Designation: D 4754 – 98 (Reapproved 2003)

Standard Test Method for Two-Sided Liquid Extraction of Plastic Materials Using FDA Migration Cell[1]

This standard is issued under the fixed designation D 4754; 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 sytrene 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 D 1898, the AOAC Methods of Analysis on Flexible Barrier Materials Exposed for Extraction, and the 1995 Recommendations for Chemistry Data for Indirect Food Additive Petitions.

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

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 similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[4]

D 1898 Practice for Sampling of Plastics[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modernized Metric System[6]

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

Flexible Barrier Materials Exposed for Extraction[7]

2.3 *Federal Document:*

1995 Recommendations for Chemistry Data for Indirect Food Additive Petitions[8]

3. Terminology

3.1 *General*—The units, symbols, and abbreviations used in this test method are in accordance with Terminology D 883 and Practice E 380.

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

[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.

[4] *Annual Book of ASTM Standards*, Vol 08.01.

[5] Discontinued. See 1997 *Annual Book of ASTM Standards*, Vol 08.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available through the Association of Official Analytical Chemists, Washington, DC.

[8] Available from Chemistry Review Branch, Office of Premarket Approval, Center for Food Safety and Applied Nutrition, Food and Drug Administration, Washington, DC 20204.

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

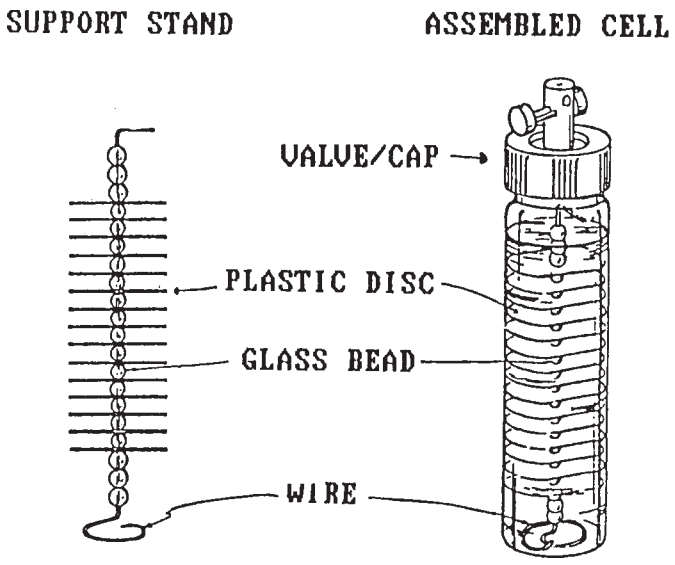

FIG. 1 FDA Migration Cell

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 Cell*[9] (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.[10] (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/cm^2.) Note that validation tests have only been 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*[9,11]—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 D 1898.

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.

11. Preparation of Apparatus

11.1 Alternately thread glass beads and 14 plastic disks onto the stainless steel wire (see Fig. 1). Prepare at least 4 sets for each liquid extractant used. Place resulting stacks of disks into 23 mL glass vials. Add 22 mL of extraction liquid and screw Mininert® caps tightly onto the vials.

[9] 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 committee [1], which you may attend.

[10] Glass beads sold at hobby shops have been found satisfactory for this purpose.

[11] 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.

11.2 Use the above prepared vials to determine the total amount of migrant(s).

11.3 To calculate migration rates, the samples should be washed to remove any surface bloom of the migrant(s). Maintain the above prepared vials at the extraction temperature for 2 h. Discard the liquid in the vials and replenish with fresh extracting liquid.

NOTE 9—Depending upon the conditions under which the test specimens were prepared, removal of any migrant surface bloom might not be warranted. Under these conditions, simply omit the wash step for removing migrant surface bloom.

12. Procedure

12.1 Place properly prepared vials in thermostatted oven or bath.

12.2 To prepare standards for quantitating the migration, place extraction solvent(s) and known quantities of the migrant(s) to be studied in a vial containing the support stand with glass bead spacers. Place these standard vials in the same thermostatted oven or bath.

12.3 To prepare the blank, place only the extraction solvent(s) in a vial containing the support stand with glass bead spacers. Place these blank vials in the same thermostatted oven or bath.

12.4 At pre-selected times, typically 5 to 8 samplings over a 2-week period, withdraw µL aliquots from each sample, standard, and blank vial and analyze using selected methodology.

NOTE 10—At each sampling, check to see if cell caps are tight.

NOTE 11—Volume withdraw is dictated by analytical procedure being utilized. If aliquots of more than 1 % by volume are removed during samplings, separate vials should be used for each test period.

12.5 Use response of standards on each day of analysis to quantitate the migrant(s).

13. Calculation

13.1 The test results can be calculated in a number of ways depending upon the application of the data.

13.2 One simple way to express the test results is to calculate the concentration of the migrant(s) in the liquid at a given time in some unit such as parts per million (ppm) or parts per billion (ppb).

13.3 A second way to express the test results is in milligrams of migrant(s) per square metre of sample exposed, *E*, as follows:

$$E=(W-B)/[(2\pi_R{}^2+CT)N]$$

where:

W = total weight of migrant(s) in the liquid, mg,
B = weight of migrant(s) in the blank, mg,
R = radius of the disk, m,
C = circumference of disk, m,
T = thickness of disk, m, and
N = number of disks per cell.

