Rubber Program Testing Instructions

Collaborative Testing Services, Inc.
Tel: 571-434-1925
Fax: 571-434-1937
rubber@cts-interlab.com

General Testing Instructions

Please review this important information before beginning testing.

Specific Testing Instructions

- 605-608: Tensile Properties of Precured Rubber
- 620: Hardness (Shore A)
- 621: Density
- 625: Hardness (Shore D)
- 630-633: Tensile Properties of Lab Cured Rubber
- 635: Compression Set
- 636: Tear Strength of Precured Rubber
- 640: O-Ring Tensile Properties
- 650: O-Ring Compression Set
- 660-664: Mooney Viscosity
- 684-689: Vulcanization Characteristics (MDR)
- 690-696: Rheological Properties (RPA)

General Testing Instructions

Please review the following information before testing.

- Participants in a multiple property test will notice that the results for each property are reported with a separate data entry form. It is not required that participants report results for all properties in multiple property test.
- Use the same instrument and procedure throughout a test.
- Report any variations.
- If the instrument fails during a test, contact CTS as soon as possible for instructions on how to proceed.
- Please submit all data entry forms using the online CTS Portal. If you are not reporting data for a test or property, leave the data entry form blank.
- Verify that selected units on the data entry forms are correct. If not, select and indicate one of the valid units listed on the data entry form (psi is the default unit for strength and stress values). The same unit must be used for all samples in a given property.
- Data must be submitted to CTS in its entirety by the data due date in order to be included in the analyses.

Tensile Properties of Precured Rubber

Analysis: 605 - 608
Test Method: ASTM D412
Pack: RT

NOTE: If also enrolled in the Hardness Test, perform that test before proceeding to the Tensile Test. The same samples are used for both.

Follow the instructions in ASTM D412. Prepare five dumbbell specimens from each plaque. Cut each dumbbell so that the lengthwise portion of the specimen is parallel to the grain direction. *Grain direction is indicated by an arrow*. Do not prepare ring specimens. Condition the samples for at least 3 hours at a temperature of 23 ± 2 °C, unless the samples were already conditioned prior to performing the Hardness Test. If you use a different test method that is comparable to D412, indicate this fact on the data entry form.

HOW TO REPORT DATA:

For Strength and Stress properties, report data in either *psi* (the default unit), *MPa* or kg/cm^2.

Data measured in *psi* should be reported as **whole numbers**. Data measured in *MPa* or kg/cm^2 should be reported with one or two places to the right of the decimal point.

Always report **Ultimate Elongation** values as percent in **whole numbers**.

Report data for those properties that are appropriate for your lab and equipment. Tensile Strength *must* be reported.

Hardness (Type A/ Shore A)

Analysis: 620
Pack: RT
Test Method: ASTM D2240

NOTE: If also enrolled in Tensile Properties of Precured Rubber, perform the Hardness test before proceeding to the Tensile test. The same samples are used for both.

Follow the instructions in ASTM D2240. Condition the samples for at least 3 hours at a temperature of 23 ± 2°C. Hardness is measured on a plied specimen formed by the five dumbbells cut from a single sheet for the tensile test. Take care to exclude air between plies when forming the specimen. Place a different dumbbell on top for each measurement, for a total of five measurements per sample. Measure hardness at enlarged end of dumbbell. Handle specimens with care since they are subsequently used for measuring tensile properties. If you use a different test method that is comparable to D2240, indicate this fact where asked on the data entry form.

HOW TO REPORT DATA: Report data up to the nearest 0.1 unit. [Whole numbers are also acceptable.]

The "CONDITIONS' section indicates the time at which hardness readings were taken in the previous test round (0-5 seconds, 5 secs, > 5 secs, or maximum hardness indicator). Update this information if necessary.

