



Designation: D2124 – 99 (Reapproved 2011)

Standard Test Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique¹

This standard is issued under the fixed designation D2124; 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 provides for the identification of certain resins, plasticizers, stabilizers, and fillers in poly(vinyl chloride) (PVC) compounds by an infrared spectrophotometric technique. In many cases, individual components may be measured quantitatively. Complementary procedures, such as chromatographic and other separations, will be necessary to separate specific components and extend the applications of this test method. Other instrumental test methods, such as optical emission or X-ray spectroscopic methods, may yield complementary information which may allow more complete or, in some cases, easier measurement of the components. The resin components covered in this test method are listed in the appendix.

1.2 PVC formulations are too varied to be covered adequately by a single test method. Using the following test method, many compounds may be separated into resins, plasticizers, stabilizers, and fillers. A number of components can be quantitatively measured. Many more can be identified and their concentrations estimated. By the use of prepared standards, one may determine the usefulness and accuracy of the test method for specific PVC formulations. This test method is applicable for the resin components listed in the appendix and for other components having similar chemical compositions and solubility characteristics. This test method can lead to error in cases where the nature of the components is not known.

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

1.4 *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.*

NOTE 1—There is no known ISO equivalent to this standard.

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.08).

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2. Referenced Documents

2.1 ASTM Standards:²

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 Definitions:

3.1.1 For definitions related to the material on infrared spectroscopy, refer to Terminology E131.

4. Summary of Test Method

4.1 The PVC compound is solvent-extracted in order to separate the plasticizer from the compound. The resin is dissolved from the remaining compound and the inorganic fillers and stabilizers separated by centrifuging. By this technique, the compound is separated into (1) plasticizers, (2) resin, and (3) inorganic stabilizers and fillers. Each may be individually analyzed by an infrared technique to identify and measure the components.

5. Significance and Use

5.1 PVC compounds are used in a wide variety of products and hence they are formulated to provide a wide range of physical properties. The physical properties required in a compound depend upon the product in which it is used. These properties are largely determined by the type, quantity, and quality of the compounding ingredients. The analytical test method described below makes use of infrared spectrophotometry for the qualitative or quantitative determination, or both, of many of these ingredients in PVC compounds. This test method may be used for a variety of applications including process control, raw material acceptance, product evaluation,

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.

*A Summary of Changes section appears at the end of this standard

and determination of changes in composition resulting from environmental testing.

5.2 This test method is directly applicable only to those components listed in the appendix and to those components which are known to be similar in chemical composition and in solubility characteristics to the chemicals listed in the appendix.

6. Apparatus

6.1 *Initial Sample Preparation*—Use any of the following apparatus, depending on shape and size of sample, for reducing solid samples to small particle sizes:

6.1.1 *Pencil Sharpener* or grater and a cold box or container capable of maintaining at least the temperature of solid carbon dioxide.

6.1.2 *Grinding Wheel*, coarse.

6.1.3 *Microtome*.

6.1.4 *Grinding or Cutting Mills*, commercial, for example, a Wiley mill (for samples larger than 1 g).

6.2 *Soxhlet Extraction Apparatus*:

6.2.1 For 0.5 and 1.0-g samples, use an extraction apparatus with a 150-mL flask and a 27 by 100-mm thimble.

6.2.2 For 0.2-g samples, use an extraction apparatus with a 30-mL flask and a 10 by 50-mm thimble.

6.3 *Mold and Press for KBr Pellets*—A mold assembly capable of pelletizing a 12.7-mm (½-in.) minimum diameter pellet under vacuum and a press capable of exerting pressures of at least 140 MPa (20 000 psi) are required to press clear KBr pellets.

6.4 *Infrared Spectrophotometer*—The spectral region from 4000 to 650 cm^{-1} (2.5 to 15 μm) is used. Refer to Practice E275, with particular emphasis on Sections 5 and 14 relating to resolution and spectral slit width measurements. An ultimate resolving power (**1**)³ of at least 1.5 cm^{-1} at 850 cm^{-1} (0.02 μm at 12 μm) is satisfactory. The suitability of the instrument should be proven in the user's laboratory. Demountable cells, 1.0-mm liquid cells, and a KBr pellet holder are the accessories used.

6.5 *Infrared Spectrophotometer, Fourier Transform (FT-IR)*, capable of attaining a 4 wave number resolution.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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.

7.2 *Alumina*, absorption.

7.3 *Carbon Disulfide (CS₂)*.

7.4 *Ether*, anhydrous.

7.5 *Potassium Bromide*, (KBr) infrared quality.

7.6 *Tetrachloroethane*, technical.

7.7 *Tetrahydrofuran* (stabilized with 0.1 % hydroquinone).

8. Component Separations

8.1 *Initial Sample Preparation*—Any test method that will increase the surface area of a sample sufficiently to permit complete plasticizer extraction in a reasonable time is satisfactory. PVC compounds as received are usually in the form of powders, granules, slabs, or offshaped pieces. Powders may be used directly. Thin sheets, 0.02 to 0.05-mm thick, molded from individual granules may be used. Granules may be pressed into slabs. Slabs or appropriately shaped pieces may be treated by one of the following techniques:

8.1.1 Buffing on a coarse grinding wheel,

8.1.2 Cooling the sample with solid carbon dioxide and grinding the brittle sample in a clean pencil sharpener or on a grater or clean file, or

8.1.3 Shaving thin slices from the sample with a microtome.

8.2 *Plasticizer Extraction*—Weigh to ± 0.2 mg approximately 1 g of fine particle size sample into a 27 by 100-mm paper extraction thimble. Place the thimble in a jacketed Soxhlet apparatus fitted with a tared 150-mL flask, and extract with 120 mL of ethyl ether for 6 h (Note 2). Remove the tared 150-mL flask, containing the ethyl ether and the extracted plasticizer, from the jacketed Soxhlet apparatus and gently heat to boil off the ethyl ether. Place the flask in an evacuated desiccator for a minimum of 1 h to remove the last traces of ethyl ether. Weigh to ± 0.2 mg the flask containing the extracted plasticizers. Calculate the percentage of plasticizers in the PVC sample as follows:

$$\text{plasticizers, \%} = \frac{\text{weight of extracted plasticizers} \times 100}{\text{weight of PVC sample}}$$

8.2.1 Keep the plasticizers for infrared identification or determination (8.4).

NOTE 2—Organometallic or organic stabilizer, if present, may partially or wholly separate from either the plasticizer or resin components and should be considered when examining these compounds.

8.3 *Separation of Stabilizers and Fillers*—Empty the resin, stabilizers, and fillers remaining in the extraction thimble into a 50-mL beaker. Add 20 mL of tetrachloroethane and heat the sample gently until the resin has dissolved. Wash the contents of the beaker quantitatively into a tared 50-mL centrifuge tube with 20 mL of tetrahydrofuran (which has been previously passed through a 150 by 12.7-mm (6 by ½-in.) diameter alumina absorption column to remove hydroquinone), swirl to mix, and centrifuge for 30 min. Decant the resin solution and reserve for infrared analysis. Wash the residue remaining in the tared centrifuge tube with 20 mL of tetrahydrofuran and centrifuge again for 30 min. Decant the solution containing the

³ The boldface numbers in parentheses refer to the list of references at the end of this test method.

⁴ *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.