Designation: D3465 – 21

Standard Guide for Purity of Monomeric Plasticizers by Gas Chromatography[1]

This standard is issued under the fixed designation D3465; 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 gas chromatographic guide covers a procedure for extending the range of purity determination of monomeric plasticizers beyond that now determined by other methods. Due to the need to volatilize the plasticizer, only monomeric plasticizers having definitive boiling points and a molecular weight less than 1000 Daltons, such as dioctyl phthalate, are applicable to this guide.

1.2 The values in SI units are to be regarded as standard.

1.3 The text of this guide references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) are not to be considered as requirements of this guide.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 9.

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

1.5 *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]

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Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique (Withdrawn 2020)[3]

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

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

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

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

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 *General*—Definitions are in accordance with Terminology D883 and Terminology D1600 unless otherwise indicated.

3.1.2 All gas chromatography terms and relationships used in this guide are consistent with, or refer to, Practice E355.

4. Summary of Guide

4.1 A test portion of the plasticizer to be analyzed is injected onto a gas chromatographic column and separated into its components, which are sensed by the detector and quantified by an electronic data acquisition system (computer). The purity is based on the percent total area response of the principal peak(s). All other components are impurities.

5. Significance and Use

5.1 Infrared techniques frequently cannot detect low-level materials. Gas chromatographic methods possess higher sensitivity and are used to extend limits of detection.

5.2 It is expected that this guide will be suitable for specifications, manufacturing control, and research and development. An area percent method of determining concentration of the components shall be used if the area percent of the plasticizer is 99.0 % or higher. However, if the area percent of

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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] The last approved version of this historical standard is referenced on www.astm.org.

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

the plasticizer is less than 99.0 % or if any question were to arise about the data, an internal standard shall be used.

5.3 Impurities potentially found in monomeric plasticizers include alcohols, dibasic acids, and monoesters.

6. Interferences

6.1 Gas chromatography (GC) retention times are dependent on several variables, and it is possible to have two or more components with identical retention times. The analyst shall take the necessary steps to ensure that adequate separation of the plasticizer components is achieved. It is possible that analysis by gas chromatography/mass spectrometry will identify the presence of overlapping components.

7. Apparatus

7.1 *Gas Chromatograph,* equipped with a flame ionization or thermal conductivity detector and capable of operating in the range from 250 to 350°C.

7.1.1 *Capillary or Packed Gas Chromatographic Column,* capable of operating in the range from 250 to 350°C that provides adequate separation and definition of components. Examples are as follows:

7.1.2 *Packed Columns,* stainless steel, 1.83 m (6 ft) long, with an outside diameter of 6.4 mm (¼ in.) and filled with Chromosorb W or WAW,[4] 60 to 80 mesh, as the solid support.

7.1.2.1 *Liquid Phase*—The liquid phase sensitivity changes the utility in detecting various impurities. Examples of detectability of alcohol impurities are illustrated as follows:

7.1.2.2 A coating of 5 to 15 % of SE-30 silicone gumstock results in reasonably symmetrical peaks and accurate quantitative measurements between 0.1 and 1 % alcoholic impurities.

7.1.2.3 A coating of 15 % Igepal CO-990[5] results in separations of similar types of esters such as diisooctyl phthalate and di (2-ethylhexyl) phthalate at the 1 % level.

7.1.2.4 A coating of 20 % Ozonite[6] is also effective for alcohols in the 0.1 to 1 % concentration level.

7.1.3 *Capillary Columns,* fused silica high-temperature capillary column with a 100 % poly(dimethylsiloxane) stationary phase; length: 15 to 30 m; inside diameter: 0.25 to 0.5 mm; film thickness: 0.1 to 0.25 µm.

7.2 *Electronic Data Acquisition System.*

7.3 *Gas Chromatographic Syringe or Autosampler,* 0.1 to 10.0 µL capacity.

7.4 *Pressure Regulators,* for all required gas cylinders.

7.5 *Flowmeter,* or other means of measuring gas flow rates.

[4] Trademark of Imerys Minerals California, Inc.
[5] Trademark of Rhodia Operations.
[6] Trademark of Alltech Associates.

8. Reagents and Materials

8.1 *Helium,* chromatographic grade.

8.2 *Hydrogen,* chromatographic grade.

8.3 *Nitrogen,* chromatographic grade.

8.4 *Purified Air.*

9. Hazards

9.1 Hydrogen is flammable. Ensure that all leaks are eliminated.

10. Preparation of the Gas Chromatograph

10.1 *Temperature Settings*—This guide requires injection port and detector temperatures of 260 to 350°C. For a plasticizer with a nominal boiling point of 410°C, the following conditions are suggested:

Injection port	260 to 350°C
Detector	300 to 350°C

10.2 Program the temperature of the column suitable to best separate the components (see 7.1 for columns). Typical settings are as follows:

10°C/min from 60 to 280°C and hold.
10°C/min from 100 to 300°C and hold for 10 min.

10.3 *Sample Size*—Select a sample size that provides an adequate chromatographic efficiency (that is, do not overload the column). A typical sample size is 1 µL.

10.4 *Carrier Gas*—Helium, flow rate 30 to 60 cm^3/min.

11. Procedure

11.1 The method of injecting the sample onto the column and analyzing for the components is given in detail in Practice E260, (specifically, Sections 10 and 13).

11.2 The method for analyzing the components shall be consistent with Fig. 1 of Practice E355.

12. Report

12.1 Report the following information:

12.1.1 Proportion of the total area related to the principal component(s) as the purity of the plasticizer, percent,

12.1.2 Retention times of impurities, relating them to corresponding retention times of known substances,

12.1.3 Proportion of total area related to the impurities in percent,

12.1.4 Column description,

12.1.5 Gas chromatograph conditions, and

12.1.6 Internal standard when used for calibration.

13. Keywords

13.1 adipate; benzoate; gas chromatography; monomeric; phthalate; plasticizers; poly(vinyl chloride); PVC; trimellitate