Designation: D6814 – 02 (Reapproved 2013)

Standard Test Method for Determination of Percent Devulcanization of Crumb Rubber Based on Crosslink Density[1]

This standard is issued under the fixed designation D6814; 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 covers the procedure for determining percent devulcanization from crosslink density measurements of devulcanized rubber and control crumb rubber in the laboratory. Percent devulcanization is a quantitative determination.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

D297 Test Methods for Rubber Products—Chemical Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 *rubber, n*—natural or synthetic elastomer that has been chemically crosslinked/vulcanized to enhance its useful properties.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *devulcanization, n*—a process of breaking down chemical crosslinks in cured rubber.

4. Summary of Test Method

4.1 The crumb rubber sample is extracted in hot acetone per Test Methods D297 and dried at 70 ± 2°C in a forced-ventilating air oven for 16 ± 1 h. The dried crumb rubber is swollen in a solvent (for examples of polymer-solvent pairing, see Appendix X1) with reagent grade purity, selection based on rubber type, for 24 h at room temperature. The solvent is replaced with fresh solvent three times during the swelling process. After swelling, the solvent is wiped quickly from the surface of the swollen crumb rubber using a clean paper towel. Measure the weight of the swollen sample in a preweighted and tared weighing bottle with closure. Dry it at 70 ± 2°C in a forced-ventilating air oven for 16 ± 1 h. Cool to room temperature in a desiccator and weigh. The density of the dried crumb rubber sample is measured using methanol instead of distilled water as an immersion liquid because of good wettability of methanol to rubber. Using the swelling ratio, polymer density, polymer-solvent interaction parameter, and the Flory-Rehner equation,[3] the crosslink density of the sample is calculated. Percent devulcanization is calculated using crosslinking density data of devulcanized crumb rubber and the control.

5. Significance and Use

5.1 It is important for rubber compounders to know the extent of devulcanization a rubber might have undergone during recycling. It allows the compounder to determine if more curing agents are needed during mixing of devulcanized rubber when used either as partial replacement or stand alone.

6. Apparatus

6.1 *Analytical Balance,* precision 0.0001 g.

6.2 *Extraction Apparatus,* glass conical flask, extraction cup, and condenser.

6.3 *Beaker,* 50 mL.

6.4 *Oven,* temperature controlled within ±2°C.

6.5 *Paper Towel.*

6.6 *Weighing Bottles with Caps.*

7. Reagents and Materials

7.1 The following reagents are used:

7.1.1 *Acetone,* USP grade,

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber 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.

[3] Flory, P. J., *Principles of Polymer Chemistry*, Cornell University Press, Ithaca, NY, 1953, Chap. 13 (Equation 38), p. 578.