Designation: F1500 − 98 (Reapproved 2014)

Standard Test Method for Quantitating Non-UV-Absorbing Nonvolatile Extractables from Microwave Susceptors Utilizing Solvents as Food Simulants[1]

This standard is issued under the fixed designation F1500; 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 is applicable to complete microwave susceptors.

1.2 This test method covers a procedure for quantitating non-UV-absorbing nonvolatile compounds which are extractable when the microwave susceptor is tested under simulated use conditions for a particular food product.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E260 Practice for Packed Column Gas Chromatography

E682 Practice for Liquid Chromatography Terms and Relationships

E685 Practice for Testing Fixed-Wavelength Photometric Detectors Used in Liquid Chromatography

F874 Test Method for Temperature Measurement and Profiling for Microwave Susceptors

F1317 Test Method for Calibration of Microwave Ovens

F1349 Test Method for Nonvolatile Ultraviolet (UV) Absorbing Extractables from Microwave Susceptors

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *microwave susceptor*—packaging materials that, when placed in a microwave field, are designed to interact with the field and provide substantial heat to the package contents.

3.1.2 *nonvolatile extractables*—those chemical species which released from microwave food packaging under simulated use conditions and are detected using an applicable nonvolatile extractables method.

4. Summary of Test Method

4.1 Nonvolatile extractables are determined by subjecting a sample of the susceptor material to microwave heating under simulated use conditions. The sample is washed with solvents covering a range of polarities. The solvent washes are combined and the solvents evaporated just to dryness. The residue is redissolved in a measured quantity of chloroform and the sample split for gravimetric or other analyses, such as HPLC or IR. For the gravimetric determination, a measured portion of the sample is filtered and evaporated and the residue weighed. For other analyses, the remainder of the sample is evaporated and may be reconstituted in dimethylacetamide prior to injection (see Test Method F1349 for quantitation of UV-absorbing nonvolatiles by HPLC), or treated appropriately prior to examination by other chromatographic or spectroscopic methods.

5. Significance and Use

5.1 This test method was developed to measure non-UV-absorbing nonvolatile extractables that may be present and migrate from a microwave susceptor material during use. It may be a useful procedure to assist in minimizing the amount of non-UV-absorbing nonvolatile extractables either through susceptor design or manufacturing processes.

5.2 Supplementation of this procedure with other analytical technologies such as high-pressure liquid chromatography, supercritical fluid chromatography, or infrared or other forms of spectroscopy may provide the analyst with additional information regarding the identification of the components of the non-UV-absorbing nonvolatile extractables in the susceptor.

[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.

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6. Apparatus and Reagents

6.1 *Microwave Oven,* 700 ± 35 W, no turntable, calibrated in accordance with Test Method F1317.

6.2 *Extraction Cell,* Waldorf, described in Test Method F1349.

NOTE 1—If the cell is not equipped with a PTFE gasket, cut a gasket ring to match the size of the sleeve from a 1⁄16 -in. PTFE sheet. Use of the gasket between the sleeve and the sample reduces damage to the sample.

6.3 *Microwave Temperature Measurement System.*

6.4 *Temperature Probe,* high temperature.

6.5 *Beaker,* 400-mL borosilicate glass.

6.6 *Hexane,* analytical reagent grade or better.

6.7 *Acetonitrile,* analytical reagent grade or better.

6.8 *Methylene Chloride,* analytical reagent grade or better.

6.9 *Chloroform,* analytical reagent grade or better.

6.10 *Dimethylacetamide,* HPLC grade or better.

6.11 *Methanol,* analytical reagent grade or better, dried over anhydrous sodium sulfate.

6.12 *Distilled Water.*

6.13 *Nitrogen,* grade suitable for solvent evaporation purposes.

6.14 *Rotary Evaporator,* or equivalent.

6.15 *Weighing Boat,* aluminum, formed by shaping aluminum foil into a round boat approximately 1.5 cm in diameter.

6.16 *Filter,* 0.45 µm, compatible with chloroform.

