



Designation: D 3421 – 75

Standard Recommended Practice for EXTRACTION AND DETERMINATION OF PLASTICIZER MIXTURES FROM VINYL CHLORIDE PLASTICS¹

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 Products²

E 260 Recommended Practice for General Gas Chromatography Procedures³

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 extractors⁴ 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.

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.

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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.