Designation: D 3421 – 75

Standard Recommended Practice for EXTRACTION AND DETERMINATION OF PLASTICIZER MIXTURES FROM VINYL CHLORIDE PLASTICS[1]

This Standard is issued under the fixed designation D 3421: 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.

1. Scope

1.1 This recommended practice covers the isolation and determination of the monomeric and polymeric constituents of plasticizer mixtures used in poly(vinyl chloride) compositions; it provides means for the analysis of compositions containing dioctyl phthalate with one or more homogeneous polyester plasticizers, and may be extended to analysis of plasticizer mixtures containing simple phthalate esters other than dioctyl phthalate.

NOTE 1—The values stated in SI units are to be regarded as the standard.

2. Applicable Documents

2.1 *ASTM Standards:*

D 494 Test for Acetone Extraction of Phenolic Molded or Laminated Products[2]

E 260 Recommended Practice for General Gas Chromatography Procedures[3]

3. Significance

3.1 Separation and identification of the components used in thermoplastics compounds is often required in efforts to relate performance properties to constitution, and in the design of fabrication control procedures.

3.2 The method obviates difficulties caused by incomplete reprecipitation of high polymeric material from compositions completely dissolved in solvents such as tetrahydrofuran.

4. Apparatus

4.1 For small samples (approximately 1 g), semimicro Soxhlet extractors[4] are adequate; these may be used with paper thimbles, or with glass shells.[5] The Wiley-Richardson apparatus (Method D 494) is useful and gives shortened extraction times due to full immersion of the sample holder in solvent vapors; this type of apparatus must be modified to eliminate contact between solvent vapors and metal parts. All-glass extractors having an outer jacket permitting solvent vapors to surround the barrel are available.[6]

5. Reagents

5.1 *Carbon Tetrachloride,* ACS, reagent grade.

5.2 *Chloroform,* ACS, reagent grade.

NOTE 2: **Caution**—Both carbon tetrachloride and chloroform are highly toxic and should be handled with extreme care, preferably within an efficient hood; inhalation of vapors is to be avoided.

5.3 *Methanol,* ACS, reagent grade.

6. Sample Preparation

6.1 Grind the sample in a Wiley polymer mill to 20-mesh particle size. If a Wiley mill is unavailable, 5 by 5-mm squares cut from sheeting of 0.6 to 2-mm thickness give satisfactory results.

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[1] This recommended practice is under the jurisdiction of ASTM Committee D-20 on Plastics, and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved June 30, 1975. Published August 1975.

[2] *Annual Book of ASTM Standards,* Part 35.

[3] *Annual Book of ASTM Standards,* Part 42.

[4] For example, Scientific Glass Apparatus Co. Catalog No. JR-5700A, Sargent S-31247A.

[5] Sargent S-31709, 31710.

[6] Scientific Glass Apparatus Catalog No. JM-5120.

6.2 When sufficient material is available, the preferred sample weight is approximately 5 g.

7. Procedure

7.1 Place 1 to 5 g of the prepared sample in a preweighed (to ± 0.0001 g) paper thimble or glass/ceramic extractor cup, and weigh the assembly to ± 0.0001 g. Place the assembly in the extractor barrel.

7.2 Attach a condenser to the extractor barrel and extract the sample 16 h with a 2+1 (by volume) mixture of carbon tetrachloride and methanol.

7.3 Cool the extract and filter through a fluted paper into a tared flask, rinsing the paper with small portions of the solvent mixture.

7.4 Evaporate the solvent to constant residual weight on a steam bath; weigh the residue to the nearest 0.0001 g.

7.5 Determine the dioctyl phthalate content by gas chromatography (see Recommended Practice E 260).

7.5.1 A general description of conditions appropriate for determination of the monomeric content when the monomeric consists of DOP follows.[7] Isothermal and programmed-temperature modes have been employed.

Column:	1.8 m by 3.18 mm (6 ft by 1/8 in.) 10 % SE-30 on Chromosorb W or Diatoport S
Temperatures:	265 to 280°C (isothermal; 265°C gave better resolution of peaks) 50 to 330°C programmed at 10°C/min.
Sample size:	1 μl
Carrier:	helium at 3.45 MPa (50 psi) gage; flow rate 60 cm³/min
Detector:	flame ionization detector[8]
Detector temperature:	320°C
Injection port temperature:	350°C
Internal standard:	dibutyl phthalate

7.5.2 Quantitatively transfer the residue (7.4) into a 100-ml volumetric flask using chloroform as a solvent. Weigh 1.5 g of dibutyl phthalate (99 % + purity) to the nearest 0.0001 g, and place in the flask. Dilute to volume with chloroform and mix thoroughly. Obtain a chromatogram of this solution using the parameters given in 7.5.1.

8. Calibration

8.1 *Calibration Factor for Dioctyl Phthalate*—Into a 100-ml volumetric flask containing approximately 50 ml of chloroform, weigh 1.5 g of dioctyl phthalate of known purity and 1.5 g of dibutyl phthalate of known purity. (Make all weighings to the nearest 0.0001 g.) Dilute to volume with chloroform and mix thoroughly. Obtain a chromatogram of this solution using the parameters given in 7.5.1.

$$\text{Calibration factor for dioctyl phthalate} = (A)\,(B)\,(C)/(D)\,(E)\,(G)$$

where:

A = weight of dioctyl phthalate, g,
B = purity of the dioctyl phthalate,
C = area under the dibutyl phthalate peak,
D = weight of dibutyl phthalate, g,
E = purity of dibutyl phthalate, and
G = area under dioctyl phthalate peak.

9. Calculation

9.1 Calculate the percent total plasticizing additives, TPA, as follows:

$$\text{TPA, \%} = [R/S(A - C)] \times 100$$

where:

R = weight of residue,
S = weight of sample,
A = weight of assembly, and
C = weight of cup.

9.2 Calculate the percent dioctyl phthalate as follows:

$$\text{Dioctyl phthalate, weight \%} = (X)\,(Y)\,(Z)\,(100)/(X')\,(Z')$$

where:

X = area under the dioctyl phthalate peak,
Y = weight of dibutyl phthalate, g,
Z = calibration factor for dioctyl phthalate,
X' = area under the dibutyl phthalate peak, and
Z' = weight of sample, 7.1.

9.3 Calculate the percent polymeric by difference.

NOTE 3—The total extract obtained by this procedure comprises, in addition to monomeric and polymeric plasticizers, stabilizer components such as aryl alkyl phosphites, and lubricants to the degree that they are present and soluble in the extraction solvent. Where peaks corresponding to these materials are observed in the gas chromatogram,

[7] Additional information regarding standard methods of sample injection and peak analysis as well as guideines for choice of (alternative) static phases is contained in ASTM D20.70.03/15.11 Joint Task Group Proposed Recommended Practice for Determining Purity of Monomeric Plasticizers by Gas Chromatography (in draft as of Jan. 1, 1971).

[8] Preference of D20.70.11 task group. D20.70.03/.15.11 method specifies use of thermal conductivity cell.