Density

Analysis: 621
Test Method: ASTM D297
Pack: RT

Follow the instructions in ASTM D297 for Density Testing (Section 16). Determine the density by use of a pycnometer, by hydrostatic weighings, or by compressed volume densimeter. Indicate in the conditions section of the data entry form which method was used. All determinations must be made with solutions at room temperature.

HOW TO REPORT DATA: **Report data to a minimum of 3 decimal places.**

Hardness (Type D/ Shore D)

Analysis: 625
Test Method: ASTM D2240
Pack: SD

Follow the instructions in ASTM D2240 for a Type D durometer. Condition the samples for at least 3 hours at a temperature of 23 ± 2°C.

Take five measurements on each specimen. Measurements should be at least 6.0 mm (0.24 in) apart.

HOW TO REPORT DATA: Report data up to the nearest 0.1 unit. [Whole numbers are also acceptable.]

The "CONDITIONS' section indicates the time at which hardness readings were taken in the previous test round (0-5 seconds, 5 secs, > 5 secs, or maximum hardness indicator). Update this information if necessary.

Tensile Properties of Lab-Cured Rubber

Analysis: 630-633
Test Method: ASTM D412 (Tensile) & ASTM D3182 (Curing)
Pack: UT

Keep samples refrigerated until curing. Cure the samples as soon as possible after receipt. Store the cured samples in an appropriate atmosphere until ready to condition and test.

Please check the weight of included samples before curing. If the weight exceeds 65g, trim the slab to approximately 65g. Please use caution. Trim a little at a time.

Cure at 320°F (160°C) for 10 minutes.

Refer to the Instructions below and to the instructions for Pack Sheet RT (PRECURED TENSILE SAMPLES) for conditioning, testing and reporting procedures.

TEST INSTRUCTIONS

*** Cure the samples as soon as possible after receipt. Store the cured samples in an appropriate atmosphere until ready to condition and test. ***

For the preparation of Standard Vulcanized Sheets, follow the instructions in ASTM D3182. The two plaques should be cured at 320°F for 10 minutes and molded into 6 by 6 by 0.08 inch samples. (Note that the thickness is approximate and may vary. Always use actual thickness measurements when computing stress area.)

After curing, condition the samples for at least 24 hours at a temperature of 23 ± 2°C before testing.

Continue to follow the instructions in ASTM D412, as well as the instructions for TENSILE PROPERTIES OF PRECURED SAMPLES found on Page 2 of these instructions, for testing the enclosed tensile samples and for reporting data.

Compression Set

Analysis: 635
Test Method: ASTM D395, Method B
Pack: CS

Sample Preparation: Condition samples for at least 3 hours at a temperature of 23 ± 2°C and 50 ± 10% relative humidity prior to performing the test.

NOTE: Verify sample thickness upon receipt. Transverse cutting and buffering of the samples per the guidelines in ASTM D395 may be needed to achieve the required thickness.

Sample Information: Follow the instructions in ASTM D395 Method B. Three Type 1 specimens have been provided. Measure thickness from the central portion of the compression button. Allow specimens to rest 30min before final measurement.

Samples should be tested at 70°C for 22 hours.

HOW TO REPORT DATA: Report data as percentage of the original deflection to the nearest whole number.

Tear Strength of Precured Rubber

Analysis: 636
Test Method: ASTM D624
Pack: TS

Follow the instructions in ASTM D624, using Die B. Condition the test pieces at a standard laboratory temperature of 23 ± 2 °C as defined in Practice D1349 for a minimum of 3 h before nicking or cutting.

Prepare five dumbbell specimens from each plaque. Cut each dumbbell so that the lengthwise portion of the specimen is parallel to the grain direction. *Grain direction is indicated by an arrow*. (Test pieces may be nicked or cut and tested immediately after conditioning but the maximum time between nicking or cutting and testing shall be 24 h.)

The rate of jaw separation shall be 500 ± 50 mm/min. (20 ± 2.0 in./min.).

For calculations, use maximum force, in N, for Type B specimens.

