Designation: F1308 – 98(Reapproved 2008)

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 and health 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.

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*—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*—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 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

[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.

[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.

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 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

[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.