Designation: F1349 – 08 (Reapproved 2014)

Standard Test Method for Nonvolatile Ultraviolet (UV) Absorbing Extractables from Microwave Susceptors[1]

This standard is issued under the fixed designation F1349; 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 determination of nonpolar and relatively polar ultraviolet (UV) absorbing components that may migrate from microwave susceptor packaging into food simulants, such as corn oil and Miglyol 812.

1.2 This test method has been collaboratively studied using bilaminate susceptors constructed of paperboard, adhesive, and a layer of polyethylene terephthalate polymer (PETE) susceptor. Adhesive and PETE related compounds were quantitated using this test method.

1.3 The values stated in SI units are to be regarded as 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 4.3.2.3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

F874 Test Method for Temperature Measurement and Profiling for Microwave Susceptors

F1317 Test Method for Calibration of Microwave Ovens

3. Apparatus and Reagents

3.1 *Microwave Oven,* 700 ± 35 W, calibrated. Refer to Test Method F1317.

3.2 *High-Pressure Liquid Chromatograph (HPLC),* consisting of:

3.2.1 *Pump,* capable of 1.5 mL/min with flow precision ±2 %.

3.2.2 *Injector,* loop-type, equipped with 20-µL loop.

3.2.3 *Guard Column,* C_8, 5 µm.

3.2.4 *Analytical Column,* C_8, 5 µm, 250 by 4.6 mm.

3.2.5 *Detector-UV Absorbance,* set for 254 nm. Adjust sensitivity to give a 70 to 100 % of full scale peak for the 5-ppm dimethylterephthalate DMT standard.

3.2.6 *Gradient Program,* 4 to 60 % Mobile Phase B in 8 min; 60 to 70 % B in 9 min; 70 to 100 % B in 7 min; 100 % B for 11 min; 100 to 4 % B in 5 min; 4 % B for minimum of 5 min. Where Mobile Phase A (*v/v*) is 85 + 15 + 0.25 % water:acetonitrile:acetic acid, and Mobile Phase B (*v/v*) is 15 + 85 % water:acetonitrile.

3.2.7 *Peak Area Integration System*—Initialize data acquisition or integration system, or both, from 5 to 35 min during the separation.

3.3 *Hexane,* LC/UV grade.

3.4 *Acetonitrile,* LC/UV grade.

3.5 *Corn Oil*—Obtain corn oil that is as pure and fresh as possible to minimize peaks in nonvolatiles extractables chromatogram. Alternatively, Miglyol 812 (a fractionated coconut oil) or synthetic fat simulant HB 307 can be used as a substitute for corn oil.

3.6 *Dimethylacetamide (DMAC),* LC/UV grade.

3.7 *Conical Bottom Test Tubes,* 50 mL, graduated.

3.8 *Bishydroxyethyleneterephthalate (BHET).*

3.9 *Diethylterephthalate (DET).*

3.10 *Dimethylterephthalate (DMT).*

3.11 *Fluoroptic Thermometry System.*

3.12 *Temperature Probes,* four, high temperature.

3.13 *Glass Beads,* 3 to 4 mm, clean thoroughly by rinsing with methylene chloride followed by soaking for 30 min in acetonitrile. Dry thoroughly before using.

