



Designation: **D7083 – 04 (Reapproved 2010)<sup>ε1</sup> D7083 – 16**

## Standard Practice for Determination of Monomeric Plasticizers in Poly (Vinyl Chloride) (PVC) by Gas Chromatography<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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<sup>ε1</sup> NOTE—Reapproved with editorial changes in July 2010.

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### 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:*<sup>2</sup>

**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

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<sup>1</sup> 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 July 1, 2010/May 1, 2016. Published October 2010/May 2016. Originally approved in 2004. Last previous edition approved in 2004/2010 as **D7083 – 04/D7083 - 04(2010)<sup>ε1</sup>**. DOI: 10.1520/D7083-04R10E01.10.1520/D7083-16.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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

- 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 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 ~~should~~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 mL~~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.