



Designation: D4754 – 11

Standard Test Method for Two-Sided Liquid Extraction of Plastic Materials Using FDA Migration Cell¹

This standard is issued under the fixed designation D4754; 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 the use of the FDA migration cell in the extraction of components and permits quantitation of individual migrants from plastic materials by suitable extracting liquids, including liquid foods and food-stimulating solvents.

1.2 This test method provides a two-sided, liquid extraction test for plastic materials that can be formed into film, sheet, or disks.

1.3 This test method has been applied to a variety of migrant/polymer systems in contact with numerous foods and food simulants.² Though most of the migrants examined were radiolabeled, the use of the FDA cell has been validated for migration studies of unlabeled styrene from polystyrene.³

1.4 This test method has been shown to yield reproducible results under the conditions for migration tests requested by the FDA. However, if the data is to be submitted to the FDA, it is suggested that their guidelines be consulted.

1.5 Because it employs two-sided extraction, this test method may not be suitable for multi-layered plastics intended for single-sided food contact use.

1.6 The size of the FDA migration cell as described may preclude its use in determining total nonvolatile extractives in some cases.

NOTE 1—For more information, see Practice D1898, the AOAC Methods of Analysis on Flexible Barrier Materials Exposed for Extraction, and the Guidance for Industry: Preparation of Pre-market Submissions for Food Contact Substances: Chemistry Recommendations, December 2007.

1.7 Analytical procedures must be available to quantitate the migrant(s) generated by this test method.

¹ 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.

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² "A Study of Indirect Food Additive Migration," Arthur D. Little, Inc., FDA Contract No. 223-77-2360.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1141.

1.8 The values stated in SI units are to be regarded as the standard.

1.9 *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.* Specific hazards statements are given in Section 8.

NOTE 2—There is no known ISO equivalent to this test method.

2. Referenced Documents

2.1 *ASTM Standards*:⁴

D883 Terminology Relating to Plastics

D1898 Practice for Sampling of Plastics (Withdrawn 1998)⁵

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modernized Metric System

2.2 *Association of Official Analytical Chemists (AOAC) Methods of Analysis*:

Flexible Barrier Materials Exposed for Extraction⁶

2.3 *Federal Document*:

Guidance for Industry: Preparation of Pre-market Submissions for Food Contact Substances: Chemistry Recommendations, December 2007⁷

3. Terminology

3.1 *General*—The units, symbols, and abbreviations used in this test method are in accordance with Terminology D883 and Practice IEEE/ASTM SI 10.

⁴ 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.

⁵ The last approved version of this historical standard is referenced on www.astm.org.

⁶ Available through the Association of Official Analytical Chemists, 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877-2417 USA.

⁷ Available from Division of Food Contact Notifications, Office of Food Additive Safety, Center for Food Safety and Applied Nutrition, Food and Drug Administration, College Park, MD 20740, USA.

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

SUPPORT STAND

ASSEMBLED CELL

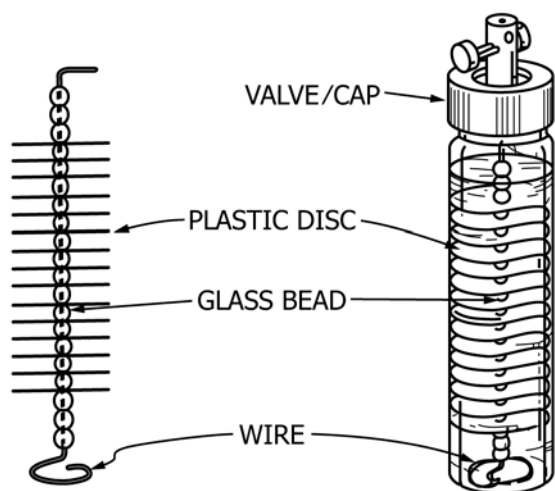


FIG. 1 FDA Migration Cell

4. Summary of Test Method

4.1 Specimens of plastic materials, formed in the shape of disks, are threaded onto a stainless steel wire with alternating glass bead spacers and placed in a glass vial. Solvent is added to the vial and the vial is capped and maintained at the desired extraction temperature. Aliquots of the liquid are removed at various times and the migrant(s) in the liquid determined by suitable analytical methods.

