



Designation: D8154 – 17

Standard Test Methods for ¹H-NMR Determination of Ketone-Ethylene-Ester and Polyvinyl Chloride Contents in KEE-PVC Roofing Fabrics¹

This standard is issued under the fixed designation D8154; 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 pertains to the determination of the relative contents of Ketone-Ethylene-Ester (KEE) and Polyvinyl Chloride (PVC) after their extraction from reinforced roofing membranes, or fabrics. Based on Proton Nuclear Magnetic Resonance Spectroscopy (¹H-NMR), the method allows for the quantification of PVC with respect to an internal standard. The KEE content is then obtained by difference. The test method is not applicable to membranes or blends that contain high molecular weight polymers other than PVC and KEE.

1.2 *Units*—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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1079 Terminology Relating to Roofing and Waterproofing
D6754/D6754M Specification for Ketone Ethylene Ester Based Sheet Roofing

¹ This test method is under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and is the direct responsibility of Subcommittee D08.18 on Nonbituminous Organic Roof Coverings.

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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. Terminology

3.1 *Definitions*—Terminology **D1079** and Specification **D6754/D6754M** shall apply to this test method.

4. Summary of Test Method

4.1 *Test Methods*—There are two related test methods to quantify PVC and KEE in roofing fabrics. In both methods the polymer blend is extracted from the fabric and purified by a series of dissolution, centrifugation, and precipitation. After drying, the blend is dissolved in deuterated tetrahydrofuran along with a ¹H-NMR internal standard. In Method A, PVC content is quantified by comparing its ¹H-NMR signal with that of the internal standard, and KEE is obtained by difference. Method B adds to Method A a calibration curve the slope of which providing for a correction factor. This correction factor is applied to the polymer content obtained by Method A to provide for greater precision on the PVC and KEE contents.

5. Significance and Use

5.1 Determination of the percentage of KEE compound in sheet roofing is of concern to many specifiers and building owners. Method A, the faster method, is best used for quality control. Method B may apply better to research and development, or investigative work.

6. Interferences

6.1 The NMR analysis of the polymer blend extracted from the sheet roofing is sensitive to impurities, including plasticizer, filler, reinforcement, and trace solvent (other than the NMR solvent). The procedure here is meant to purify the blend for proper analysis.

7. Equipment and Apparatus

7.1 *Glassware*. Erlenmeyer flask and stopper, 125 mL. Weighing bottle, 25 mL.

7.2 *Porcelain ware*. Büchner funnel, 10 cm in diameter.

7.3 *Platform shaker*. Must accept 125 mL Erlenmeyer, and allow for swirling at 120 to 150 rpm.

7.4 *Metal spatula*. To stir the suspension during the precipitation step.