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

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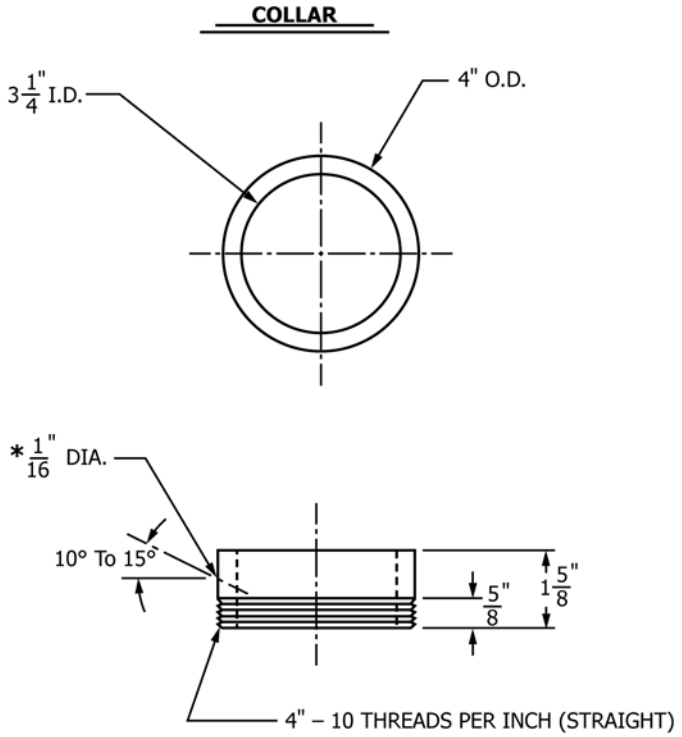
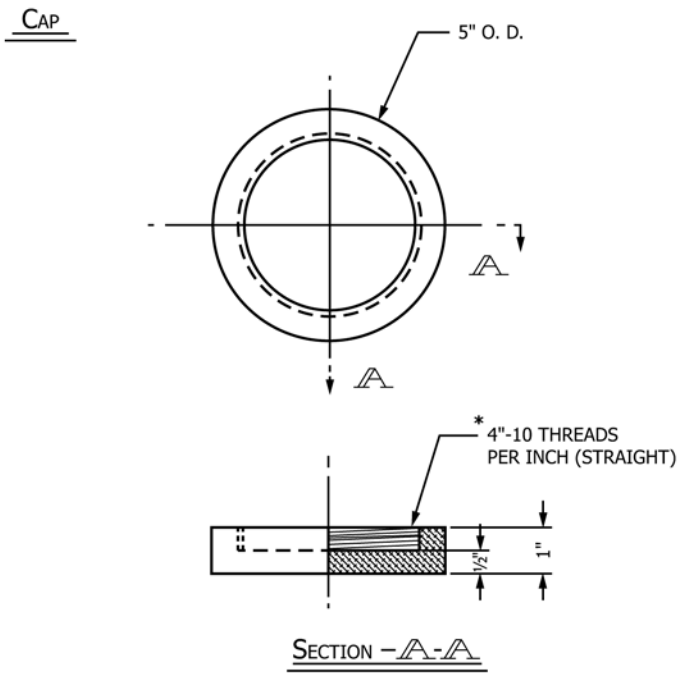
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



NOTE 1—The 1/16-in. (1.6-mm) diameter hole is for a Luxtron MIW temperature sensing probe. Number of holes and location may vary by application.

FIG. 1 Collar Section of Waldorf Polytetrafluoroethylene Microwave Nonvolatile Extraction Cell



NOTE 1—Relieve thread at bottom. Collar must seal to bottom of cap.

