Designation: F34 – 13 (Reapproved 2018)

Standard Practice for Construction of Test Cell for Liquid Extraction of Flexible Barrier Materials[1]

This standard is issued under the fixed designation F34; 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 practice covers the construction of test cells which may be used for the extraction of components from flexible barrier materials by suitable extracting liquids, including foods and food simulating solvents.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3 *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.*

1.4 *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 Standards:*[2]

D1193 Specification for Reagent Water

D4754 Test Method for Two-Sided Liquid Extraction of Plastic Materials Using FDA Migration Cell

2.2 *AOAC International Methods of Analysis:*

Official Method 964.15 Extraction from Flexible Barrier Materials[3]

2.3 *Code of Federal Regulations, Title 21*[4]—The following regulations cite this standard:

21CFR 176.170 (d)(3)

21CFR 177.1330 (e)(4)

21CFR 177.1360 (b)

21CFR 177.1670 (b)

Appendix VI (b) Cells for Migration Testing, Guidance for Industry: Preparation of Premarket Submissions for Food Contact Substances: Chemistry Recommendations (2007)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *flexible—for purpose of this practice*, a term applying only to those flexible materials which can be inserted in the test cell without affecting their barrier properties.

4. Summary of Preactice

4.1 This test cell defines the surface area of a flexible barrier structure that may contribute extractable/leachable substances to a packaged article. Although stainless steel and other materials are cited in the body of the practice, the test cell may be constructed of any durable material that does not contribute to the extractable content. The test cell should be evaluated for leaks after manufacture. It should be washed of manufacturing or milling fluids that may contribute to the extraction result.

4.2 Both pure chemicals as well as food simulants may be used as extracting solvents. FDA regulations usually specify the extracting fluid of choice, but other solvents may be chosen based on known or expected effects on the exposed surface.

4.3 The extractable content is usually measured by gravimetric analysis after evaporation of the extracting fluid, but other types of analysis may be performed if considered to be appropriate. Some methodologies are cited in the FDA regulations.

[1] This practice is under the jurisdiction of ASTM Committee F02 on Primary Barrier Packaging and is the direct responsibility of Subcommittee F02.15 on Chemical/Safety Properties.

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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.

[3] This method is available through the Association of Official Analytical Chemists, International, 481 North Frederick Ave., Suite 500, Gaithersburg, MD 20877 or from the AOAC website at www.aoac.org.

[4] Federal Regulations may be purchased from the U.S. Government Printing Office Bookstore, 710 North Capitol Street, N.W., Washington, D. C. or may be downloaded from the internet. The website for the chemistry guidelines document may be found at:
http://www.fda.gov/Food/GuidanceComplianceRegulatoryInformation/GuidanceDocuments/FoodIngredientsandPackaging/ucm081818.htm

4.4 Unless otherwise specified, the test cell is designed to be used at room temperature and ambient environmental conditions.

5. Significance and Use

5.1 Knowledge of extractives from flexible barrier materials may serve many useful purposes. A test cell constructed as described in this practice may be used for obtaining such data. Another test cell has been found equivalent to the one described in this practice. See the appendix for the source of the alternate cell.

5.2 United States Federal Regulations 21CFR 176.170 (d)(3), 21CFR 177.1330 (e)(4), 21CFR 177.1360 (b), 21CFR 177.1670 (b), and 21CFR Appendix VI (b) cite this standard practice as the basis for determining the amount of extractables from the surface of a package or multilayer film or modified paper in contact with food. In some cases, it is the only practice defined for this purpose. No alternative detail is given in the regulations for conducting extractions.

5.3 Test Method D4754 is not an equivalent to this test method. It is for two-sided extraction of films having the same material on both of the exposed surfaces of the film.

6. Reagents and Materials

6.1 *Purity of Reagents*—ACS (American Chemical Society) reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] 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. Good analytical practice suggests that a blank determination using the extracting solvent and a cell with no sample be run in conjunction with each series of determinations 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 *Purity of Water*—Type IV water; Specification D1193.

6.3 *n-Heptane,* ACS reagent grade.

[5] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC.

