Designation: F3414 – 20

Standard Test Method for Determining Ortho-Phthalate Concentration in Flooring Containing Polyvinyl Chloride[1]

This standard is issued under the fixed designation F3414; 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 test method covers determining the concentration of ortho-phthalate(s) in flooring containing polyvinyl chloride (PVC).

1.2 This test method does not purport to address or supersede any regulatory requirements.

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

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.*

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 Standard:*[2]

F141 Terminology Relating to Resilient Floor Coverings

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration standard, n*—stock standard containing the ortho-phthalate(s) of interest at a known concentration either purchased from a certified reference material provider or prepared in-house from materials of known purity and concentration and used to calibrate the measurement system.

3.1.2 *certified reference material, CRM, n*—solutions containing known amounts of ortho-phthalate that are used to evaluate the performance of the analytical instrument system.

3.1.2.1 *Discussion*—CRMs are obtained from a source external to the laboratory and are not made from the stock standard. For example, CRMs are available from the National Institute of Standards and Technology (NIST).

3.1.3 *laboratory reagent blank, LRB, n*—aliquot of solvents that is treated exactly as a sample including exposure to glassware, apparatus, and conditions used for a particular test but with no added sample.

3.1.3.1 *Discussion*—LRB data are used to assess contamination from the laboratory environment.

3.1.4 *ortho-phthalate, n*—type of plasticizer used to make solid resins such as polyvinyl chloride flexible

3.1.4.1 *Discussion*—The term ortho is a prefix used in organic chemistry to indicate the position of non-hydrogen substituents on an aromatic ring. Ortho describes a molecule with substituents at the one and two positions on an aromatic ring. See Fig. 1.

3.1.5 *resilient flooring, n*—organic floor surfacing material made in sheet or tile form or formed in place as a seamless material of which the wearing surface is non-textile.

3.1.5.1 *Discussion*—The resilient floor covering classification by common usage includes, but is not limited to, asphalt, cork, linoleum, rubber, vinyl, vinyl composition, and polymeric poured seamless floors. Resilient in this sense is used as a commonly accepted term but does not necessarily define a physical property. **F141**

3.1.6 *sample, n*—individual consumer product or a group of identical consumer products from a batch to be tested.

3.1.7 *solvent blank, n*—gas chromatograph (GC) or higher grade solvent selected to run in between samples within a sequence to prevent cross contamination within the instrument.

3.1.7.1 *Discussion*—Solvent blanks can be selected based on laboratory preference and instrument, for example, methanol or tetrahydrofuran (THF). Solvent blanks shall be replaced every 10 duplicates or 20 total samples.

3.1.8 *stock standard, n*—ortho-phthalate(s) purchased from commercial source at the highest available purity used to prepare calibration standards.

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[1] This test method is under the jurisdiction of ASTM Committee F06 on Resilient Floor Coverings and is the direct responsibility of Subcommittee F06.20 on Test Methods.

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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.

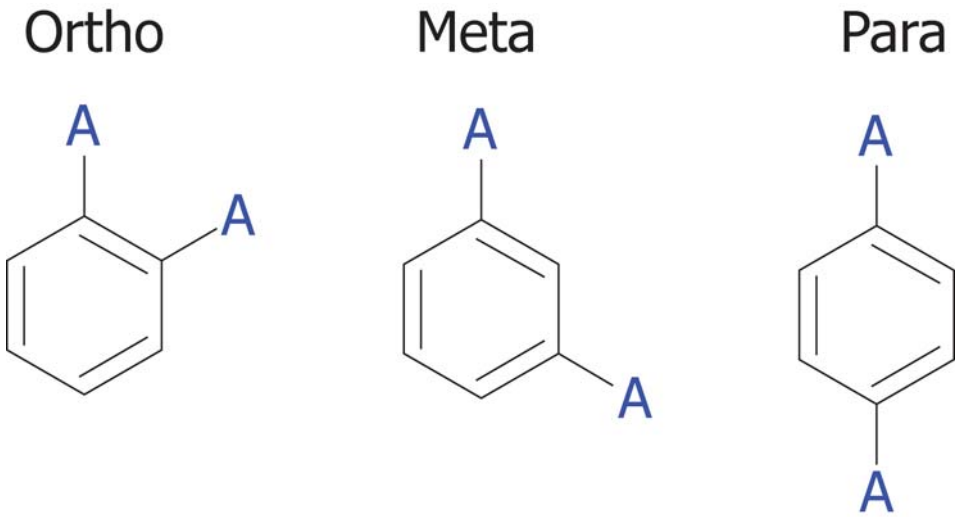

FIG. 1 Potential Positions of Non-Hydrogen Substituents on a Hydrocarbon Ring

3.1.8.1 *Discussion*—Stock standards shall be replaced before their expiration date.

4. Significance and Use

4.1 This test method can be used to determine ortho-phthalate content in resilient flooring.

5. Apparatus

5.1 *Sealable glass vials,* with polytetrafluoroethylene (PTFE) or silicone liner, size 20 mL or larger.

5.2 *Analytical balance,* capable of weighing to ±0.0001 g.

5.3 *Cryogenic mill*—(or suitable alternative to grind samples to powder).

5.4 *Ultrasonic bath.*

5.5 *PTFE filters,* 0.45 µm.

5.6 *Gas chromatograph-mass spectrometer (GC-MS,*) with an auto-sampler, split/splitless inlet, programmable GC oven, and capable of selective ion monitoring.

