tan δ decreases (see 1.2.5). Usage of this Method A aids in determining the optimum production part process conditions for new compounds, verification of process conditions for existing compounds and analysis of suspect parts for degree of cure, no other usage of this Method A procedure is intended or recommended.

1.1.2 Method B. Use Method B if NMR is available. See Appendix B, B1.1 to B1.5.

1.1.3 Method C. Use Method C for solvent extraction technique or Swell Index Extracted (SIE). The solvent chosen has a significant effect on test results and shall only be selected by the compound owner. The RCI solvent values tend to be lower than NMR, however the reporting to develop the maximum RCI using the Appendix B, Table B1 would be the same. See Appendix B, B1.6.

1.2 Applicability.

1.2.1 Test new, unused, or assembled parts, not exposed to actual operating heat or fluids after assembly.

1.2.2 Parts from dynamometer durability, bench testing, field, warranty returns or consumer use are not to be evaluated.

1.2.3 The compound molder uses this procedure to setup optimum production part cure conditions as a reference, process verification, or when required for problem solving.

1.2.4 A T2 (1) cure index value below the maximum, means higher cure level.

1.2.5 Test Method A. Some types and grades of ACM Post Cure (PC) specimens can exhibit chemical structure rearrangement (low energy state) resulting in an increase in tan δ (decrease expected). This may imply a full PC that looks like a marginal press cure. If PC tan δ increases, avoid PC testing of these types of ACM compounds. (See Table 1). An alternative State of Cure (SOC) test method should be considered in this case.

Table 1: Tan δ of ACM PC Specimens

	PC/1 h	PC/2 h	PC/4 h
Tan δ	0.1408	0.1519	0.1732
Tan δ	0.1507	0.1599	0.1676
Tan δ	0.1655	0.1517	0.1826
Average	0.152	0.155	0.174
Standard	0.010	0.004	0.006

1.3 Remarks. This document describes methods for evaluating the SOC of Non-Post Cure (NPC) or PC elastomers. For NPC elastomers, a rheometry curve, e.g., Moving Die Rheometer (MDR), of the compound under evaluation is generated. At points along the curve (Tc 50, Tc 70, and Tc 90), specimens are removed and cooled to prevent further curing. Each Tc "x", e.g., time to a cure with "x" representing that percent cure level on the rheometer curve, specimen is tested using DMA/DMTA (Method A) to find the tan δ value. The part specimen tan δ value is measured and compared to the Tc "x" values taken at controlled points on the cure curve. For PC elastomers, tan δ values are compared from a known PC conditioned specimen to verify if the unknown specimen was PC. The results can confirm if the unknown was PC for a sufficient time at the correct temperature.

Note: It is worth noting that samples from the field where exposure to lubricants has occurred are not suitable for use with this method. Even after extraction of the lubricant has been performed, the Method A is not suitable in this case.

Note: All materials supplied to this standard must comply with GMW3059, Restricted and Reportable Substances, including the requirement to obtain a GM-approved International Material Data System (IMDS) submission.

2 References

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

2.1 External Standards/Specifications.

ASTM D2084	ASTM D5289	ISO 3417	ISO 6502
ASTM D3182	ASTM D5992		

2.2 GM Standards/Specifications.

GMW3059

2.3 Additional References.

- Component Technical Specification (CTS).
- Drawing.
- **Method A.** Alpha Technologies, Alpha Technologies, 6279 Hudson Crossing Parkway, 200 Hudson, Ohio 44236, USA, (www.alpha-technologies.com). Alpha Technologies Rheometer 2000 series or equivalent. TA Instruments, 159 Lukens Drive, New Castle, DE 19720, (info@tainstruments.com).
- **Method B.** Bruker Company, 40 Manning Road, Billerica, Massachusetts 01821, www.bruker.com. Bruker MQ20 Minispec or equivalent. Analysis Software, Origin Lab Corporation, 36 Washington Street, Suite 221, Wellesley Hills, Massachusetts 02481, (www.originlab.com).
- Statement of Requirements (SOR).

3 Resources

3.1 Facilities.

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.

