Designation: D 3346 – 03

Standard Test Methods for Rubber Property—Processability of Emulsion SBR (Styrene-Butadiene Rubber) With the Mooney Viscometer (Delta Mooney)[1]

This standard is issued under the fixed designation D 3346; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods[2] explain the use of the shearing disk viscometer to obtain an indication of the processability of non-pigmented emulsion styrene-butadiene rubbers (SBR). They may also be used to separate those polymers that are easy to process from those that are difficult to process within a group of polymers of the same type.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)[3]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[3]

D 3896 Practice for Rubber from Synthetic Sources—Sampling[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

3. Terminology

3.1 *Definitions:*

3.1.1 Δ *Mooney* —the difference in Mooney viscosity recorded for a rubber at specified times during a test.

3.1.2 *Mooney Viscosity*—arbitrary measure of the viscosity of a rubber determined in a Mooney shearing disk viscometer, indicated by the torque required to rotate the disk embedded in a rubber specimen and enclosed in the die cavity under specified conditions.

4. Summary of Test Methods

4.1 In Test Method A, the difference in Mooney viscosity at 100°C (212°F) is determined at two specified times. Either massed or unmassed samples may be used.

4.2 In Test Method B, the Mooney viscosity difference for unmassed samples is determined between the minimum recorded directly after starting the rotor and the subsequent maximum (see Fig. 1).

5. Significance and Use

5.1 These empirical tests have been found to be suitable for ranking a series of unpigmented emulsion SBR in order of processability. They may also be used for comparing a production lot with a standard of known processability characteristics. The difference between Mooney viscosities at two specified times will rank those emulsion SBR polymers that differ appreciably in this property according to their processability. The actual values obtained for a given polymer depend on whether or not the sample was massed, and may vary between laboratories or with the type of machine used, and with the specified times at which Mooney viscosity values were taken.

6. Apparatus

6.1 The apparatus shall be in accordance with the Apparatus section of Test Methods D 1646.

6.2 The large rotor shall be used.

7. Sample Preparation

7.1 For sampling procedure, refer to Practice D 3896.

7.2 Condition the sample until it has reached room temperature of 23 ± 3°C (73 ± 5.4°F) throughout.

7.3 The tests can be performed using specimens taken from either massed or unmassed samples.

---

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.12 on Processability Tests.

Current edition approved June 10, 2003. Published June 2003. Originally approved in 1974. Last previous edition approved in 2000 as D 3346-00.

[2] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11–1005.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

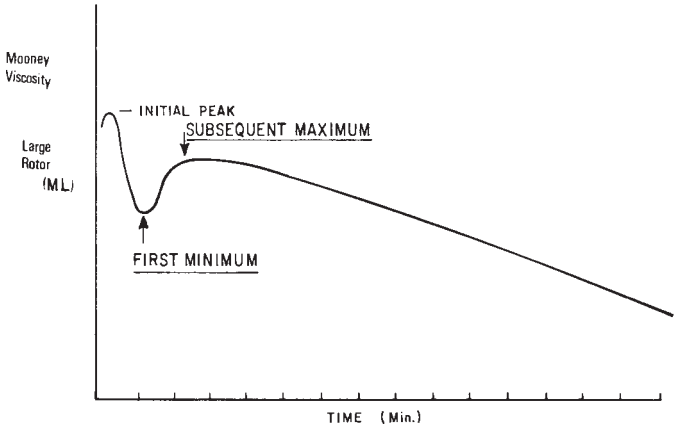

FIG. 1 Typical Mooney versus Time Curve for Processability Test of SBR

7.3.1 To mass a sample, pass 250 ± 5 g of the sample between the rolls of the standard laboratory mill described in Practice D 3182. The mill shall have a roll temperature of 50 ± 5°C (122 ± 9°F) and a distance between the rolls of 1.4 ± 0.1 mm (0.055 ± 0.005 in.) as determined by a lead slug. Immediately fold the sample in half and insert the folded end into the mill for a second pass. Repeat this procedure until a total of nine passes have been completed. Immediately insert the rubber without folding into the mill for a tenth pass. Do not allow the sample to rest between passes or to band on the mill rolls at any time.

7.3.2 Allow the massed samples to rest at room temperature for at least 30 min before preparing the specimens and measuring their viscosity.

7.4 Condition unmassed samples until they have attained room temperature throughout before preparing the specimens and measuring their viscosity.

8. Test Temperature

8.1 The test temperature shall be 100 ± 0.5°C (212 ± 9°F). For a description of a suitable temperature-measuring system, refer to 6.1.3 of Test Methods D 1646.

9. Calibration of Viscometer

9.1 Calibrate the shearing disk viscometer in accordance with the Specimen Preparation section of Test Methods D 1646.

10. Specimen Preparation

10.1 The test specimen shall consist of two pieces of the material being tested having a combined volume of 25 ± 3 cm^3. This volume is approximately 1.67 times the volume of the test cavity when the large rotor is used, and ensures that the cavity is completely filled. For convenience the mass of the test specimen of correct volume may be calculated as follows:

$$m = v \times d = 25\ cm^3 \times d \quad (1)$$

where:

m = mass, g.

v = volume in cm^3 = 25 cm^3, and

d = density in Mg/m^3 (g/cm^3).

NOTE 1—Mg/m^3 and g/cm^3 are numerically equivalent.

