



State of Cure for Elastomeric Parts

1 Scope

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. Accurate and fast measure for degree of cure using thermal mechanical analysis intended for, but not exclusive of ethylene acrylic (AEM), fluorocarbon (FKM), hydrogenated nitrile butadiene rubber (HNBR), polyacrylate (ACM), and silicone (MVQ) elastomers. This procedure accommodates small to moderate size specimens with equipment available in most plant or independent test laboratories. As cure state increases, hysteresis decreases. Tan delta is a measure of hysteresis. As cure state increases, tan delta decreases (see 1.3.5). This procedure aids in the determination of 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 uses are intended or recommended.

1.2 Foreword. This document describes a procedure for evaluating the state of cure of non-post cured or post cured elastomers. For non-post cured elastomers, a rheometry curve (e.g., Moving Die Rheometer) 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" specimen is tested using Dynamic Mechanical Analysis to find the tan delta (δ) value. The part specimen tan delta is measured and compared to the Tc "x" values taken at controlled points on the cure curve. For post cured elastomers, tan delta values are compared from known post cure conditioned specimen to verify if the unknown specimen was post cured. The results can confirm if the unknown was post cured for a sufficient time at the correct temperature.

1.3 Applicability.

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

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

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

1.3.4 This procedure is relevant if the Dynamic Mechanical Thermal Analysis (DMTA) data produced can discriminate between reference specimen cure conditions.

1.3.5 Some types and grades of ACM post cure specimens can exhibit chemical structure rearrangement (low energy state) resulting in an increase in tan delta (decrease expected). This may imply a full post cure that looks like a marginal press cure. If post cure tan delta increases, avoid post cure testing of these types of ACM compounds. (See Table 1).

Table 1: Tan Delta of Polyacrylate Post Cure Specimens

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

2 References

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

2.1 External Standards/Specifications.

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

2.2 GM Standards/Specifications.

None

2.3 Additional References.

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

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 Dynamic Mechanical Analyzer (DMA/DMTA) with Tension Mode. 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. Rheometric Scientific, DMTA IV or equivalent.

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), Moving Die Rheometer (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 non-post cured 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.

3.3.1 Sampling Parts from Production Molds. Consider as many of these steps as is practical when sampling test specimens:

- 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.



Figure 1: DMTA Clamp

3.4 Test Time.

Calendar time: 2 days
Test hours: 0.1 hours to 0.2 hours
Coordination hours: 24 hours to 48 hours

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 state of cure.

3.6 Personnel/Skills. A technician with laboratory experience of one (1) month or more proficiency and training in this technique and instrumentation is needed to perform this procedure.

4 Procedure

4.1 Preparation. Trim DMA 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 No Post Cure Specimens.

4.3.1.1 Use Moving Die Rheometer or Oscillating Die Rheometer to generate reference specimens following the steps in the appropriate ASTM or ISO standard test method for cure testing equipment (3.2.2).

4.3.1.2 Rubber Process Analyzer 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.