3.1.3 A suitable area or room for conducting testing with a stable and known environment (see 4.2.1).

3.2 Equipment.

3.2.1 DMA/DMTA with Tension Mode (Method A). Instrument should be capable of low modulus measurements and minute $\tan \delta$ readings, e.g., 0.001 MPa and $\tan \delta$ increments of 0.00001 generated in tension. The decisive test of suitability will be the capacity to distinguish cured rubber from partially cured rubber. Unit must have a capability of measurements up to 150 °C at a rate to equilibrium not exceeding 15 minutes.

Note: DMA/DMTA with tension mode.

Note: Values in 3.2.1 are a reflection of the instrument sensitivity and not the reported $\tan \delta$ value.

3.2.2 Dynamic Mechanical Rheological Tester. Examples include the Rubber Process Analyzer (RPA), MDR or Oscillating Die Rheometer (ODR) that complies with ASTM D2084, ASTM D5289, ISO 3417 or ISO 6502. The RPA is a dynamic mechanical rheological test instrument designed to measure the properties of polymers and rubber compounds before, during, and after cure. The MDR measures both viscous and elastic components for cured and uncured dynamic properties. The ODR output data correlates with the degree of vulcanization as a function of cure time. The output can be displayed, graphically recorded or transmitted to a computer for further analysis. See Appendix A, A4.

Note: Alpha Technologies Rheometer 2000 series (www.alpha-technologies.com) or equivalent.

3.2.3 Ice Water Bath. Sufficient mix to maintain 0 °C to 2 °C for adequate cooling of NPC reference specimens.

3.3 Test Vehicle/Test Piece. A section of rubber, roughly rectangular or cylindrical, e.g., O-ring, cut to fit clamps. See Figure 1. Typical finished part specimens with 2 mm to 4 mm cross section must not affect test equipment results.

Figure 1: DMTA Clamp



3.3.1 Sampling Parts from Production Molds. Consider as many opportunities as is practical when sampling test specimens for example:

- Locate a critical design feature.
- From the coldest part of the tool (single or multiple cavity).
- Farthest from the gate.
- The center of the thickest cross section.

3.4 Test Time.

	Method A	Method B	Method C
Calendar time:	2 days (48 h)	2 days (48 h)	3 days (72 h)
Test time:	0.2 h	0.8 h	72 h
Coordination time:	48 h	48 h	96 h

3.5 Test Required Information. Report $\tan \delta$ values for the test specimen(s) along with those of the referenced Tc 50, Tc 70, and Tc 90, quenched and unquenched. Provide conclusion of findings as to adequate SOC.

3.6 Personnel/Skills. A technician with laboratory experience of one month or more proficiency and training in this technique and instrumentation is needed to perform this procedure.

4 Procedure

4.1 Preparation. Method A; trim DMA/DMTA specimens to approximate length, thickness, and width of 15 mm × 2 mm × 2 mm. Exact specimen dimensions are not critical to producing a satisfactory curve. Equipment that requires larger thickness may not be satisfactory. See Appendix A, A3.1.2.

4.1.1 For testing, known specimens should be run on the same machine as the baseline specimen.

4.2 Conditions.

4.2.1 Environmental Conditions. Office air conditioning to minimize dust, control temperature, and humidity. Room temperature is 23 °C ± 3 °C, 20% to 90% Relative Humidity (RH).

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.

4.2.3 Shipping and Handling of Test Specimens. Specimens for laboratory analysis must be kept sealed in non-reactive bags, free from outside contaminants and temperatures not exceeding 45 °C. Specimens of differing elastomer type and/or cure system must be in separate sealed bags.

4.3 Instructions. Preparation of reference specimens.

4.3.1 NPC Specimens.

4.3.1.1 Use MDR or ODR to generate reference specimens, follow the steps for cure per testing equipment (see 3.2.2). When selecting test temperature, pay special attention to the thermal lag of the instrument. Thermal lag is the time required for the upper and lower die to return to the prescribed temperature after loading the rubber into the die set. Typically, this is approximately 30 s. Therefore, the test time needs to reflect this thermal lag and accordingly the Scorch Time (t_{s2}), should always be more than the thermal lag time; i.e., $t_{s2} \geq 30$ s.

