Designation: F1308 – 98 (Reapproved 2014)

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.

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 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-gage 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 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 chamber 2 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.

10. Calibration

10.1 Cut a 10 by 65-mm portion of susceptor blank material (prepared in 7.17) and insert carefully into the 20- mL headspace vial. Add 10 µL of internal standard solution and immediately cap and crimp vial with PTFE side of septum toward vial.

10.2 Heat sample in air oven (or autosampling device) at 90°C for 10 min. (**Warning—**When handling a hot syringe, be sure hands are adequately protected.)

10.3 Fill gas-tight syringe with 1 mL of air, close valve, and insert needle through septum into vial. Open valve, and inject air into vial. Draw ½ mL of gas from vial into syringe, and inject back into vial. Repeat two times. Draw exactly 1 mL of gas into syringe, and close valve. Withdraw needle, insert into injector of GC equipped with an FID detector, and inject.

NOTE 1—Consistent technique from injection to injection of standards and sample is very important. The analyst should strive to achieve a consistent handling time of 30 s or less for this step. Alternatively, use optional automated headspace sampling system to introduce headspace gases onto GC system for analysis.

10.4 Review chromatogram of blank sample to ensure against extraneous peaks. In some cases, bottled air may be necessary to ensure against contamination from laboratory air. Similarly, peaks arising from septa, vials, etc. need to be investigated and eliminated.

10.5 Repeat 10.1 through 10.3 using 10 µL of Standard 1 (or 10 µL of distilled water and 2 µL of alternate Standard 1) in place of the internal standard solution(s).

10.6 Repeat 10.1 through 10.3 using 10 µL of Standard 2 (or 10 µL of distilled water and 2 µL of alternate Standard 2) in place of the internal standard solution(s).

10.7 Repeat 10.1 through 10.3 using 10 µL of Standard 3 (or 10 µL of distilled water and 2 µL of alternate Standard 3) in place of the internal standard solution(s).

10.8 Construct a standard calibration curve as follows for each compound being quantitated.

10.8.1 For Standard 1 (or alternate Standard 1), the concentration of each analyte in micrograms per square inch is equal to the specific gravity of that analyte.

10.8.2 For Standard 2 (or alternate Standard 2), the concentration of each analyte in micrograms per square inch is equal to the specific gravity of that analyte divided by 2.

10.8.3 For Standard 3 (or alternate Standard 3), the concentration of each analyte in micrograms per square inch is equal to the specific gravity of that analyte divided by 5.

10.8.4 From the chromatograms of the standard solutions, measure the area of the analyte peak and the area of the internal standard peak for each of the three standard levels. Divide the area of the analyte by the area of the internal standard to give the relative peak area of the analyte in each case.

10.8.5 Plot the concentration of analyte in micrograms per square inch versus the relative peak area.

11. Procedure

11.1 Place the number of mL of room-temperature distilled water determined in 8.1.10 into the type of container determined in 8.1.10. (**Warning—**Add a number of carborundum boiling stones to guard against superheating of the water. Place the beaker in the rear of the microwave oven.)

11.2 Insert a 10 by 65-mm sample carefully into a 20-mL headspace vial.

11.3 Inject 10 µL of internal standard solution (or 10 µL distilled water and 2 µL alternate internal standard solution) into vial with susceptor.

11.4 Immediately place septum over vial, PTFE side toward vial, apply crimp cap and crimp securely.

11.5 Place vial on its side in the center of the microwave oven, crimp top toward the right of the oven, and susceptor with active side up. Apply full power to the sample for the time determined in 8.1.3.

11.6 Immediately remove sample from oven and place in 90°C air oven or heated sample holder for autosampling for 10 min. (**Warning—**When handling a hot syringe, be sure hands are adequately protected.)

11.7 Fill the gas-tight syringe with 1 mL of air, close the valve, and insert the needle through the septum into the vial. Open the valve, and inject air into the vial. Draw ½ mL of gas from the vial into the syringe, and inject back into the vial. Repeat two times. Draw exactly 1 mL of gas into the syringe, and close the valve. Withdraw the needle, insert into the injector of the GC equipped with an FID detector, and inject.

