Designation: F1308 – 98 (Reapproved 2023)

Standard Test Method for Quantitating Volatile Extractables in Microwave Susceptors Used for Food Products[1]

This standard is issued under the fixed designation F1308; 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 complete microwave susceptors.

1.2 This test method covers a procedure for quantitating volatile compounds whose identity has been established and which are evolved when a microwave susceptor sample is tested under simulated use conditions.

1.3 This test method was collaboratively evaluated with a variety of volatile compounds (see statistical evaluation). For compounds other than those evaluated, the analyst should determine the sensitivity and reproducibility of the method by carrying out appropriate spike and recovery studies. The analyst is referred to Practice E260 for guidance.

1.4 For purposes of verifying the identity of or identifying unknown volatile compounds, the analyst is encouraged to incorporate techniques such as gas chromatography/mass spectroscopy, gas chromatography/infrared spectroscopy, or other techniques in conjunction with this test method.

1.5 A sensitivity level of approximately 0.025 μg/in.^{2} is achievable for the compounds studied in Table 1. Where other compounds are being quantitated and uncertainty exists over method sensitivity, the analyst is referred to Practice E260 for procedures on determining sensitivity of chromatographic methods.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.* Specific safety hazards warnings are given in 10.2, 11.1, and 11.6.

1.8 *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 Chromatography

F1317 Test Method for Calibration of Microwave Ovens

2.2 *TAPPI Standards:*

T 402 Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products[3]

TIS 808 Equilibrium relative humidities over saturated salt solutions[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *microwave susceptors, n*—a packaging material which, when placed in a microwave field, interacts with the field and provides heating for the products the package contains.

3.1.2 *volatile extractables, n*—those chemical species which are released from the microwave susceptor and can be detected in the headspace under conditions simulating those under which the susceptor is used. Extractability does not necessarily mean migration of the extractable species to the product being heated on the susceptors.

4. Summary of Test Method

4.1 Volatile extractables are determined by subjecting a sample of the susceptor material to microwave heating, followed by headspace sampling and gas chromatography. Qualitative analysis may be carried out on a gas chromatograph (GC) coupled to an appropriate detector capable of compound

[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.

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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] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

TABLE 1 Analyte Recovery Without Microwaving

Compound	(*n*)[A]	Recovery Mean, %	Within Laboratory Variability, %	Overall Variability, %	Note(s)[B]
Benzene	5	97.7	7.8	9.0	
2-Butoxy-ethanol	4	98.7	6.7	8.4	1
Dibutyl Ether	5	109.7	16.5	23.7	
Dodecane	3	101.1	10.7	10.7	1, 2
2-Furfural	4	99.7	11.7	12.0	1
Furan-2-Methanol	3	100.0	14.1	16.4	1, 3
Isobutyl Alcohol	4	96.0	7.1	7.9	4
Methylene Chloride	5	103.5	16.7	22.6	
2-Propanol	3	99.9	11.4	12.0	4
Styrene	5	100.8	8.5	9.3	
Toluene	4	102.7	9.9	10.9	4
Overall		101.1	11.6	14.4	

[A] *n* = number of laboratories submitting data on compound.
[B] Notes: Collaborating laboratories provided the following reasons for not submitting data on a particular analyte:
1. The analyst felt interaction was occurring among various analytes and spent several days investigating. The laboratory manager refused to allow additional time for collaborative study.
2. The analyst questioned the solubility of the analyte and did not add to the spike mixture.
3. A fresh standard was not prepared fresh daily. This compound degrades measurably in water in 24 h.
4. The analyst experienced coelution of peaks under conditions of collaborative study on his/her particular system.

identification. Volatile extractables are quantitated by comparison with standards of known concentration.

5. Significance and Use

5.1 This test method is intended to measure volatile extractables that may be emitted from a microwave susceptor material during use. It may be a useful procedure to assist in minimizing the amount of volatile extractables either through susceptor design or manufacturing processes.

5.2 Modification of this procedure by utilizing appropriate qualitative GC detection such as a mass spectrometer in place of the flame ionization detector may provide identification of volatile extractables of unknown identity.

6. Interferences

6.1 *Gas Chromatography*—Because of the potentially large number of chemical species that can be analyzed using this methodology, not all species will be resolved from one another on a particular GC column under a given set of conditions. Techniques available to the analyst to verify the identity of the species being quantitated include retention time comparisons using alternate GC conditions or using an alternate GC column to verify identification. Good judgement of chromatographic results is always important.[4,5,6] Refer to Practice E260 for guidance.

