Designation: D 2124 – 99$^{\epsilon 1}$

Standard Test Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique[1]

This standard is issued under the fixed designation D 2124; 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.

ϵ^1 Note—Editorially removed obsolete item from the References section in April 2002.

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 parentheses 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 similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

E 131 Terminology Relating to Molecular Spectroscopy[2]
E 168 Practices for General Techniques of Infrared Quantitative Analysis[2]
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]
E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions related to the material on infrared spectroscopy, refer to Terminology E 131.

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

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

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.08).

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[2] *Annual Book of ASTM Standards*, Vol 03.06.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

method may be used for a variety of applications including process control, raw material acceptance, product evaluation, 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 E 275, with particular emphasis on Sections 5 and 14 relating to resolution and spectral slit width measurements. An ultimate resolving power (**1**)[4] 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.[5] 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* (CS_2).

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 remaining resin. Repeat the operation. Dry the tared centrifuge tube containing the stabilizer and filler in an oven at 110°C for

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

[5] *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.

1 h, cool, weigh, and calculate the percentage of inorganic stabilizer and filler as follows:

$$\text{inorganic stabilizer and filler, \%} = \frac{\text{weight of stabilizer and filler} \times 100}{\text{weight of PVC sample}}$$

8.3.1 Keep the stabilizer and filler for infrared analysis. Usually carbon black and color pigments are included in this portion.

8.4 *Resin*—Calculate the percentage of resin by difference (100 minus the total percent of plasticizers, stabilizers, and fillers).

9. Infrared Analysis of Extracted Plasticizers

9.1 The extracted plasticizers may be run on the infrared spectrophotometer as liquid films for identification or in CS_2 solution for quantitative determinations.

9.2 *Identification of Plasticizers*—Most plasticizers for PVC compounds are liquid at room temperature. A few secondary plasticizers may be solid but would be suspended or dissolved in primary plasticizers. A demountable cell with NaCl windows and a 0.025-mm spacer usually suffices to give a strong plasticizer spectrum (**1**). Scan the spectrum from 4000 cm^{-1} to 650 cm^{-1} (2.5 to 15 µm). By reference to a collection of plasticizer spectra the plasticizers in the sample may be identified (**2, 3, 4, 5, 6, 7, 8**). With experience, rough estimates of concentrations may be made to enable preparation of matching standards for quantitative analysis.

9.3 *Quantitative Analysis of Plasticizers*—The variety of plasticizers and their possible combinations in PVC compounds is extensive. It is impossible to specify a single procedure that determines quantitatively all plasticizers with equal precision and bias. The following procedure is useful for a number of plasticizers and their combinations, particularly if either dioctyl phthalate or tricresyl phosphate is the primary plasticizer. The user should decide whether the efficiency, precision, and bias of the procedure is satisfactory for a specific combination of plasticizers to be analyzed.

9.3.1 Weigh 60 ± 0.2 mg of extracted plasticizer (from 8.2) into a 25-mL Erlenmeyer flask equipped with a glass stopper; add 20.00 mL of CS_2 to dissolve the plasticizers. Take care to avoid loss of solvent by keeping the Erlenmeyer flask stoppered when possible. Run the resultant 3.00-mg/mL plasticizer solution on the infrared spectrophotometer (**9**) in a 1.0-mm liquid cell. Run a compensating 1.0-mm liquid cell or a variable path cell suitably adjusted, filled with CS_2 in the reference beam. After proper cleaning and drying of the sample cell, run an equivalent blank of CS_2 in the sample cell versus the reference cell.

9.3.2 A chart presentation on absorbance versus frequency (wavelength) paper of 20 cm/100 cm^{-1} for frequency and 18 cm for zero to 1.0 absorbance range is satisfactory. However, other chart presentations may be used. For dioctyl phthalate and tricresyl phosphate the spectra ranges from 1800 to 1650 cm^{-1} (5.4 to 6.1 µm) and from 1345 to 1090 cm^{-1} (7.4 to 9.2 µm) are useful. Dioctyl phthalate bands at 1725 cm^{-1} (5.80 µm), 1270 cm^{-1} (7.87 µm), and 1121 cm^{-1} (8.92 µm), and tricresyl phosphate band at 1191 cm^{-1} (8.4 µm) are satisfactory. The dioctyl phthalate band chosen will depend, in part, on secondary plasticizer interferences. Choice of bands for other plasticizers is left to the discretion of the user. At the analytical band frequency (wavelength) chosen, absorbances for the sample spectrum ($A_s + A_b$) and the blank spectrum (A_b) are measured. Net absorbance due to sample component A_s is $(A_s + A_b) - A_b$.