5.6.1 Related instrumentation, such as GC or liquid chromatography (LC) with advanced MS options, for example, ion trap or tandem mass spectrometry, can be used for qualitative assessment using the GC conditions in Table 1 and Fig. 2.

5.6.2 The ions in Table 2 shall be used for identification; the bolded ion is the target ion. Provided retention times may vary based upon column length and age. Based upon the purity of the standard, some phthalates are a mix of structural isomers and may appear over a retention time range as opposed to a singular peak. The variation in retention times will have no effect on the target ions present.

TABLE 1 Gas Chromatography Conditions

Column	DB-5MS; 30 m × 0.25 mm ID × 0.25 µm
Flow mode	1 mL/min, constant flow (He or H2 gas)
Inlet mode	20:1 split
Injection amount	1 µL[A]
Inlet temperature	290 °C
Solvent delay	4.5 min
Initial oven temperature	Hold time 150 °C, 1 min
Ramp 1	30 °C/min, 280 °C
Ramp 2	15 °C/min, 310 °C
Final hold time	3 min or longer
Auxiliary temperature	290 °C

[A] If using methanol, use 0.5 µL.

5.7 *GC vials,* size 2 mL.

5.8 *Volumetric glassware.*

5.9 *Volumetric pipettes.*

6. Reagents and Materials

6.1 Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 The materials used for sampling and analyses are:

6.2.1 Tetrahydrofuran (THF) (C_4H_8O, THF), CASRN 109-99-9, GC grade or higher.

6.2.2 Hexane (C_6H_{14}), CASRN 110-54-3, GC grade or higher.

6.2.3 Acetonitrile (C_2H_3N), CASRN 75-05-8, GC grade or higher

6.2.4 Methanol (CH_3OH), CASRN 67-56-1, GC grade or higher.

6.2.5 CRMs containing phthalates (such as NIST SRM 2860 or Korea Research Institute of Standards and Science CRM 113-03-006).

6.2.6 Benzyl benzoate ($C_{14}H_{12}O_2$, BB), CASRN 120-51-4, analytical grade or higher.

[3] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

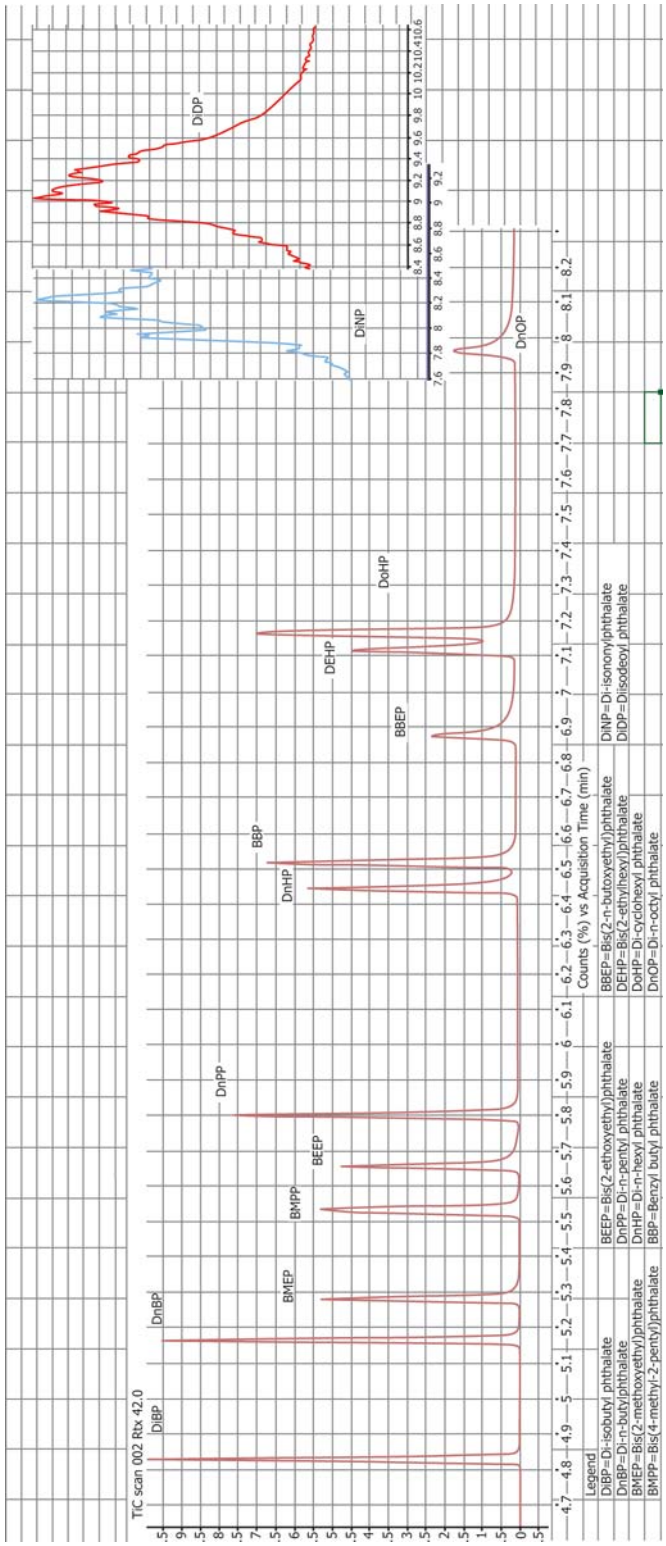

FIG. 2 TIC of the Internal Standard and all Phthalates of Interest Overlaid with an Extracted Ion Chromatogram Isolating DINP (Red, m/z 293)