7. Procedure

7.1 *Construction of Test Cell* (Fig. 1, Fig. 2)—Assemble as follows:

7.1.1 Two 8 by 11⅜ by ⅛-in. (203 by 289 by 3.2-mm) No. 316 stainless steel plates, degreased.

7.1.2 One ¼ by 1½-in. (6.4 by 38-mm) U-shaped virgin TFE-fluorocarbon gasket, grooved on both sides as shown.

7.1.3 Twelve ¼ by 1-in. (6.4 by 25.4-mm) stainless steel bolts with wing nuts.

7.1.4 One ¼ by 1 by 8-in. (6.4 by 25.4 by 203-mm) TFE-fluorocarbon gasket plug tapered to provide a tight fit.

7.1.5 Six ⅛ by ⅜ by 3-in. (6.4 by 9.5 by 76-mm) plates.

NOTE 1—The use of stainless steel washers on the outside of each plate (7.1.1) may prevent the cells from leaking.

7.2 *Pre-Use Conditioning of Test Cell:*

7.2.1 Wash stainless steel plates and TFE-fluorocarbon gaskets in aqueous surfactant solution. Rinse with reagent water and dry at 122°F (50°C). Wash with *n*-heptane and redistilled acetone.

7.2.2 For new TFE-fluorocarbon gaskets, immerse in *n*-heptane overnight. Rinse gaskets with fresh *n*-heptane. Dry gaskets at 122°F (50°C).

7.3 *Use of Test Cell:*

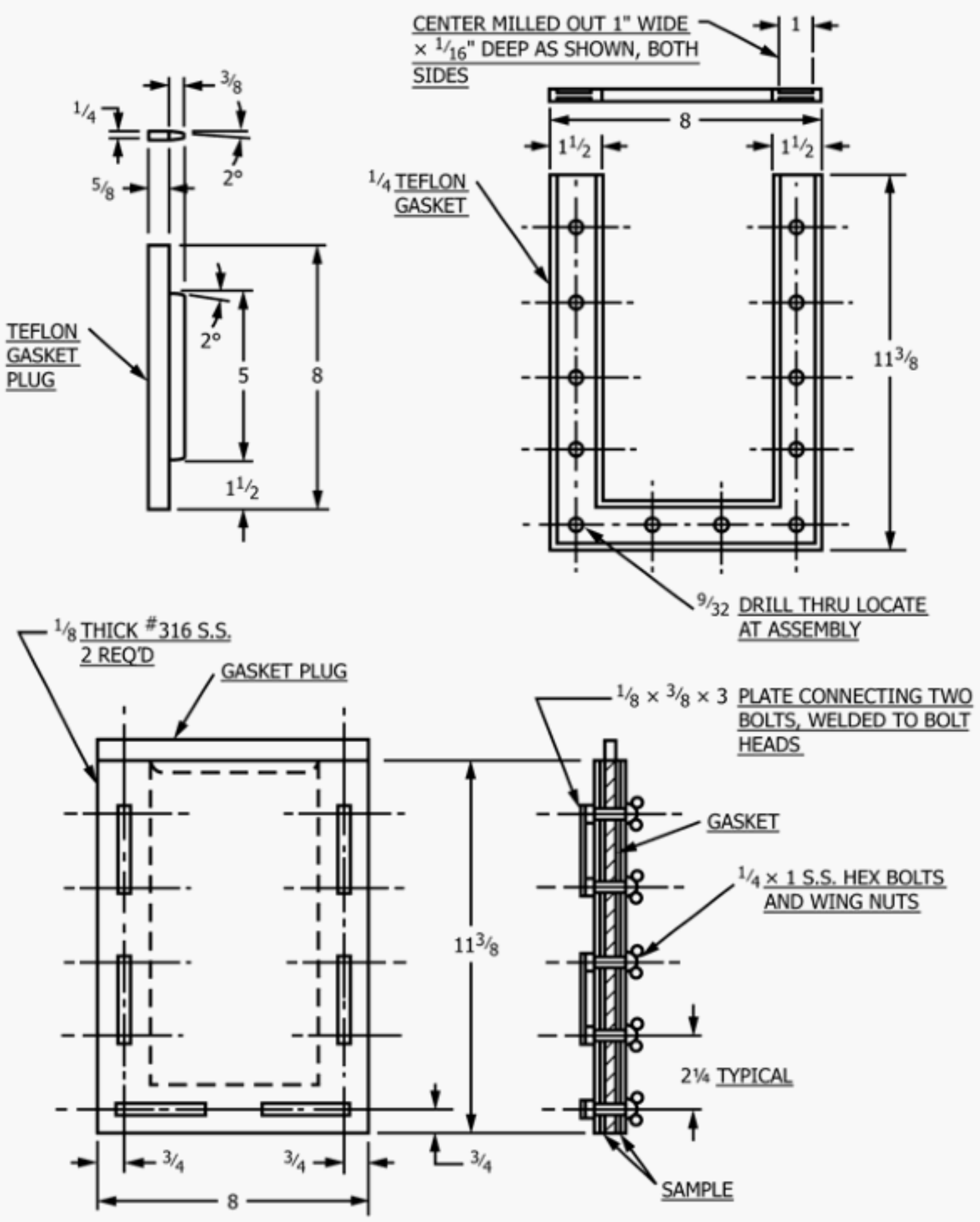
7.3.1 Place one stainless steel plate of the cell on a flat surface with bolts protruding up through the holes in the plate. Place a prepunched (Note 2) specimen (side to contact liquid *up*) on the plate with one edge of the specimen aligned with the bottom of the plate, two edges aligned with the sides of the plate, and the bolts passing through the prepunched holes. Place a TFE-fluorocarbon gasket on the specimen with the outer edges of the gasket aligned with the cell bottom and sides. If desired, place a second prepunched specimen (side to contact liquid *down*) on top of the gasket. If only one specimen sheet is used for the test, place an inert barrier sheet such as TFE-fluorocarbon or clean foil in place of the second specimen. Place a second stainless steel plate on top of the assembly. Place wing nuts on the bolts and tighten.