13.4 A third and more rigorous way to express the test results is to calculate diffusion and partition coefficients for the plastic/migrant(s)/liquid system used. These calculations, however, are beyond the scope of this test method. A brief description is contained in Appendix X1.

TABLE 1 Precision for Migration of Residual Styrene from Polystyrene

Time, h	Values in ppm				
	Avg	Sr^A	$S_R{}^B$	r^C	R^D
4	0.222	0.022	0.11	0.062	0.31
24	0.979	0.080	0.12	0.226	0.34
72	2.282	0.153	0.27	0.433	0.76
168	3.875	0.198	0.46	0.560	1.30
240	4.790	0.197	0.62	0.558	1.75
336	5.711	0.276	0.82	0.781	2.32

[A] S_r = *within-laboratory standard deviation of the average,*
[B] S_R = between-laboratories standard deviation of the average,
[C] r = 2.83 S_r, and
[D] R = 2.83 S_R.

14. Report

14.1 Report the test results, either as concentration of migrant(s) per unit volume of liquid, or concentration of migrant(s) per square metre of disk area.

15. Precision and Bias[3]

15.1 Table 1 is based on a round-robin conducted in 1984 in accordance with Practice E 691. Twelve laboratories reported results (ppm) for styrene migration from polystyrene disks into 50/50 ethanol/water at 49°C. The disks were prepared at one source. Five replicate sample cells were then setup by each laboratory which conducted the studies. One aliquot was taken from each cell at six times over 2 weeks. A test result was obtained from the analysis of each aliquot. Each laboratory reported 30 test results.

NOTE 12— The following explanations of I_r and I_R (see 15.2) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round-robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 15.2 through 15.2.3 would then be valid for such data.

15.2 *Concept of r* and *R*—Since S_r and S_R have been calculated from data produced by 12 laboratories, and for test results that were averages from testing 5 sample cells:

15.2.1 *Repeatability, r* (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the *r* value for that material.

15.2.2 *Reproducibility, R* (Comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results should be judged not equivalent if they differ by more than the *R* value for that material.

15.2.3 Any judgement made in accordance with 15.2.1 and 15.2.2 would have an approximate 95 % (0.95) probability of being correct.

15.3 The integrity of this cell was evaluated by carrying standards along with the actual migration cells. Each of the 12 participating laboratories analyzed standards at four different concentrations, six times over the 2-week migration period. From the detector response for these four standards, an average

detector response per 1 ppm of styrene was calculated (that is, slope of linear least squares fit, forced through origin). The coefficient of variation for the six average detector responses for the 12 laboratories ranged from 0.6 to 9.5 % and averaged 4.9 %.

15.4 *Bias*—There are no recognized standards on which to base an estimate of bias for this test method.

16. Keywords

16.1 diffusion; food packaging; indirect additives; migration cell; migration testing; polymer extraction

APPENDIX

(Nonmandatory Information)

X1. STYRENE DIFFUSION COEFFICIENTS (D_p) IN POLYSTYRENE (49°C)

X1.1 Although slightly beyond the scope of this test method, a diffusion coefficient for component migration can be calculated from the data obtained. The only additional information needed is the component concentration in the polymer disks.

X1.1.1 Diffusion of a migrant in a polymeric material is governed by Fick's Law. In its simplest form, where no partitioning between the polymer and the solvent in contact with it occurs, this migration can be expressed by:

$$Mt = 2Cp(DpT/\pi)^{1/2}$$

where:

Mt = mg/cm^3 at time, *T*, s,
Cp = mg/cm^3 in polystyrene disks, and
Dp = diffusion coefficient in disks, cm^2/s.

X1.1.2 From the slope, m, of a linear, least squares fit of *Mt/Cp* versus $T^{1/2}$.

X1.1.3 *Dp* can be calculated as follows:

$$Dp - \pi(m/2)^2$$

TABLE X1.1 Diffusion Coefficients

Laboratory	Dp × 10^{-12}cm/s
1	1.9
2	1.6
3	1.7
4	1.5
5	1.9
6	1.7
7	1.4
8	1.8
10	3.3
11	2.0
16	1.9
19	2.5
	avg = 1.9 (1.8 if #10 omitted)
	CV = 26.6 % (16.3 % if #10 omitted)

X1.2 Sytrene migration at 49°C was calculated from the data reported by the twelve laboratories participating in this round-robin. The residual styrene concentration in the polystyrene disks was determined by HPLC analysis to be 2348 ppm (n = 4, CV = 3.6 %). The *Dp* values are given in Table X1.1.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 4754 – 98:

(*1*) Changed reference in Note 1 and Section 2.3.

(*2*) Added the reference to Terminology D 883 to Sections 2.1 and 3.

(*3*) Changed Footnote 7 reference.

(*4*) Replaced I_r and I_R with *r* and *R* in Section 15 and Table 1.

(*5*) Added migration testing, food packaging, and diffusion to Section 16.

(*6*) Changed DP to D_p in title of Appendix X1.