Note: If the thermal lag is not respected the instrument will not properly capture the curing reaction and the output will always be lagging and the constants that are to be determined from the curve will not be correct.

4.3.1.2 RPA Test Conditions. Test parameters will vary between cure testing equipment, thus different types of instruments may not give the same results.

4.3.1.3 Run the rheology to find times needed for Tc 50, Tc 70, and Tc 90 cure (see Figure 2). It may be necessary to run finished compounds at a lower temperature than usual. Aim for increments of at least 10 s between the reference specimens, to allow experimental discrimination.

4.3.1.4 By program or manual settings, open the cure testing equipment die at the prescribed time for Tc 50 reference specimen.

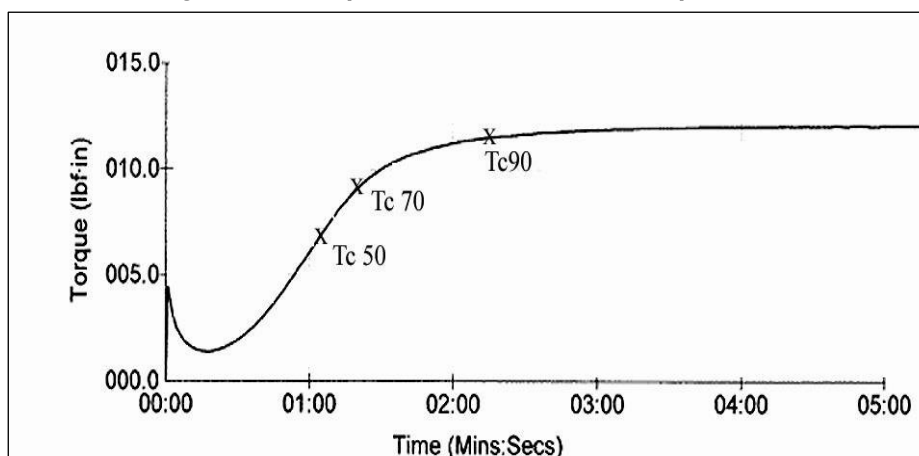
4.3.1.5 After removing the reference compound at the prescribed time, in < 15 s, immediately immerse the reference specimens in ice water bath for a minimum of 20 minutes. Due to the difficulty in removing ODR specimens, drop the rotor and specimen in the bath.

4.3.1.6 After 20 minutes or more, remove the reference specimens and blot dry. Seal the completely dry specimens in a labeled plastic bag.

4.3.1.7 Perform 4.3.1.3 through 4.3.1.5 for the reference Tc 70.

4.3.1.8 Perform 4.3.1.3 for Tc 90. Immediately cut the hot Tc 90 specimen in two pieces. One will be allowed to sit at ambient temperature overnight (16 h), as Tc 90 unquenched. The other will be quenched by immersion in ice water for a minimum of 20 minutes.

Figure 2: Example of MDR Scan of NPC Specimen



4.3.2 PC Specimens.

4.3.2.1 Prepare ASTM D3182 test slab by normal press cure conditions for the production compound.

4.3.2.2 Cut one slab into four sections and prepare 0 h, 1 h, and 2 h, and full PC at the PC temperature for that compound for the application under study. Place last three sections in a hot air circulating oven and record entry time and temperature. Remove specimens at the designated times and compound specific full PC time.

4.3.3 Method A DMA/DMTA Test Conditions. Use the following instrument settings in tension for NPC and PC specimens:

- Tensile clamp.
- Strain: 1.5%.
- Fixed frequency: 10 Hz.
- Clamp distance: 10 mm.
- Temperature: 100 °C.

4.3.3.1 Mount the reference or test specimen and trim off any excess with cutters. Close specimen compartment, and turn the oven on.

4.3.3.2 Start run when the temperature reaches 100 °C.

4.3.3.3 Output: Tan δ at 10 minutes. See Figure 3, Figure 4 and Figure 5.

4.3.4 Each compound reference studied must have adequate cure curve separation, e.g., Figure 3, to permit bracketing of the unknown test specimen cure state.