The test pieces shall be cut from the prepared sample and shall be of such dimensions that they will fit within the die cavity without projecting outside it before the viscometer is closed. A 45 mm (1.75 in.) diameter die may be used to assist in preparing the test pieces. A hole punched in the center of one of the test pieces facilitates the centering of the rotor stem. It shall not be permissible to slip the test piece around the rotor stem by cutting it edgewise. The test specimen shall be as free of air as it is practical to make it and shall be free of pockets which may trap air against the rotor and die surfaces.

11. Procedure

11.1 Measure the viscosity in accordance with the Procedure section of Test Methods D 1646, Part A. The duration of the test shall include a 1 min preheat followed by 15 min for massed samples and 7 min for unmassed samples.

11.2 Use the large rotor.

11.3 Use a rotor speed of 0.21 ± 0.002 rad/s (2.0 ± 0.02 r/min).

11.4 *Test Method A*— When the difference between Mooney viscosity at two specified times is required, calculate as follows:

ML 15 min – *ML* 1.5 min for massed samples, or

ML 7 min – *ML* 1 min for unmassed samples.

11.4.1 Negative numbers that are large in magnitude indicate good processability for the polymers tested.

11.5 *Test Method B*— When the difference required is between the minimum viscosity recorded and the subsequent maximum viscosity, calculate as follows:

$$ML_{max} - ML_{min}$$

where:

ML_{min} = minimum viscosity reached shortly after starting the rotor, and

ML_{max} = subsequent maximum viscosity.

11.5.1 The smaller the magnitude of $ML_{max} - ML_{min}$, the better the processability for the polymers tested.

11.5.2 When $ML_{max} - ML_{min}$ (see Fig. 1) is used as a measure of processability, readings must be taken at a minimum of 5 s intervals during the period when viscosity is rising after a rapid initial fall. The use of a recording device capable of following the complete viscosity-time curve is recommended.

11.6 The same processability ranking is obtained for a series of rubbers using specimens cut directly from the slab, or using massed samples as long as the same procedure is used throughout the series of viscosity tests.

12. Report

12.1 The report on the processability shall include the following information for the rubber(s) under test and for a reference sample:

12.1.1 Identification of the sample tested,

12.1.2 Method of sample preparation,

12.1.3 Temperature of test and point of measurement except for referee purposes where the complete temperature-time

curve shall be provided together with a description of the method of measurement,

12.1.4 Type of dies used, and

12.1.5 Time in minutes for which the specimen is permitted to warm up before starting the rotor.

12.1.6 *Test Results:*

12.1.6.1 *Test Method A*:

ML 15 min – *ML* 1.5 min for massed sample, or

ML 7 min – *ML* 1 min for unmassed sample.

12.1.6.2 *Test Method B*:

ML_{max} − ML_{min} (for unmassed sample).

13. Precision and Bias

13.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

13.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

13.3 A Type 1 (interlaboratory) precision was evaluated in 1989. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the value, as specified by these test methods, obtained on one determination of the property in question.

13.4 For Test Method A, both massed and unmassed, three materials (rubbers) were tested in six laboratories. For Test Method B, two materials (rubbers) were tested in six laboratories. For both Test Methods A and B all materials (rubbers) were tested on two separate days.

13.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1, in ascending order of material average or level, for each of the materials evaluated, and for each test method.

13.6 The precision of these test methods may be expressed in the format of the following statements that use an appropriate value of *r*, *R*, (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with an average level in Table 1 closest to the average level under consideration at any given time, for any given material in routine testing operations.

13.7 *Repeatability*— The repeatability, *r*, has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

13.8 *Reproducibility*— The reproducibility, *R*, has been established as the appropriate value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

13.9 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

13.10 When the average level or value of any measured property approaches zero, the value of (*r*) and (*R*) may approach very large values depending on the general degree of precision of the test method. This should be kept in mind in reviewing Table 1.

13.11 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for these test methods, since the value of the test property is exclusively defined by the test methods. Bias therefore cannot be determined.

TABLE 1 Type 1 Precision

NOTE—
Sr = Repeatability standard deviation, in measurement units,
r = Repeatability, in measurement units,
(r) = Repeatability, (relative) percent,
SR = Reproducibility standard deviation, in measurement units,
R = Reproducibility, in measurement units, and
(R) = Reproducibility, (relative) percent.

Material	Average Level	Within Laboratory			Between Laboratories		
		Sr	*r*	(*r*)[A]	*SR*	*R*	(*R*)[A]
Part 1—Test Method A—Massed							
Rubber 3	−23.5	0.89	2.52	10.7	1.26	3.56	15.1
Rubber 2	−15.4	0.81	2.30	14.9	1.28	3.62	23.4
Rubber 1	−11.9	0.82	2.32	19.4	1.61	4.56	38.3
Part 2—Test Method A—Unmassed							
Rubber 3	−9.8	0.42	1.18	12.0	0.73	2.08	21.2
Rubber 2	−1.1	0.79	2.24	(208)	0.79	2.24	(208)
Rubber 1	2.8	0.46	1.31	46	0.70	1.99	70.0
Part 3—Test Method B—Unmassed							
Rubber 2	1.48	0.079	0.22	15.1	0.39	1.11	75.2
Rubber 1	3.19	0.56	1.59	49.7	0.60	1.70	53.3

[A] See discussion in 13.10 on (*r*) and (*R*).

14. Keywords

14.1 Δ Mooney; Mooney; processability; SBR; styrene-butadiene rubber; viscosity