HOW TO REPORT DATA:

Report data in either *kN/m* (the default unit), or *lbf/in* (To convert from lbf/in to kN/m, multiply by 0.175.)

Data measured in *kN/m* should be reported with one or two places to the right of the decimal point.

O-Ring Tensile Properties

Analysis: 640-642, 647-649
Test Method: ASTM D1414
Pack: OR

Properties Measured: Tensile Strength at Break, Ultimate Elongation, Stress at 100% Elongation (100% Modulus), Hardness Shore A, Hardness Shore M, Density. Participants may choose which of these properties to measure and report.

NOTE: If reporting for the Hardness and Density Tests, perform those tests before proceeding to the Tensile Test. The same samples are used for both.

Test Parameters: Follow the parameters in ASTM D1414. Referenced within D1414 are ASTM D412, D2240, and D297.

Sample Information: Sufficient material has been provided to complete at least five measurements per sample. Additional sample material may be used for equipment set up or substitution if one of the specimens appears flawed.

Sample Preparation: Condition the samples for at least 3 hours at a temperature of 23 ± 2 °C

How to Report Data:

- Tensile Stress at Break – Report to the nearest 0.1 or 0.01 *MPa* or report in *psi*
  - Data measured in *psi* should be reported as whole numbers. Data measured in *MPa* should be reported with one or two places to the right of the decimal point.
- Ultimate Elongation – Report to nearest whole number for %
- 100% Modulus – Report to the nearest 0.1 or 0.01 *MPa* or report in *psi*
- Hardness Shore A & Shore M: Report data up to the nearest 0.1 unit. [Whole numbers are also acceptable.]
- Density: Report data to a minimum of 3 decimal places

O-Ring Compression Set

Analysis: 650
Test Method: ASTM D1414/D395
Pack: OC

Properties Measured: Compression Set.

Test Parameters: Follow the parameters in ASTM D1414. Referenced within D1414 is ASTM D395. Samples should be tested at 70°C for 22 hours.

Sample Information: Sufficient material has been provided to complete at least three measurements per sample.

Sample Preparation: Condition the samples for at least 3 hours at a temperature of 23 ± 2 °C

How to Report Data: Report data as percentage of the original deflection to the nearest 0.1 unit. [Whole numbers are also acceptable.]

Mooney Viscosity

Analysis: 660-661
Test Method: ASTM D1646 – Part A
Pack: MV

You will need both packages 1 & 2 to complete the test.

The samples do *not* require mill massing. The materials used in this testing cycle are described on the data entry form. Follow the procedure in ASTM D1646 on conditioning and preparing the specimens. Use the large rotor and a die temperature of 100°C. Allow the specimen to warm in the die cavity for one minute before starting the rotor. Three specimens from each of the four samples should be tested.

Specimens should be prepared so as to avoid the edges of the sample whenever possible.

HOW TO REPORT DATA: Report the data to the first decimal place.

(This deviates from the test method, which calls for whole numbers.)

For NBR and SBR samples, report data as **ML 1 + 4**. For butyl samples, report data as **ML 1 + 4** and/or **ML 1 + 8**.

If following a test method other than D1646, be sure to indicate this fact on the data entry form.

IMPORTANT: The use of film during testing is NOT recommended. If film is used, indicate the type of film used in the "comments" section of the data entry form. Your data entry form may indicate if film was used by your lab in the previous testing round. Update this information as necessary.

Film should never be placed against the rotor; film is only used between the die and the specimen.

Stress Relaxation

Analysis: 662-664
Test Method: ASTM D1646 – Part B
Pack: MV

Refer to ASTM D1646 - Part B - Measuring Stress Relaxation, *and* the instructions below for conducting the Mooney Stress Relaxation portion of the test.

Stress Relaxation must follow a viscosity test.