NOTE 2—Use the cell plate as a template and any suitable sharp instrument to prepunch the specimen through the bolt holes in the plate.

7.3.2 Preheat the assembly (including the TFE-fluorocarbon gasket plug) to the test temperature and retighten the nuts so that the assembly is liquid-tight. The assembly is ready to use.

8. Keywords

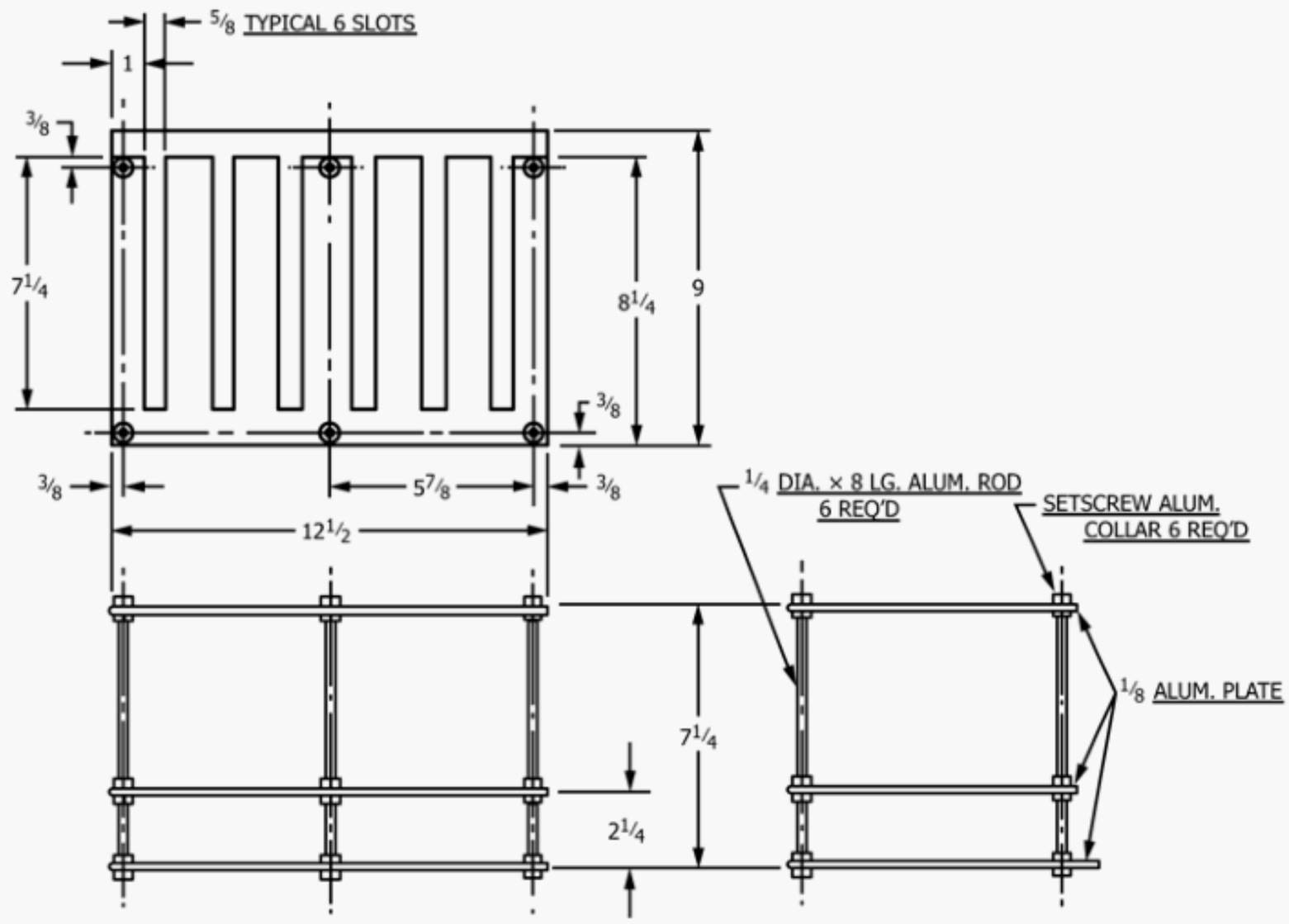
8.1 extractables; flexible barrier materials; food simulating solvents; test cells