NOTE 2—Consistent technique from injection to injection of standards and sample is very important. The analyst should strive to achieve a consistent handling time of 30 s or less for this step. Alternatively, use optional automated headspace sampling system to introduce headspace gases onto the GC system for analysis.

11.8 Chromatograph the sample under the conditions used for establishment of the standard curve.

11.9 An empty vial containing only 10 µL of internal standard solution (or 10 µL distilled water and 2 µL alternate internal standard solution) should be carried through the entire procedure to ensure against artifactual peaks.

12. Calculation

12.1 Calculate analyte extracted from the susceptor as follows:

12.1.1 Measure the area of the analyte peak and the area of the internal standard peak. Divide the area of the analyte peak by the internal standard peak area to obtain the relative peak for that analyte.

12.1.2 From the standard curve in 10.8.5 determine the concentration of analyte in micrograms per square inch.

13. Precision and Bias

13.1 This test method was collaboratively studied on a susceptor of metallized polyethyleneterephthalate bonded to paperboard with ethylene vinyl acetate adhesive in five laboratories. Two volatile compounds were found in the susceptor tested. Each laboratory ran the test in triplicate with the following results:

Compound	Mean, µg/in.^{2}	Within Laboratory Variability, %	Overall Variability, %
Toluene	0.088	9.9	21.4
Furfural	3.1	24.7	32.8

13.2 Bias of this test method was determined by recovery studies. Collaborating laboratories were asked to spike a sample of the susceptor material prepared per 7.17 with a variety of volatile compounds at the three levels used for calibration. These samples were then taken through all but the microwave treatment step of the procedure. The results obtained are shown in Table 1.

13.2.1 The high-level spike had overall recovery of 100.5 ± 3.8 %, the mid-level spike an overall recovery of 99.5 ± 14.5 % and the low-level spike of 103.6 ± 20.7 %. Three of the laboratories used the regular standard calibration procedure, and two laboratories used the alternate procedure. The overall recovery for the regular procedure was 102.7 ± 17.3 %; for the alternative procedure the overall recovery was 99.4 ± 10.9 %.

13.3 Collaborating laboratories also were asked to spike the susceptor material as received with a variety of volatile compounds at the three levels used for calibration and carry the spiked material through the entire procedure. The results obtained are shown in Table 2.

13.3.1 The high-level spike had an overall recovery of 90.4 ± 38.3 %, the mid-level spike an overall recovery of 87.5 ± 31.6 %, and the low level of 105.6 ± 45.7 %. Three laboratories ran the regular standard calibration procedure, and two laboratories ran the alternate standard procedure. The recovery for the regular standard procedure was 92.2 ± 37.7 %; for the alternate standard the recovery was 96.3 ± 42.3 %.

TABLE 2 Analyte Recovery with Microwaving

Compound	(*n*)[A]	Recovery Mean, %	Within Laboratory Variability, %	Overall Variability, %	Notes[B]
Benzene	5	101.2	18.7	21.2	
2-Butoxy-ethanol	4	96.7	28.2	67.9	1
Dibutyl Ether	5	90.4	12.3	17.5	
Dodecane	3	95.5	26.3	37.3	1, 2
Furan-2-Methanol	3	80.4	42.2	77.0	1, 3
Isobutyl Alcohol	4	93.9	9.5	19.7	4
Methylene Chloride	5	99.0	25.3	26.5	
Styrene	5	93.8	14.0	21.7	
Toluene	4	77.9	14.7	21.2	4
Overall		95.6	23.0	38.1	

[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.

14. Keywords

14.1 extractables, volatile, quantitation, in microwave susceptors; fluoroptic thermometry; gas chromatography, static headspace; microwave susceptors; microwave suseptors, volatile extractables in; susceptors, microwave; volatile extractables, quantitation, in microwave susceptors