9.3.3 Prepare plasticizer standards by dissolving the pure plasticizers of interest in CS_2 to give a series of standard solutions covering the 3.0 to 0.5-mg/mL range for each plasticizer. Run these standard plasticizer solutions under conditions identical to those under which the samples are run to obtain the net absorbances of the components at a series of concentrations. Plot Beer's law curves of net absorbances versus concentrations in milligrams per millilitre for each component. All quantitative manipulations shall be in accordance with Practices E 168.

9.3.4 Use the net absorbance of a specific plasticizer in conjunction with the appropriate Beer's law curve to determine the concentration in milligrams per millilitre.

9.3.5 Calculate the percentage of plasticizer in the PVC compound as follows:

$$\text{specific plasticizer, \%} = (AB/3W) \times 100$$

where:
A = concentration of plasticizer, mg/mL,
B = total weight of extracted plasticizers, mg, and
W = weight of PVC sample, mg.

10. Direct Infrared Determination of Plasticizers

10.1 The use of this procedure usually presupposes that a complete formulation analysis is not required and that the plasticizers to be determined are known.

10.2 Weigh 0.25 g (±0.25 mg) of fine particle size sample into a 10 by 50-mm extraction thimble. Place the thimble in a micro Soxhlet extraction apparatus. Extract for 6 h with 20 mL of CS_2. Transfer the CS_2 containing the extracted plasticizers to a 25-mL volumetric flask, dilute to the mark with CS_2, and mix thoroughly. Run this solution on the infrared spectrophotometer. Instrumental conditions and techniques, preparation of standards, and Beer's law curves are the same as those specified in 9.2.

10.3 *Calculation*—Calculate the percentage of a specific plasticizer in the PVC compound as follows:

$$\text{specific plasticizer, \%} = (25A/W) \times 100$$

where:
A = concentration of plasticizer, mg/mL, (from curve), and
W = weight of PVC sample, mg.

11. Infrared Analysis of Stabilizers and Fillers

11.1 *Identification*—The stabilizers and fillers, as a dry powder after separation from the resin, may be identified by running on the infrared spectrophotometer as a Nujol mull or a KBr pellet. Prepare the Nujol mull by adding a few milligrams of powder to a drop of Nujol in a small mortar and mixing; run the resultant mull as a film between two NaCl plates held in a demountable cell mount. Prepare the KBr pellet by adding approximately 1 mg of powder to 600 mg of dry KBr powder and mixing 1 min in a vibrator mixer. Place the mixture in a 12.7-mm (½-in.) diameter mold assembly, hold under vacuum for 3 min, and press for 3 min at a minimum pressure of 140 MPa (20 000 psi) while still under vacuum. Higher pressures will produce more stable pellets. Place the resultant pellet in a holder and run it on the infrared spectrophotometer (**1**). By comparison to reference spectra (**2, 3, 8, 10, 11, 12, 13**) the stabilizer and filler components, in many cases, may be identified.

11.2 *Quantitative Analysis of Stabilizers and Fillers*—Analyze these components by the KBr pellet technique. Weigh 1 mg of stabilizer and filler powder and add to 600 mg of dry KBr powder. Prepare KBr pellets as described in 11.1, place in a holder in the sample beam, and run on an infrared spectrophotometer (**8**). A chart presentation of absorbance versus frequency (wavelength) paper of 20 cm/100 cm^{-1} and 18 cm for zero to 1.0 absorbance range is satisfactory. Other chart presentations may be used at the discretion of the user. Base line techniques, in accordance with Practices E 168, are used to determine absorbances of the bands of interest, and Beer's law curves of net absorbance versus percentage of component in total stabilizers and fillers are plotted. The net absorbances are those which would result if the stabilizers and fillers were exactly 1 mg in 600 mg of KBr powder. The percentage of component in the PVC sample may be calculated from the weight of stabilizers and fillers in the PVC sample previously determined. Standard samples for preparation of Beer's law curves are prepared by mixing the pure compounds of interest in appropriate amounts to give a set of matched standards. The following bands are usable in many cases: basic lead carbonate, 1410 cm^{-1} (7.09 µm); calcined clay, 1075 cm^{-1} (9.30 µm); calcium carbonate, 877 cm^{-1} (11.4 µm); antimony oxide, 741 cm^{-1} (13.5 µm); basic lead sulfate, 1130 cm^{-1}, (8.85 µm); and dibasic lead phthalate, 1535 cm^{-1} (6.51 µm).

