Designation: D4620 – 02 (Reapproved 2017)

# Standard Test Method for Evaluating the Effective Surface Area of Zinc Oxide in Rubber[1]

This standard is issued under the fixed designation D4620; 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 This test method specifies the standard materials, mixing procedure, and test method for ranking zinc-oxide samples according to their specific surface area in a standard test formula based on chloroprene rubber (CR).

1.2 The ranking is based on the cure time of the standard CR formulation.

1.3 The accurate surface area cannot be determined by this test method, since factors other than surface area may influence the cure times to some extent (for example acidity, heavy metal traces, etc.).

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

1.5 *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:*[2]

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D3190 Test Method for Rubber—Evaluation of Chloroprene Rubber (CR)

D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

## 3. Summary of Test Method

3.1 Separate formulations of a chloroprene-rubber compound are prepared containing 3-methyl-thiazolidine-thione-2 and other compounding materials. One or more formulation(s) contain the experimental or candidate zinc-oxide grade(s) to be evaluated and the other standard formulation(s) one or more zinc-oxide grade(s) of known surface area.

3.2 The cure times (t'90) of the formulations are measured as specified in Test Method D2084 or D5289 and used to rank the unknown zinc-oxide with respect to the standard containing zinc oxide of known surface area.

3.3 Longer cure times indicate lower surface areas, and shorter cure times indicate higher surface areas.

## 4. Significance and Use

4.1 The surface area of zinc-oxide in rubber can significantly affect cure activation and vulcanizate properties.

4.2 The specific surface area of zinc-oxide is usually measured by nitrogen absorption which requires the use of equipment not normally found in rubber laboratories. This test method allows a ranking of zinc-oxide samples according to their surface areas with respect to a known standard using a simplified procedure involving mixing of rubber compounds and measuring cure times with oscillating disk or rotorless cure meters.

4.3 This test method may be used for quality control, research and development work, and comparison of different zinc-oxide samples.

## 5. Standard Test Formula

5.1 The standard test formula is similar to Formula B in Test Method D3190, except for the curative (see Table 1).

5.2 For meaningful results, it is necessary to use the same lot of raw materials within each test series.

## 6. Mixing Procedures

6.1 The mill mixing procedure outlined in Test Method D3190 (6.1.2., Method B and 6.4) is employed.

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**TABLE 1 Standard Test Formula**

| Materials | Parts by Mass |
|---|---|
| Chloroprene-rubber (mercaptan modified)[A] | 100.0 |
| Antioxidant[B] | 1.0 |
| Magnesium-oxide[C] | 4.0 |
| N 774 black | 30.0 |
| Zinc-oxide | 5.0 |
| 3-methyl-thiazolidine-thione-2 (curative)[D] | 0.5 |
| | 140.5 |

[A] Mooney viscosity 40–50 (ML 1 + 4' at 100°C (212°F), see Test Methods D1646) medium crystallization rate.
[B] Octylated diphenylamine.
[C] Scorchguard O available from Rhein Chemical Corp., 1008 Whitehead Rd. Ext., Trenton, NJ 08638.
[D] Vulkacit CRV/LG available from Miles, Inc., Polysar Rubber Division, 2603 W. Market St., Akron, OH 44313 or Bayer AG., Business Group Rubber, 5090 Leverkusen, Germany.

6.2 Mill-roll temperatures are adjusted to 50 ± 5°C (122 ± 9°F) and stock temperatures should reach 75 ± 5°C (167 ± 9°F) to assure melting and satisfactory dispersion of the curative.

6.3 It is recommended that the 3-methyl-thiazolidine-thione-2 be pulverized in order to prevent any loss by granules popping off the mill during the mixing cycle.

## 7. Testing

7.1 Individual formulations containing one or more zinc-oxide samples of unknown and known surface areas are mixed and t'90 times are measured in accordance with Test Method D2084 or Test Method D5289, using a temperature of 160°C (320°F).

7.2 The t'90 times of the formulation(s) containing zinc-oxide of unknown surface area are ranked with respect to the known standard(s) and each other.

7.3 As zinc-oxide standard IRM 91[3] or any other grade of known surface area, or both can be employed.

7.4 Similar t'90 times indicate similar surface areas, compared to the standard.

7.5 Shorter t'90 times indicate a higher surface area compared to the standard.

7.6 Longer t'90 times indicate a lower surface area compared to the standard.

## 8. Report

8.1 The report shall include the following information:
8.1.1 Proper identification of samples,
8.1.2 Cure meter results: t'90 times, test method used, and
8.1.3 Ranking of samples according to t'90 times, indicating whether the samples have a higher, lower, or similar surface area compared to the known standard(s).

## 9. Precision and Bias

9.1 A precision and bias statement is not applicable, since this test method employs data only to provide a ranking of samples with respect to each other.

## 10. Keywords

10.1 curemeter; surface area; zinc oxide

[3] Available from R. E. Carroll, Inc., P.O. Box 5806 Trenton, NJ 08638–0806.