NOTE 3— Significant migration loss due to volatility may occur if migration is carried out at temperatures exceeding 50°C for periods greater than 2 weeks.

5. Significance and Use

5.1 Knowledge of migrants from plastic materials may serve many useful purposes, such as testing for compliance with food additive regulations. The procedure described in this test method is recommended as suitable for obtaining such data on many migrant(s)/plastic(s) combinations.

6. Apparatus

6.1 FDA Migration Cell⁸ (Fig. 1), consisting of:

6.1.1 Glass Vials, 23-mL,

6.1.2 Mininert® Slide Valve Caps,

6.1.3 Stainless Steel Wire (20-gage), and

6.1.4 Glass Bead (5-mm diameter), containing hole slightly larger than diameter of stainless steel wire.⁹ (Available at local hobby shops.)

NOTE 4—The apparatus, disk size, and number of disks are described for the 23-mL vial. Alternative vial sizes and corresponding test specimen sizes may be substituted. (The volume-to-surface area ideally should be between 155 and 0.31 mL/cm².) Note that validation tests have only been

⁸ The sole source of supply of the FDA Migration Cell components known to the committee at this time is Supelco, Inc., P.O. Box 628, 146 S. Water St., Bellefonte, PA 16823. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

⁹ Glass beads sold at hobby shops have been found satisfactory for this purpose.

conducted using the 23-mL vials.

NOTE 5—Recommend one-time use of mininert valve (that is, discarding it at completion of study).

6.2 Hot-Air Oven or Static Thermostatted Water Bath, with suitable safety provisions and capable of maintaining the desired extraction temperature within ±1°C.

6.3 Thermostatted Shaker Water Bath^{8,10}—Some migrant/ plastic/liquid combinations may involve significant partitioning and would benefit by having the cells shaken throughout the migration study.

6.4 Liquid Syringes, for removing liquid aliquots from the cells and transferring them to the analytical instrumentation.

6.5 Analytical Instrumentation, as required by the method chosen to determine the migrant(s).

7. Reagents and Materials

7.1 Purity of Reagents—All solvents shall be HPLC or chromatographic grade and shown to be free of interferences in the detection region of the migrant(s).

8. Hazards

8.1 The usual safety precautions for handling flammable solvents are recommended when such solvents are used for extraction.

9. Sampling

9.1 Sample the plastic in accordance with Practice D1898.

9.2 Select representative samples of the plastic to be tested from available stock on hand. Film, pellets, powders, sheet, and, in some cases, actual end-use articles are suitable. Protect the samples from exposure to liquids or contamination by migration from contact with other materials.

NOTE 6—See RR:D20-1141 for details regarding sample test specimens.

10. Test Specimen

10.1 Test specimens in the form of round disks (11 by 1 mm) are prepared from the plastic to be tested. Disks can be stamped out of sheets of actual end-use articles of non-brittle plastic by means of the appropriate sized cork borer. Alternatively, disks can be formed by using a heated press and an appropriate shim or mold containing holes the size of the disk. Holes can be put in the center of the disk by means of a drill or a heated wire.

NOTE 7—Whenever possible, plastic from actual end-use articles should be tested.

NOTE 8—When actual end-use articles are tested, the cut edges of the disks may have a different structure than the surfaces, and henceforth the migration rates may be altered. Because the area of the surfaces is much greater than that of the cut edges, the effect of the edges would be limited. If a significant edge effect is suspected, however, tests can be run comparing disks formed by using a heated press with disks cut from a sheet formed under similar conditions.

¹⁰ The sole source of supply of the thermostatted shaker water bath known to the committee at this time is Precision Scientific, 3737 W. Cortland St., Chicago, IL 60647.