12. Infrared Analysis of PVC Resin

12.1 *Identification of Resin*—The resin obtained during component separations is in a tetrachloroethane-tetrahydrofuran solution. Evaporate a few millilitres of the solution a few drops at a time on a microscope slide. Gentle heating will accelerate drying. When the resultant film is dry, peel it from the microscope slide. Dry the film in a vacuum desiccator or vacuum oven to reduce solvent spectral interferences. It is advisable to prepare a number of films from each sample in order to obtain one of suitable quality and thickness. Mount the film in the infrared spectrophotometer (**1**) and record its spectrum from 4000 cm^{-1} (2.5 µm) to 650 cm^{-1} (15 µm).

12.2 The PVC may be identified by its overall infrared spectrum (**2, 3, 14, 15, 16**). If the resin is a copolymer of vinyl chloride and vinyl acetate, a carbonyl band will be present at 1742 cm^{-1} (5.74 µm), and if the amount of acetate is greater than approximately 5 %, a band attributed to the acetate group is present at 1020 cm^{-1} (9.80 µm). Take care in the interpretation of carbonyl bands in the spectrum of the resin since these may also arise from the following:

12.2.1 Copolymers other than acetate (for example acrylate),

12.2.2 Incomplete extraction of certain polymeric ester plasticizers,

12.2.3 Oxidation of the resin, and

12.2.4 Esterification of the resin by certain compounding ingredients.

12.3 Usually the carbonyl bands due to residual polymeric plasticizers and to oxidation are at lower frequencies than those due to copolymers or to esterification of the resin.

13. Report

13.1 Report the following information:

13.1.1 Description of the material tested, that is, the name, color, manufacturer, and other pertinent data,

13.1.2 Description of sample preparation,

13.1.3 Description of spectrophotometer used,

13.1.4 Statement of sections of method used in analysis,

13.1.5 Statement of sections of method modified,

13.1.6 Statement of additional methods used in analysis,

13.1.7 Indication of possible interferences in analytical determinations, and

13.1.8 Indication of precision and bias.

14. Precision and Bias [6]

14.1 The precision and bias quoted are for PVC formulations comprised of components listed in the appendix. The precision and bias should also apply to PVC formulations that contain plasticizers, fillers, and stabilizers similar in both chemical composition and solubility characteristics to those listed in the appendix. The precision and bias may not be applicable in cases where the nature of the components is not known.

14.2 *Plasticizer Analysis*—The multilaboratory accuracy of the plasticizer analysis is given by a precision of ±0.75 % (3S %) as defined in Practice E 177, and a bias of −0.314 %. In relative percent the coefficient of variation is ±0.79 % (R1S %).

14.3 *Filler and Stabilizer Analysis*—The multilaboratory accuracy of the filler and stabilizer analysis is given by a precision of ±0.45 % (3S %) as defined by Practice E 177 and bias of −0.10 %. In relative percent the coefficient of variation is ±1.49 % (R1S %).

14.4 *Resin Analysis*—The multilaboratory accuracy of the resin analysis is given by a precision of 0.60 % (3S %) as defined by Practice E 177 and a bias of +0.38 %. In relative percent the coefficient of variation is ±0.51 % (R1S %).

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[6] Supporting data are available from ASTM Headquarters. Request RR: D20–22.

APPENDIX

(Nonmandatory Information)

X1. COMPONENTS COVERED BY THIS TEST METHOD

X1.1 Resin

X1.1.1 PVC.

X1.2 Plasticizers

X1.2.1 Di(2-ethyl hexyl) phthalate (DOP).
X1.2.2 Epoxidized soy bean oil.
X1.2.3 Tricresyl phosphate (TCP).

X1.3 Stabilizers

X1.3.1 Basic lead carbonate.
X1.3.2 Tribasic lead sulfate.
X1.3.3 Dibasic lead phthalate.

X1.4 Fillers

X1.4.1 Clay.
X1.4.2 Calcium carbonate.
X1.4.3 Antimony trioxide.

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SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 2124 – 99: Added ISO equivalency statement (Note 1) and 6.5.

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