FIG. 2 Cap Section of Waldorf Polytetrafluorethylene Nonvolatile Extraction Cell

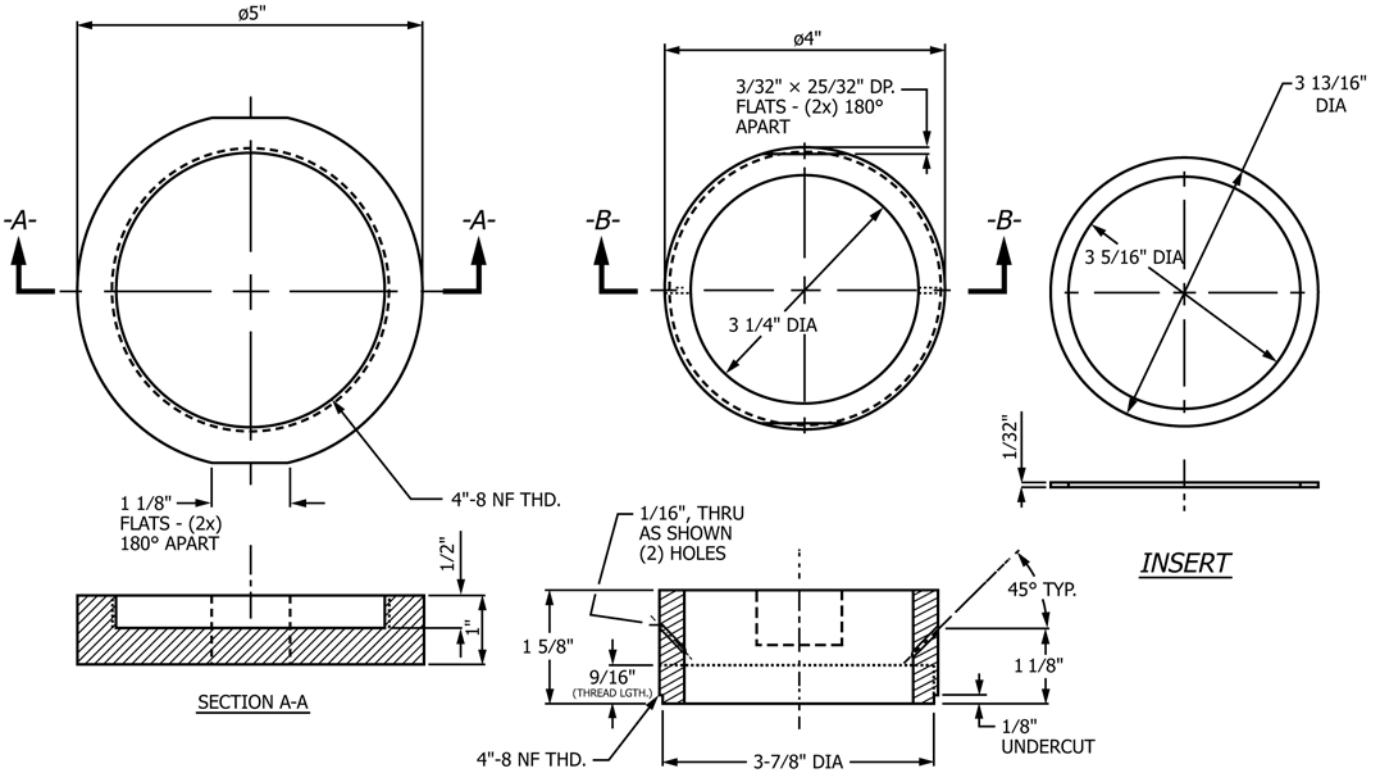


FIG. 3 Suggested Modifications to Waldorf Cell

3.14 *Recommended Microwave Nonvolatile Extraction Cell*—Waldorf Polytetrafluoroethylene cell.[3] (See Figs. 1-3). This cell must be constructed by a machine shop experienced in working with polytetrafluoroethylene (PTFE). After microwaving oil in the cell, the cell should be rinsed with methylene chloride to remove residual oil and prevent carry-over.

3.15 *Solvent Concentration Apparatus*—Kuderna-Danish evaporative concentrator, rotory evaporator; or Zymark TurboVap at a nitrogen pressure of 30 psi and a water bath temperature of 50°C.

4. Procedure

4.1 *Temperature Measurement:*

4.1.1 Refer to Test Method F874 to determine the time and water load specifications.

4.2 *Sample Preparation and Microwave Heating:*

NOTE 1—Always be sure the microwave oven is at ambient temperature before starting any temperature measurement or heating procedure to ensure consistency of output. Cooling of the microwave oven can be expedited by using ice in beakers or crystallization dishes or by using cold packs such as "blue ice."

4.2.1 Select a representative piece of the susceptor sample to be tested. If the susceptor is part of a package, trim excess material from around susceptor. Determine the area of the active susceptor material. The susceptor should be cut to fit into a Waldorf PTFE Cell with the screw seal ring firmly seated against the susceptor surface. Use of the Waldorf PTFE cell reduces the risk of spilling hot oil and in addition, gives a reproducible surface area (53.5 cm^2) for extraction. Alternatively, cut a 13 by 18-cm rectangular piece of the active susceptor material, form an extraction boat with sides 1.5 cm high (boat configuration = 1.5 by 10 by 15 cm, approximately 150 cm^2 of surface area). Staple the corners of the boat securely.

4.2.2 Add 53.2 g of Miglyol 812 of corn oil to the Waldorf PTFE Cell. Alternatively, add 22.5 g oil and 75 g of glass beads to the extraction boat.

4.2.3 Measure the mass of the room-temperature distilled water load as determined in 4.1.1 into a 600-mL beaker and add a boiling chip to this beaker.

4.2.4 Place Waldorf PTFE Cell or extraction boat containing the oil in the center of the microwave oven. Always position cell/extraction boat in the same position for subsequent runs.

4.2.5 Insert the temperature sensing probes through preformed holes in the walls in Waldorf PTFE Cells (shown in Fig. 1 and in the lower center sketch of Fig. 2), or in the case of the extraction boat, tape the probe to the wall of the oven such that the probe tip maintains contact with the extraction boat. Manipulate the probes until they make good firm contact with the active face of the susceptor material.