6.17 *Round-Bottom Flask,* 250 mL, with neck to fit rotary evaporator.

6.18 *Vial,* 20 mL.

6.19 *Heat Lamp,* 125 W, or equivalent.

6.20 *Boiling Stones.*

6.21 *Watchglass,* 8.5 or 9.0-cm diameter.

7. Sampling

7.1 The sample of microwave susceptor selected for extraction should be representative of the entire susceptor.

7.2 The sample should be undamaged, that is, lamination intact, uncreased (unless this is the normal configuration) and unaltered.

7.3 Carefully cut a circular portion of the susceptor large enough to fit the Waldorf cell with the top threaded sleeve removed. Be sure the sample is cut large enough to fill the entire bottom of the cell. Carefully trim away any frayed edges before testing.

7.4 Preclean the susceptor to remove dust and fibers by blowing a stream of nitrogen over the surface for a few seconds, or by gently brushing the surface with a camel hair brush.

8. Procedure

8.1 Calibrate the microwave oven in accordance with Test Method F1317 to ensure that it is 700 ± 35 W.

8.2 Determine the sample test conditions by using the method for temperature profiling of microwave susceptors in use in accordance with Test Method F874.

8.3 Place the precut susceptor sample into the bottom section of the Waldorf cell. Carefully place the polytetrafluoroethylene polymer (PTFE) gasket on top of the susceptor to prevent tearing when the cell sleeve is threaded in. Thread the top sleeve of the cell into the bottom section of the cell, trapping the susceptor sample securely between the gasket and the bottom of the cell.

8.4 Carefully insert a temperature probe (6.4) through the small temperature probe port opening of the cell and ensure that it maintains good contact with the susceptor surface. Insert a second probe onto a different area of the susceptor in the same way.

8.5 Place 50 mL of distilled water and a boiling chip into a 400-mL beaker and place the beaker in the center rear of the oven. Place a watchglass over the opening of the Waldorf cell.

8.6 Place the Waldorf cell in the center of the microwave oven, and microwave the sample on high power for the time determined during the temperature profiling procedure.

8.7 Compare the temperature profiles obtained in 8.6 with those obtained from the susceptor in contact with product. If the two profiles are in reasonable agreement, proceed to 8.8, otherwise repeat 8.3 through 8.6, using more or less water in the beaker to adjust the profile appropriately.

8.8 Without removing the sample, watchglass, or fiber optic probes from the cell, allow the sample to cool for 5 min.

8.9 Remove the temperature probe(s) from the cell. Rinse the bottom of the watchglass covering the Waldorf cell with 20 mL of hexane, pouring the solvent into the cell. Swirl the solvent in the cell for 10 s, then pour it into a 250-mL roundbottom flask. Repeat using a second 20-mL aliquot of hexane.

8.10 Repeat 8.9 using two 20-mL aliquots of methylene chloride.

8.11 Repeat 8.9 using two 20-mL aliquots of acetonitrile.

8.12 Repeat 8.9 using two 20-mL aliquots of methanol.

8.13 Using a rotary evaporator with a water bath temperature of 50°C, reduce the volume of the combined solvents in the round-bottom flask to approximately 10 mL. Transfer the remaining solvent to a 20-mL vial. Rinse the roundbottom flask with two 1 mL portions of acetonitrile and combine with the contents of the vial.

8.14 Apply a gentle stream of nitrogen to the solvent in the vial. Apply gentle heat as necessary to expedite evaporation. Evaporate just to dryness, avoiding any heating after all the solvent is evaporated.

8.15 Pipet 10 mL of chloroform into the vial. Swirl with gentle heating to dissolve the residue in the vial.

8.16 Dry a clean aluminum weigh boat by placing under a heat lamp for 5 min. Allow to cool and weigh, recording this weight as "tare." Filter 8 mL of the chloroform solution

through the 0.45-µm filter into the tared weigh boat and rinse the filter with a further 1 mL of chloroform.

8.17 Place the weigh boat under the heat lamp and evaporate the solvent to a constant weight (±0.5 mg). Record this weight in milligrams as "A."

