Designation: D 5902 – 05

Standard Test Method for Rubber—Determination of Residual Unsaturation in Hydrogenated Nitrile Rubber (HNBR) by Iodine Value[1]

This standard is issued under the fixed designation D 5902; 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 Wiijs procedure for the determination of unsaturation (iodine value) in hydrogenated nitrile rubbers.

1.2 This test method is applicable only to those hydrogenated nitrile rubbers derived from copolymers of acrylonitrile and butadiene.

1.3 Iodine values are reported in centigrams of iodine per gram of HNBR [cg(I_2)/g]. Higher iodine values indicate higher levels of unsaturation.

1.4 This test method is appropriate for calculating the percent residual unsaturation of hydrogenated nitrile rubber if the iodine value of the base polymer before hydrogenation has been determined.

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]

D 1193 Specification for Reagent Water

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 A sample of the raw, unvulcanized rubber is dissolved in chloroform.

3.2 The dissolved sample is reacted with Wiijs solution.

3.3 When the reaction is completed, potassium iodide solution is added.

3.4 The resultant solution is then back-titrated with sodium thiosulfate solution and the iodine value is calculated.

4. Significance and Use

4.1 Hydrogenated nitrile rubbers are available at different levels of unsaturation and different acrylonitrile content. Highly saturated grades offer optimum resistance to aging, such as exposure to heat, ozone and chemicals, and can be cured effectively only with peroxides or high energy radiation.

4.2 Partially unsaturated grades can be cured by sulfur systems in addition to peroxides and high energy radiation.

4.3 This test method provides a technique to determine the unsaturation level of hydrogenated nitrile rubbers in the raw, unvulcanized state. It can be used for research and development, quality control, and referee purposes.

5. Apparatus

5.1 *Erlenmeyer Flasks*, with ground glass stoppers (300 mL).

5.2 *Flask Shaker.*

5.3 *Pipets.*

5.4 *Constant Temperature Bath.*

6. Reagents

6.1 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I of Specification D 1193.

6.3 *Chloroform.*

6.4 *Wiijs Solution* (prepared in accordance with Test Method D 1959).

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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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] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.