

Designation: F1500 - 98 (Reapproved 2019)

Standard Test Method for Quantitating Non-UV-Absorbing Nonvolatile Extractables from Microwave Susceptors Utilizing Solvents as Food Simulants¹

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.5 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

E260 Practice for Packed Column Gas ChromatographyE682 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 Pro-

filing for Microwave SusceptorsF1317 Test Method for Calibration of Microwave OvensF1349 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-UVabsorbing 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,

¹ This test method 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.

Current edition approved March 1, 2019. Published May 2019. Originally approved in 1994. Last previous edition approved in 2014 as F1500 – 98(2014). DOI: 10.1520/F1500-98R19.

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.

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.

6. Apparatus and Reagents

6.1 *Microwave Oven*, 700 \pm 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 $\frac{1}{16}$ -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 \pm 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 polytetrafluo-roethylene 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.