Designation: D4315 – 94 (Reapproved 2017)

Standard Test Methods for Rubber Compounding Material—Zinc Oxide[1]

This standard is issued under the fixed designation D4315; 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 cover chemical and physical tests for zinc oxide that are considered to be important to its use in rubber compounding.

1.2 The tests appear in the following order:

Test	Section
Zinc Oxide	5
Lead and Cadmium	6
Sulfur	7
Heat Loss	8
Surface Area	9
Sieve Residue	10

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* For specific precautions, see 5.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments

D1193 Specification for Reagent Water

D3037 Test Method for Carbon Black—Surface Area by Nitrogen Adsorption (Withdrawn 1999)[3]

D3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing (Withdrawn 1987)[3]

D3280 Test Methods for Analysis of White Zinc Pigments

D4075 Test Methods for Rubber Compounding Materials—Flame Atomic Absorption Analysis—Determination of Metals

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 These test methods may be used for quality control of zinc oxide used as a compounding material and for research and development studies where zinc oxide is used in a rubber mix.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water in accordance with Specification D1193.

ZINC OXIDE

5. Procedure for Determination of % Zinc Oxide

5.1 Analysis of white zinc pigments is covered in Test Methods D3280.

5.2 Reagents and safety precautions for Test Methods D3280 apply.

5.3 Section 6 of Test Methods D3280 describes the analysis of total zinc in zinc oxide using diphenylamine as internal indicator.

5.4 Section 7 of Test Methods D3280 describes the analysis of total zinc in zinc oxide using uranyl acetate as external indicator.

LEAD AND CADMIUM

6. Procedure for Determination of Lead and Cadmium in Zinc Oxide

6.1 Test Methods D4075 is the standard test method for metal analysis in compounding materials using the flame atomic absorption technique. Analysis for cadmium and lead in zinc oxide are described in Sections 11 through 20 of Test Methods D4075.

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

SULFUR

7. Procedure for Determination of Sulfur in Zinc Oxide

7.1 Analysis of white zinc pigments is covered in Test Methods D3280.

7.2 Reagents and safety precautions for Test Methods D3280 apply.

7.3 Section 9 of Test Methods D3280 describes the analysis of total sulfur in zinc oxide.

HEAT LOSS

8. Procedure for Determining Heat Loss in Zinc Oxide

8.1 Use Test Methods D280, Method A, for pigments that do not decompose at 110°C.

SURFACE AREA

9. Surface Area of Zinc Oxide

9.1 Use Procedure D of Test Methods D3037. Test Methods D3037 are written for carbon black using a flowing gas surface area analyzer. It shall be followed with the following exceptions:

9.2 Sections 33.11 and 33.12 shall read:

Accurately weigh a clean, dry, empty sample cell and record the mass. Using a small funnel, pour the zinc oxide into the sample cell using the following table as a guide for the approximate sample mass:

Surface Area, 10^3 m^2/kg	Zinc Oxide Mass, g
9	0.20–0.35
4	0.50–0.70

9.3 Section 33.16 shall read:

Outgas the Zinc Oxide at 200°C for 20min

9.4 Section 33.21 shall read:

An attenuator setting of one is usually satisfactory for the surface areas and masses.

NOTE 1—See 9.2.

9.5 Sections 33.33.1, 33.33.2 and 33.33.3 should be disregarded as not relevant as there are currently no ASTM approved reference standards for zinc oxide surface areas.

SIEVE RESIDUE

10. Procedure for Sieve Residue of Zinc Oxide

10.1 *Apparatus:*

10.1.1 *Sieves,* 75-mm (3-in.) or 200-mm (8-in.) with 45-µm (325-mesh) wire cloth.[4]

10.1.2 *Balance,* with a sensitivity of ±0.1 mg.

10.1.3 *Drying Oven,* maintained at 100 ± 10°C.

10.1.4 *Weighing Dish.*

10.1.5 *Aerator,* for hose connected to a tap-water supply.

10.2 *Reagents:*

10.2.1 *Tap Water.*

10.3 *Sampling:*

10.3.1 Sampling shall be at the discretion of the analyst to obtain a representative sample of the lot to be tested.

10.4 *Procedure:*

10.4.1 Weigh a 10 to 100-g sample to ±0.1 g in a suitable weighing dish. Small samples are used with the 75-mm (3-in.) sieve, large samples for the 200-mm (8-in.) sieve.

10.4.2 Place the sample in the sieve and wash with a stream of tap water, directed onto the sieve from a hose. If any lumps are detected after most of the zinc oxide has passed through the sieve, gently manipulate the lumps to break them up using finger tips or a small brush. This operation serves to break up any persistent lumps of zinc oxide but is not stringent enough to force coarse, hard particles through the sieve. Continue washing with tap water until no more residue will pass through the sieve. When no more residue will pass through the sieve, place the sieve in an oven maintained at 100°C until it is dry.

10.4.3 Remove the sieve from the oven and transfer any weighable residue to a small weighing dish, which has been previously weighed to ±0.1 mg and weigh.

10.5 *Calculations:*

10.5.1 Calculate the percent sieve residue R as follows:

$$R = W/S \times 100 \quad (1)$$

where:

R = sieve residue, %,
W = mass of sieve residue, g, and
S = mass of sample, g.

10.6 *Report:*

10.6.1 The report shall include the following information:

10.6.1.1 Proper identification of the sample.

10.6.1.2 Results of two individual percent sieve residue determinations and their average.

11. Precision and Bias

11.1 Precision and bias statements are in the process of being prepared in the form specified by Practice D3040. They will be added to these test methods when completed.

12. Keywords

12.1 chemical and physical tests for zinc oxide; heat loss; lead and cadmium in zinc oxide; sieve residue; sulfur in zinc oxide; surface area; zinc oxide; zinc oxide percent

[4] Detailed requirements for this sieve are given in Specification E11.