4.2.6 Microwave the cell or alternate extraction boat using the time specifications as determined in Test Method F874. Record the probe temperatures, preferably at 5-s intervals, but at intervals not to exceed 15 s.

[3] The sole source of supply of the apparatus known to the committee at this time is Read Plastics, 12331 Wilkins Ave., Rockville, MD 20852. 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.

4.3 *Quantitative Analysis:*

4.3.1 *Standard Curve:*

4.3.1.1 Prepare a standard mixture of 10 ppm (*w*/*v*) each of BHET, DMT, DET, and any other identified UV components (see appendix) of the susceptor in DMAC. Proceed to generate chromatograms using high pressure liquid chromatography in accordance with 3.2.5, 3.2.6, and 4.3.2.8. Retention times for BHET, DMT, and DET will be approximately 7.6, 16.6, and 21.5 min respectively.

4.3.1.2 Repeat with a standard of 5 ppm of DMT.

4.3.1.3 Repeat with a standard of 1 ppm of DMT.

4.3.1.4 Construct a plot of area response of DMT versus concentration.

4.3.1.5 For quantitation of PETE oligomers use the following response factor to construct an area response plot; $(mass/area)_{DMT}/(mass/area)_{Trimer} = 0.912$.

4.3.2 *Quantification of Extractables from Susceptor:*

4.3.2.1 Prepare and place the sample in the microwave oven in accordance with 4.2.1 – 4.2.6.

4.3.2.2 Microwave at full power using the time determined in 4.1.1.

4.3.2.3 Stir oil in boat or cell. **Warning—**Be extremely careful when handling the Waldorf PTFE cell or extraction boat. Use protective gloves. Severe burns can result from extremely hot oil.

4.3.2.4 Weigh 3 ± 0.03 g of stirred oil into a 50-mL beaker. Add 25 mL of hexane, stir, and transfer to a 125-mL separatory funnel.

4.3.2.5 Rinse the beaker with an additional 25-mL portion of hexane and add to the separatory funnel. Rinse the beaker with 25 mL of acetonitrile and add to the separatory funnel. Shake the separatory funnel and draw off the acetonitrile phase into a 50-mL conical test tube or into a Kuderna-Danish evaporative concentrator with a 10-mL receiver or other solvent concentration apparatus. Rinse the beaker with a second 25-mL portion of acetonitrile, add to the separatory funnel, shake, draw off acetonitrile, and add to the previous acetonitrile extract.

4.3.2.6 Concentrate the combined acetonitrile extracts to 0.4 to 0.5 mL in a 65°C water bath under a gentle stream of nitrogen or using a Turbo Vap or on a steam bath in a Kuderna-Danish evaporative concentrator with a 10-mL receiver and three-ball Snyder column.

4.3.2.7 Cool, take residue in test tube to 2 mL with DMAC.

4.3.2.8 Inject onto HPLC system, with or without filtering as desired, and separate using gradient conditions defined in apparatus in 3.2.6. Dilute sample if necessary if any extractant peaks are excessively large.

4.3.2.9 To determine a Miglyol 812 or corn oil or blank, place the proper amount of oil in a borosilicate petri dish. Place a fresh susceptor in the oven, place the petri dish on the susceptor and proceed through 4.3.2.1 – 4.3.2.8.

4.3.2.10 For reference, various oligomers of polyethylene terephthalate (PETE) will have the following retention times relative to DET:

cyclic trimer	= 2.8
tetramer	= 5.2
pentamer	= 6.8
hexamer	= 7.8
heptamer	= 8.8
octamer	= 9.8
nonamer	= 10.2

4.3.2.11 Subtract blank oil peak contributions from the sample chromatograms. Sum all the remaining peak areas in the sample chromatogram.

4.3.2.12 Using the area versus concentration plot for DMT, find the quantity of extractables present in the concentrated corn oil extract (QA ppm).

5. Calculation

5.1 Calculate susceptor extractables as follows:

$$(\mu g/in.^2) = 6.4516 \cdot QA \cdot ((TO/OS) \cdot V)/A \quad (1)$$

where:

QA	=	quantity of component in oil extract, ppm,
TO	=	total mass of oil in cell (53.5 g),
OS	=	mass of oil sampled from boat or cell (3.00 g),
V	=	final volume of concentrated extract, mL (2.0 mL),
A	=	surface area extracted, cm^2 (150 cm^2 for boat). For the Waldorf Cell area circle $A = 0.25 \cdot \pi \cdot d^2$ (53.52 cm^2), and
6.4516	=	$cm^2/in.^2$

6. Report

6.1 Report the following information:

6.1.1 A representative sample chromatogram.

6.1.2 The name and concentration in micrograms per square inch of the individual migrants found in the oil. Include the data for each sample analyzed and all replicate samples.