SI Equivalents

in.	mm	in.	mm	in.	mm
1/16	1.6	5/8	15.9	2¼	57
1/8	3.2	3/4	19	3	76
1/4	6.4	1	25.4	8	203
9/32	7	1½	38	11⅜	289
3/8	9.5				

FIG. 1 Test Cell Assembly



SI Equivalents

in.	mm	in.	mm	in.	mm
⅛	3.2	1	25.4	7¼	184
¼	6.4	2¼	57	8¼	210
⅜	9.5	5⅞	149	12½	317
⅝	15.9				

FIG. 2 Rack for Holding Test Cells

APPENDIX

(Nonmandatory Information)

X1. AN ALTERNATE TEST CELL FOR LIQUID EXTRACTIONS OF FLEXIBLE BARRIER MATERIALS

X1.1 An alternate test cell has been found to be useful for liquid extractions of flexible barrier materials.[6]

X1.2 The test cell requires less solvent than the original cell described in this practice, and maintains an adequate test surface area for extractions. Food and Drug Administration (FDA) laboratories have successfully used this cell to perform extraction studies of PETE films with both water and Miglyol 812[7] (a fractionated coconut oil) over a temperature range from 60 to 120°C. [8,9]

X1.3 Reference is made to this cell in the Guidance for Industry: Preparation of Premarket Submissions for Food Contact Substances: Chemistry Recommendations (2007).[4]

[6] The test cell is a compression migration cell originally designed at Dow Chemical Company. There is no manufacturer of these cell systems known to the committee. If you are aware of 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.

[7] The sole source of supply of Miglyol 812 known to the committee at this time is Sasol Germany, GmbH, Arthur-Imhausen-Str 92, 58453 Witten, Germany. Miglyol 812 may be purchased in North America from Peter Cremer, 3117 Southside Ave., Cincinnati, OH. Local suppliers may also be found through an internet search. 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.

[8] Begley, Timothy H., and Hollifield, Henry C., "Food Packaging Made from Recycled Polymers: Functional Barrier Considerations," Chapter 36, *Plastics, Rubber, and Paper Recycling: A Pragmatic Approach.* ACS Symposium Series 609, 1995, pp. 445–475.

[9] Piringer, O., Begley, T.H., et al.," Migration from Food Packaging Containing a Functional Barrier: Mathematical and Experimental Evaluation," *J. Agr. and Food Chem.*, Vol 46, No. 4, 1998, pp. 1532–1538.