



Collaborative Testing Services, Inc.

Rubber Specific Test Instructions

Please review the following information before testing.

It is not required that participants report results for all properties in multiple property tests.

Please return **all** data sheets to CTS for processing. If you are not reporting data for a test or property, RETURN THE BLANK DATA SHEET TO CTS.

If you are faxing the data sheets to CTS, you **MUST** use a cover sheet indicating the total number of pages you are sending. CTS will contact participants if the number of data sheets received is less than what is indicated on the cover page.

<u>Analysis</u>	<u>Pack</u>	<u>Analysis Name</u>
605-608	RT	Tensile Properties of Precured Rubber
620	RT	Hardness (Shore A)
621	RT	Density
625	SD	Hardness (Shore D)
630-633	UT	Tensile Properties of Lab Cured Rubber
660-661	MV	Mooney Viscosity
662-664	MV	Stress Relaxation
669-674	OD	Vulcanization Characteristics (ODR)
684-689	MD	Vulcanization Characteristics (MDR)
690-696	RP	Rheological Properties (RPA)

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 somewhere on the data sheet.

HOW TO REPORT DATA:

For Strength and Stress properties, report data in either *psi* (the default unit), *MPa* or *kg/cm²*.

Data measured in *psi* should be reported as **whole numbers**. Data measured in *Mpa* or *kg/cm²* 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.

Verify that pre-printed units on the data sheets are correct. If not, select and indicate one of the valid units listed on the data sheet (*psi is the default unit for strength and stress values*). The same unit must be used for all samples in a given property.

Hardness (Type A/ Shore A)

Analysis: 620 **Test Method:** ASTM D2240 **Pack:** RT

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 $23 \pm 2^{\circ}\text{C}$. 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 sheet.

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

The “CONDITIONS” section may indicate 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 datasheet 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 $23 \pm 2^{\circ}\text{C}$.

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 may indicate 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 $23 \pm 2^\circ\text{C}$ 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.

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 sheet. 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 sheet.

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 sheet. Your data sheet 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.

Mooney Stress Relaxation has been added to the Mooney Viscosity test as an on-going RESEARCH TEST. Stress Relaxation must follow a viscosity test. Stress Relaxation results should *not* be treated as proficiency test data.

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. **t₈₀** = Time (in seconds) from disk stop to 80% decay of the Mooney viscosity
2. **X₃₀** = 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 sheet.

Vulcanization Characteristics (ODR)

Analysis: 669-674 **Test Method:** ASTM D2084 **Pack:** OD

Refrigerate samples until ready to condition and test.

Follow the instructions in ASTM D2084. For this program, please perform all tests using a steel disc. If using a titanium disc, report this deviation from the test method on your data sheet. From each sample, prepare three specimens having a mass of 9 to 10 grams (volume 8 to 9 cm³). The specimens should be at room temperature ($23 \pm 2^\circ\text{C}$) before inserting into the instrument.

Test each specimen for a period of **30** minutes from closure of dies.

The following data should be recorded for each specimen:

- 1) the time period between closure of dies and a torque increase corresponding to $M_L + 0.1(M_H - M_L)$, t'_{10}
- 2) 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}
- 3) the time period between closure of dies and a torque midway between minimum and maximum, t'_{50}
- 4) the time period between closure of dies and a torque increase corresponding to $M_L + 0.9(M_H - M_L)$, t'_{90}
- 5) the minimum torque, M_L
- 6) the maximum torque

Test Conditions: vulcanization temperature of $160 \pm .5^\circ\text{C}$; Frequency of disc oscillation of 1.7 Hz (100 cycles per min);

Amplitude of one degree ($\pm 1^\circ$ arc):

Recommended torque ranges are 0 – 50 lbf·in or 0 – 100 lbf·in

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**.* Verify that you have indicated a valid unit for torque data (*lbf·in is the default unit*) on your data sheets. 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.

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 cm³. 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 sheets. 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 datasheet to select the conditions for the appropriate material type. This will be EPDM, unless otherwise noted on the datasheet.

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 ($23 \pm 3^{\circ}\text{C}$) 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.