Designation: D7083 – 16

Standard Practice for Determination of Monomeric Plasticizers in Poly (Vinyl Chloride) (PVC) by Gas Chromatography[1]

This standard is issued under the fixed designation D7083; 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 practice provides for the separation and identification of monomeric plasticizers in poly (vinyl chloride) (PVC) compounds by gas chromatography (GC).

1.2 The text of this practice references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in Tables and Figures) shall not be considered as requirements of this standard.

1.3 Test Method D2124 is an alternative infrared procedure.

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

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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D2124 Test Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique

D3465 Test Method for Purity of Monomeric Plasticizers by Gas Chromatography

D7823 Test Method for Determination of Low Level, Regulated Phthalates in Poly (Vinyl Chloride) Plastics by Thermal Desorption—Gas Chromatography/Mass Spectrometry

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

IEEE/ASTM SI-10 Practice for Use of the International System of Units (SI), the Modernized Metric System

3. Terminology

3.1 *Definitions:*

3.1.1 For definition of plastic terms used in this standard, see Terminologies D883 and D1600.

3.2 For units, symbols, and abbreviations used in this standard refer to Practices E594, E355, or IEEE/ASTM SI-10.

3.3 *Abbreviations:*

3.3.1 *DOA*—Bis (2-ethylhexyl) adipate

3.3.2 *DBP*—Dibutyl phthalate

3.3.3 *DOP*—Bis (2-ethylhexyl) phthalate

3.3.4 *FTIR*—Fourier Transform Infrared Spectroscopy

3.3.5 *GC*—Gas Chromatography

3.3.6 *GC/MS*—Gas Chromatography/Mass Spectrometry

3.3.7 *GPC*—Gel Permeation Chromatography

3.3.8 *HPLC*—High Performance Liquid Chromatography

3.3.9 *PVC*—Poly (Vinyl Chloride)

3.3.10 *TCP*—Tricresyl phosphate

3.3.11 *TOP*—Tris (2-ethylhexyl) phosphate

3.3.12 *TOTM*—Trioctyl trimellitate

4. Summary of Test Method

4.1 A PVC sample is extracted in accordance with Test Method D2124 and the extract residue is re-dissolved in a solvent. The resulting solution is then analyzed by GC to determine the identity of each plasticizer component. The plasticizer components are identified by retention time, or chromatographic fingerprint, or both.

5. Significance and Use

5.1 Separation and identification of plasticizer components in PVC is necessary to correlate performance properties with polymer composition. This test method provides a means of

[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.

Current edition approved May 1, 2016. Published May 2016. Originally approved in 2004. Last previous edition approved in 2010 as D7083 - 04(2010)ε1. DOI: 10.1520/D7083-16.

[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

determining monomeric plasticizers including adipates, azelates, benzoates, citrates, phthalates, sebacates, and trimellitates.

5.2 Other methods successfully used to analyze plasticizers are column chromatography, HPLC, GPC, FTIR, and GC/MS.

5.3 This method is not applicable to plasticizers with molecular weights over 700 g/mol including epoxidized soybean oil and polymeric plasticizers.

6. Interferences

6.1 Retention times for GC are dependent on several variables and it is quite possible to have two or more components with identical retention times. The GC operator shall take the necessary steps to insure that adequate separation of the plasticizer components is achieved.

6.2 A source of interference can be from solvent impurities. Run a solvent blank prior to analysis of an extract.

7. Apparatus

7.1 Gas chromatograph equipped with a flame ionization detector and capable of operating in the 250 to 350°C range.

7.2 A capillary or packed gas chromatographic column capable of operating in the 250 to 350°C range such as a 30-metre 100 % poly (dimethylsiloxane) phase fused silica open tubular capillary column; 0.32 mm ID with a 0.25 µm film thickness, or a stainless steel packed column; 1.83 m long with an outside diameter of 6.4 mm, and filled with Chromosorb W or WAW, 60 to 80 mesh, as the solid support and coated with a liquid phase as recommended in Practice D3465.

7.3 Integrator or data handling system, capable of measuring the net peak area.

7.4 Gas chromatographic syringe or autosampler, 0.1-10.0-µL capacity.

7.5 Analytical balance, capable of weighing to + 0.001 g.

7.6 Pressure regulators, for all required gas cylinders.

7.7 Flowmeter, or other means of measuring gas flow rates.

8. Reagents and Materials

8.1 Carrier gas Helium, chromatographic grade or Nitrogen, chromatographic grade.

8.2 Hydrogen, chromatographic grade

8.3 Air, chromatographic grade

8.4 Dichloromethane, or a solvent suitable for re-dissolving the extract from Test Method D2124, spectral quality or chromatographic grade. The same solvent is used for standard preparation and dissolution of the extract from Test Method D2124, subsection 8.2.

8.5 Standards of appropriate monomeric plasticizers for use in constructing an external calibration curve.

NOTE 2—Should the sample contain unknown plasticizers, it is recommended that GC/MS, FTIR or another suitable technique be used to identify the plasticizers.

9. Safety and Precautions

9.1 *Hydrogen is flammable*—ensure all leaks are eliminated.

10. Sample Preparation

10.1 Refer to the extraction procedure in Test Method D2124, subsection 8.2.

11. Preparation of the Gas Chromatograph

11.1 *Example Conditions:* The following conditions were used to obtain the example chromatograms shown in Fig. 1.

NOTE 3—Other columns, detectors, or chromatographic conditions, or a combination of the three, can be used to accomplish the same or a similar separation. If different plasticizers (than those in Fig. 1) are present in the sample extract, these conditions are allowed to be modified for adequate separation (see 14.2).

11.1.1 *Chromatographic Column:* 30-m by 0.32-mm fused silica open tubular capillary column with a 100 % poly (dimethylsiloxane) stationary phase, film thickness 0.25 mm.

11.1.2 *Carrier Gas:* Helium, flow rate 1.9 mL/min (40 cm/sec).

11.1.3 *Injection Port:* Split injection, 350°C, split ratio 50 + 1.

11.1.4 *Detector:* Flame Ionization, 350°C

11.1.5 *Temperature Program:* Initial oven temperature 200°C hold for one minute, ramp to 320°C at 10°C/min. Hold for three minutes.

11.1.6 *Sample Size:* 1.0-mL of the re-dissolved extract. It is possible to analyze the extract neat. When the sample is analyzed neat, the sample size shall be small enough to maintain adequate chromatographic efficiency (that is, do not overload the column).

12. Procedure

12.1 *Gas Chromatographic Separation*

12.1.1 Ensure that the gas chromatograph is set to provide resolution (R) of at least 1.5 for all plasticizers analyzed using this method. The resolution calculation is based on:

$$R = (T_{R.2} - T_{R.1})/0.5(W_1 - W_2) \quad (1)$$

where $T_{R.2}$ and $T_{R.1}$ are the retention times of the adjacent peaks and W_1 and W_2 are the peak widths at the base of the two peaks.

12.1.2 Re-dissolve the extract residue from Test Method D2124, subsection 8.2 in 5-mL dichloromethane. Inject 1.0 µL of this extract into the gas chromatograph. It is possible to inject the extract neat. When the sample is injected neat, the sample size shall be small enough to maintain adequate chromatographic efficiency (that is, do not overload the column).

12.1.3 Run the separation for the length of time necessary to elute all components.

12.1.4 Record the chromatogram and measure the peak area of the components of interest using a computer or an integrator.