8.18 Repeat 8.9 to 8.17 using solvents which have not been exposed to a susceptor. Record the final weight in milligrams after evaporation as" B."

8.19 Evaporate the remaining 2 mL of chloroform solution from 8.15 to dryness. At this point the residue may be redissolved in 2 mL of dimethylacetamide with gentle heating, filtered through a 0.45-µm filter and injected onto an HPLC system operated in accordance with Test Method F1349. (See Practices E260, E682, and E685 for further information regarding HPLC set-up and use.) Other sample preparation schemes can be developed for specific applications involving other chromatographic or spectroscopic techniques. The analyst should take the steps necessary to ensure that a representative sample of the residue is obtained, and that the analytes have not been degraded by the sample preparation scheme chosen.

9. Calculation

9.1 Calculate total nonvolatile extractable as follows:

$$\text{Total nonvolatile extractable } (\text{mg/in.}^2) = \frac{(A - tare - B) \times 10}{8.3 \times 8}$$

where:

A = weight from 8.17,
B = weight from 8.18,
$tare$ = weight from 8.16,
8.3 = square inches of susceptor exposed in Waldorf cell,
8 = volume of solvent evaporated, and
10 = total volume of sample.

10. Precision and Bias

10.1 Six laboratories participated in a collaborative study of nonvolatiles recovered from a bilaminate PETE/adhesive/paperboard susceptor construction obtained from a single source. Duplicate analyses were performed at heating times of 2 and 5 min using water loads specific to the individual microwave ovens. Participants were asked to evaluate three extraction procedures: Test Method F1349 using Miglyol 812 in place of corn oil; Test Method F1349 on extracts from a dough similar to a pizza crust consisting of 10:40:50 (w/w/w) Miglyol 812 + water + low-protein flour; and this procedure. Each laboratory was supplied with bilaminate susceptors, Miglyol 812 and flour from single lots, and appropriate standard materials for HPLC quantitation. Table 1 lists the statistical results for the determination of diethyleneglycol dibenzoate (DEGDB) and polyethylene terephthalate trimer (PETE) migrating from the susceptors, normalized to correct for the various sample areas used by individual collaborators.

NOTE 2—UV quantitation was used to establish the test method's validity because of the lack of other widely available detection methods. Precision for non-UV-absorbing compounds is expected to be similar for this procedure.

10.2 Since no absolute method is available for comparison, no statement regarding bias can be made for this test method.

11. Keywords

11.1 extractables, nonvolatile; extractables, non-UV absorbing; extraction cell, Waldorf; food simulant, corn oil; food simulant, dough; food simulant, Myglyol; food simulant, solvents; HPLC; microwave; microwave susceptors; Myglyol; nonvolatiles; susceptor; susceptors, microwave; temperature measurement, microwave

TABLE 1 Statistical Results for Determination of DEGDB and PETE Migrating From Susceptors

Analyte/Matrix	Amount Extracted, µg/in.^{2}					
	Average	S_{lab}	S_r	S_r CoV, %	S_R	S_R CoV, %
DEGDB, 2 min						
Dough	13.88	3.82	2.93	21	4.81	35
Miglyol 812	42.20	12.79	22.46	53	25.85	61
Test Method F1500	43.25	4.57	14.09	32	14.81	34
DEGDB, 5 min						
Dough	24.15	7.27	8.17	34	10.93	45
Miglyol 812	86.26	21.82	24.54	28	32.84	38
Test Method F1500	85.10	8.70	32.08	38	33.24	39
PETE, 2 min						
Dough	1.70	0.56	1.38	81	1.49	87
Miglyol 812	54.33	7.46	16.06	30	17.71	33
Test Method F1500	8.29	1.35	3.14	38	3.42	41
PETE, 5 min						
Dough	3.31	1.82	2.04	62	2.73	82
Miglyol 812	88.56	2.93	13.90	16	14.20	16
Test Method F1500	15.65	1.89	6.10	39	6.39	41
Gravimetric						
2 min	254	71	90	35	114	45
5 min	729	186	594	81	622	85

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