6.2 *Apparatus*—Because this test method is designed for trace volatiles, and is highly sensitive, contaminants on vials, septa, syringes, etc. can lead to misinterpretation of results. Preparing apparatus properly and carrying out blank determinations as specified in the procedure is essential to minimize this possibility.

7. Apparatus and Reagents

7.1 *Microwave Oven*—Calibrated, 700 ± 35 W, no turntable. See Test Method F1317.

7.2 *Humidity Chambers*, operated at 50 % RH and 23 °C.

7.2.1 Requirements for constant temperature-humidity chambers and equilibrium relative humidities over saturated salt solutions are outlined in TAPPI Methods T 402-om-88, and TIS 808-03.

7.3 *Vials,* headspace, 20 mL (actual volume 21.5 mL). To ensure against extraneous peaks in the gas chromatographic traces, wash vials thoroughly and dry in a 125 °C air oven for a minimum of 4 h before using.

7.4 *Vial Crimp Caps.*

7.5 *Septa,* Polytetrafluoroethylene (PTFE)/silicone. To ensure that the septa are free of volatiles, cover the bottom of a 15 cm petri dish with septa, PTFE-polymer side up. Microwave at full power for 10 min. Place microwaved septa into a vacuum (greater than 29 in.) oven at 130 °C for 16 h.

7.6 *Crimping Tool* for vials.

7.7 *Syringe,* 2 mL, gas-tight with valve. Store syringe in 90 °C oven between uses.

7.8 *Gas Chromatograph* equipped as follows:

7.8.1 *FID Detector,* compatible with capillary columns.

7.8.2 *Injector,* split/splitless compatible with capillary columns.

7.8.3 *Automated Headspace Sampler, Optional.*

7.8.4 *Column,* DB-5, 30 m, 0.25 mm inside diameter, 1 µm film thickness, or 0.32 mm. (A short piece of deactivated 0.25 mm fused silica column may be placed between the injector and the column to serve as a guard column.)

7.8.5 *Peak-Area Integration System* compatible with GC system. Alternatively, a chart recorder and hand integration can be used.

7.9 *Fluoroptic Thermometry System* .

7.10 *Temperature Probes,* high temperature.

7.11 *Beaker,* 600 mL.

7.12 *Oven,* hot air, set for 90 °C.

7.13 *Stopwatch.*

7.14 *4-Heptanone.*

7.15 *Standard Solutions—Regular Method:*

7.15.1 *Internal Standard Solution* (245 µg/mL 4-Heptanone)—To approximately 950 mL of distilled water in

[4] McCown, S. M., and Radenheimer, P., "An Equilibrium Headspace Gas Chromatographic Method for the Determination of Volatile Residues in Vegetable Oils and Fats," *LC/GC*, Vol 7, No. 11, 1989, pp. 918–924.

[5] McNeal, T. P., and Breder, C. V., "Headspace Gas Chromatographic Determination of Residual 1,3-Butadiene in Rubber-Modified Plastics and Its Migration from Plastic Containers Into Selected Foods," *Journal of the Association of Analytical Chemists*, Vol 70, No. 1, 1987, pp. 18–21.

[6] McNeal, T. P., and Breder, C. V., "Headspace Sampling and Gas-Solid Chromatographic Determination of Residual Acrylonitrile in Acrylonitrile Copolymer Solutions," *Journal of the Association of Official Analytical Chemists*, Vol 64, No. 2, 1981, pp. 270–275.

a 1-L volumetric flask add 300 µL of 4-heptanone. Mix well and dilute to volume with water.

7.15.2 *Standard Solution 1:* (Prepare fresh daily.)—To approximately 475 mL of internal standard solution in a 500 mL volumetric flask, add 50 µL of each of the compounds to be quantitated. Mix well, and dilute to volume with internal standard solution. If difficulty is experienced with dissolution of analyte, alternate standard solution procedure may overcome this difficulty.

7.15.3 *Standard Solution 2*—Repeat 7.14.2 using 25 µL of each compound.

7.15.4 *Standard Solution 3*—Repeat 7.14.2 using 10 µL of each compound.

7.16 *Standard Solutions—Alternate Method:*

7.16.1 *Alternate Internal Standard Solution* (1225 µg/mL 4-Heptanone)—To approximately 150 mL of helium-sparged orthodichlorobenzene (ODCB) in a 200 mL volumetric flask add 300 µL of 4-heptanone. Mix well and dilute to volume with ODCB.