6.1.3 A representative sample time-temperature profile.

7. Precision and Bias

7.1 *Precision:*

7.1.1 Two different microwave susceptor samples were used in two separate collaborative studies. Both susceptors were laminates of PETE-adhesive-paperboard.

7.1.2 Six laboratories participated in the first study. All susceptor samples were prepared as extraction boats (1.5 by 10 by 15 cm) containing 22.5 g of corn oil and 75 g of glass beads. All susceptor samples were heated for a fixed time (5.0 min) using a fixed water load (250 g) in the microwave oven. Table 1 lists the means and standard deviations for the determination of PETE cyclic oligomers that migrated to corn oil. Table 2 lists the values for the coefficients of variation of this test

TABLE 1 Determination and Deviations for PETE Oligomers That Migrated to Corn Oil

	Mean, mg/in.^{2}	Standard Deviation, mg/in.^{2}
Cyclic trimer	0.148	0.023
Cyclic tetramer	0.021	0.006
Cyclic pentamer	0.012	0.004
Total oligomers	0.197	0.031

TABLE 2 Coefficients of Variation for PETE Oligomers

Cyclic trimer	Intralab COV	5.2 %
	Interlab COV	14.9 %
Cyclic tetramer	Intralab COV	8.6 %
	Interlab COV	28.2 %
Cyclic pentamer	Intralab COV	6.0 %
	Interlab COV	33.8 %
Total oligomers	Intralab COV	9.2 %
	Interlab COV	12.4 %

TABLE 3 Precision and Interlaboratory Reproducibility for Total PETE Oligomers and DEGDB Migrating to Miglyol 812

	Heating Time, min	RSD_r,%	RSD_R, %
PETE Oligomers	2	30	33
	5	16	16
DEGDB	2	53	61
	5	28	38

method for PETE oligomers based on the collaboration of laboratories (1989) reporting triplicate analyses.

7.1.3 In 1992, six laboratories participated in a second study. All susceptor samples were evaluated using the Waldorf PTFE cell. Susceptor samples were heated for 2 and 5 min using a fixed water load (100 g). After concentrating, the extracts were analyzed for total PETE oligomers and diethylene glycol dibenzoate (DEGDB) using HPLC with UV detection. All analyses were performed in duplicate. Table 3 lists the intralaboratory RSD_r and interlaboratory RSD_R for total PETE oligomers and DEGDB determined in this study.

7.1.4 The two studies had significant differences (different susceptor constructions, different water loads, and different microwave heating times) and cannot be considered replicate investigations. Of the data obtained from the two studies, only the total PETE oligomers determined after 5 min microwave heating can be compared and are in reasonable agreement.

7.2 *Bias*—Since no absolute method is available for comparison, no statement can be presented for this test method.

8. Keywords

8.1 extraction cell, Waldorf; extractables, microwave susceptors; extractables, nonvolatile by HPLC; extractables, nonvolatile UV absorbing; fat simulant, corn oil; fat simulant, Myglyol; fluoroptic thermometry; microwave susceptors; Myglyol; migration; nonvolatile extractables; nonvolatile extractables, quantitation of, by HPLC; PETE; susceptors, microwave, PETE

APPENDIX

(Nonmandatory Information)

X1. RECOMMENDED PRACTICE

X1.1 Initially the total UV absorbing components present in the polymer, adhesive, and paperboard should be determined. This may be accomplished by shredding two 8 by 10-in. (20 by 25.4-cm), or equivalent, surface area susceptor sheets and placing the shreds in a Soxhlet extractor. This shredded susceptor is then serially Soxhlet extracted with hexane, chloroform, and acetonitrile (or similar solvents that will not dissolve the polymer) for 3 h each. After each 3-h interval, the solvent is gently evaporated until a few millilitres of residual solvent remain, before the addition of the next solvent. After the third solvent extraction, the solvent is concentrated and prepared for HPLC analysis using UV detection. This extract should contain those UV absorbing materials that will potentially migrate to the corn oil under microwave heating conditions. Identification of unknown UV peaks can be performed by HPLC-mass spectrometry (MS) or by collecting the unknown HPLC peak and trying gas chromatography-MS.