Labs are encouraged to use this time to

Practice this aspect of Mooney testing;

Verify that the equipment and software are operating properly;

Confirm that technicians are adequately trained to perform the test and interpret the data

Discover how Stress Relaxation fits into their QA/QC efforts.

Please note the test times:

For NBR or SBR samples - Relaxation **test run for 2.0 min after ML 1 + 4 test @ 100°C**

For butyl samples - **Relaxation test run for 2.0 min after ML 1 + 8 test @ 100°C**

There are three properties that can be analyzed in the Stress Relaxation test. Labs may report for one, two, or all three of the following properties:

1. $\mathbf{t_{80}}$ = Time (in seconds) from disk stop to 80% decay of the Mooney viscosity
2. $\mathbf{X_{30}}$ = Percent decay of the Mooney viscosity at 30 seconds after disk stop
3. **A** = area (in M-s) under the stress relaxation curve power law model for a time span from 1 s to t_f, the time of the end of the stress relaxation test

Important!: In order to analyze Stress Relaxation data, it is imperative that you indicate the *Mooney Viscometer manufacturer & model* and the *software* used on your data entry form.

Vulcanization Characteristics (MDR)

Analysis: 684-689
Test Method: ASTM 5289
Pack: MD

Test Method: Follow the instructions in ASTM D5289.

Test Conditions: Vulcanization temperature of **160°C** Amplitude of oscillation: **±.5° of arc**

Use only the film provided with the samples. Failure to use the enclosed film OR the use of a different film may generate non-comparable results.

Sample Preparation: Prepare three specimens of each sample such that a specimen has a volume of 3 to 5 cm^3. Refer to ASTM D5289 for additional guidelines on sample preparation. The specimens should be at room temperature (23 ± 2°C) before inserting into the instrument.

Test each specimen for a period of **15** minutes from closure of dies.

Properties: The following data should be recorded for each specimen:

1) the time period between closure of dies and increase of torque of 1 dN·m or 1 lbf·in above the minimum torque, t_S1;
2) the time period between closure of dies and a torque midway between minimum and maximum, t'50
3) the time period between closure of dies and a torque increase corresponding to ML + 0.9(MH - ML), t'90
4) the minimum torque
5) the maximum torque

HOW TO REPORT DATA: Report all values to one or two places to the right of the decimal point.

Record time properties (t_s1, t'50, t'90) in ***decimal minutes or seconds.*** Verify that you have indicated a valid unit for torque data *(lbf·in is the default unit)* on your data entry form. The same unit must be used for all samples in a given property. Labs have the option of reporting both t_s1 and t'10, neither property, or may choose one of the two.

Rheological Properties (RPA)

Analysis: 690-696
Test Method: ASTM D6204, Parts A&B
Pack: RP

Test Method: Follow the instructions in ASTM D6204.

Test Conditions: Please refer to ASTM Method D6204 for the full list of conditions for preconditioning, the conditioning step, Part A, and Part B. Refer to the data entry form to select the conditions for the appropriate material type. This will be EPDM, unless otherwise noted on the data entry form.

DO NOT perform a conditioning step *between* parts A and B of the test. Perform the conditioning step before Part A, and then proceed to Part A, followed by Part B.

Sample Preparation: Prepare three specimens of each sample such that a specimen has a volume which is appropriate for the test cavity volume of your instrument. Refer to ASTM D6204 for additional guidelines on sample preparation.

The specimens should be at room temperature (23 ± 3°C) before inserting into the instrument.

Properties: The following data should be recorded for each specimen:

(1) Part A: G'(storage shear modulus) at 20 Hz (Test Code 690)
(2) Part A: G" (loss shear modulus) at 20 Hz (Test Code 691)
(3) Part B: G'(storage shear modulus) at 1.0 Hz (Test Code 695)
(4) Part B: G" (loss shear modulus) at 1.0 Hz (Test Code 696)

HOW TO REPORT DATA: Report all values to one or two places to the right of the decimal point.

You are not required to report all properties.