7.16.2 *Alternate Standard Solution 1*— To approximately 75 mL of alternate internal standard solution in a 100 mL volumetric flask, add 50 µL of each of the compounds to be quantitated. Mix well, and dilute to volume with alternate internal standard solution.

7.16.3 *Alternate Standard Solution 2*— Repeat 7.15.2 using 25 µL of each compound.

7.16.4 *Alternate Standard Solution 3*— Repeat 7.15.2 using 10 µL of each compound.

7.17 *Susceptor Blank*—Obtain a representative sample of susceptor material to be tested. Bake in an air oven overnight at 100 °C or higher to remove any volatile materials present. Store blank susceptor strips in humidity chamber 1 at 50 % RH and 23 °C until equilibrium moisture content is reached. An exposure time of 24 h is generally adequate for most paper-based products. Strips should remain in the conditioning environment until needed for analysis.

7.18 *Syringe Needle,* 13 gage.

7.19 *Variable Voltage Transformer, Optional*—This can occasionally be used for minor adjustments to line voltage to bring power output of the microwave oven into the specified range.

8. Instrument Setup

8.1 Determine sample test conditions as follows:

8.1.1 Set up microwave susceptor in the configuration of its intended use, that is, a popcorn bag filled with popcorn, a pizza disk with pizza on top, etc.

8.1.2 Place temperature probes (7.10) on susceptor surface, disturbing the normal food load as little as possible. If the susceptor has areas where the food does not normally contact the surface, place the probes in these areas. Place the product in the center of the microwave oven.

8.1.3 Cook the product in accordance with normal directions, for the maximum cooking time. Record this time. Record the probe temperature(s), preferably at 5 s intervals, but at intervals not to exceed 15 s during cooking.

8.1.4 Place 250 mL of room-temperature distilled water into a 600 mL beaker. Place the beaker in the center rear of the microwave oven.

8.1.5 Cut a 10 mm by 65 mm ($6.5\ cm^2 = 1\ in.^2$) portion from the susceptor sample to be tested. Insert carefully into the 20 mL headspace vial.

8.1.6 Using a 13 gauge syringe needle, pierce a hole into a headspace vial septum. Place the septum on the vial and crimp.

8.1.7 Insert one temperature probe (7.10) through the septum hole into the vial and manipulate it until it is in contact with the active face of the susceptor material. Place the vial on its side in the center of microwave oven, crimp end toward right of the oven, and susceptor with active face up.

8.1.8 Microwave at full power, recording the probe temperature, preferably at 5 s intervals, but at intervals not to exceed 15 s.

8.1.9 Plot the temperatures from 8.1.3 and 8.1.8 on the same graph.

8.1.10 Compare the plots. If the trace from 8.1.8 closely approximates or is slightly higher than the plot from 8.1.3 then the test time will be equal to the maximum product cook time of the product in that oven. If the trace is substantially higher or lower than that of the susceptor with product, then adjust the mass or surface area, or both, (by changing container size) of the water (using a fresh sample of room temperature distilled water) as necessary to achieve a similar profile. Record the mass of water and type of container that gives the best agreement between the test sample and the product temperature profiles.

8.2 Set up the gas chromatographic system to meet the following criteria.

8.2.1 *Injector Temperature*—250 °C.

8.2.2 *Detector Temperature*—250 °C.

8.2.3 *Column Temperature:*

8.2.3.1 *Initial*—40 °C for 4 min.

8.2.3.2 *Program*—Adjust to give a retention window of:

(1) At least 15 min for volatile compounds bracketed by 2-propanol and dichlorobenzene, retention time for 2-propanol of approximately 3 min and retention time for dichlorobenzene of approximately 20 min.

(2) Providing a separation of Di-*n*-butyl ether and styrene of $R = 0.5$ or greater. For a 30 m by 0.25 mm column this is approximately 4 °C/min with a nominal carrier flow of 1.5 mL/min.

8.2.4 Attenuation or sensitivity, or both, set to give an internal standard peak height of 60 % to 90 % of full scale on recorder or integrator.

9. Sampling

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

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

9.3 Carefully cut a 10 mm by 65 mm ($6.5\ cm^2 = 1\ in.^2$) portion from the susceptor. Carefully trim away any frayed edges before testing. Store susceptor test strips in humidity