Designation: F1519 – 98 (Reapproved 2014)

Standard Test Method for Qualitative Analysis of Volatile Extractables in Microwave Susceptors Used to Heat Food Products[1]

This standard is issued under the fixed designation F1519; 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 identifying volatile extractables which are released when a microwave susceptor sample is tested under simulated end use conditions. The extractables are identified using gas chromatography/mass spectrometry (GC/MS).

1.3 This test method was evaluated for the identification of a variety of volatile extractables at a level of 0.010 µg/in.^{2} of susceptor surface. For extractables not evaluated, the analyst should perform studies to determine the level of extractable at which identification is achievable.

1.4 The analyst is encouraged to run known volatile extractables and/or incorporate techniques such as gas chromatography/high resolution mass spectrometry (GC/HRMS), gas chromatography/infrared spectroscopy (GC/IR) or other techniques to aid in verifying the identity of or identifying unknown volatile extractables. The analyst is referred to Practice E260 for additional guidance.

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

1.6 *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

F874 Test Method for Temperature Measurement and Profiling for Microwave Susceptors

F1308 Test Method for Quantitating Volatile Extractables in Microwave Susceptors Used for Food Products

F1317 Test Method for Calibration of Microwave Ovens

3. Terminology

3.1 *Definitions:*

3.1.1 *diffusion trapping*—the collection of volatile extractables on an adsorbent by means of the mass diffusion of the volatile extractables (**1**).[3]

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

3.1.3 *volatile extractables*—those compounds that give > 50 % recovery in spike and recovery studies using the applicable volatile extractables method. Extractability does not necessarily imply migration of the extractable species to the food product being heated on the susceptor.

4. Summary of Test Method

4.1 The volatile extractables are released from the susceptor when it has been heated to its end use heating conditions (temperature and heating time) using a thermostatically controlled oil bath or calibrated microwave oven. The released volatile extractables are concentrated by diffusion trapping on an adsorbent. After adsorption is complete, the adsorbent is heated to desorb the volatile extractables onto a gas chromatographic column (Refs **1–2**). The volatile extractables are then separated using a gas chromatograph and detected by a mass spectrometer. The volatile extractable identifications are confirmed by comparing their retention times and mass spectra to reference compounds under identical GC/MS conditions.

5. Significance and Use

5.1 This test method is intended to identify volatile extractables that may be emitted from microwave susceptor material during use. It may be a useful procedure to assist in minimizing the amount and type of volatile extractables produced. The

[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] The boldface numbers in parentheses refer to a list of references at the end of this test method.

susceptor design, materials used or manufacturing processes involved can be evaluated.

6. Interferences

6.1 *Gas Chromatography/Mass Spectrometry*—The GC conditions or column given may not exhibit sufficient resolution to identify all the volatile extractables. Alternate techniques should be used to identify the unresolved volatile extractables such as alternate GC conditions, an alternate GC column, GC/HRMS, and/or GC/IR. The retention time and mass spectrum or infrared spectrum of the volatile extractable should be verified with a reference standard.

6.2 *Apparatus and Materials*—Method interferences may be caused by contamination from vials, septa, syringes, etc., leading to misinterpretation of results at trace levels. All of the materials must be routinely demonstrated to be free from contamination under conditions of the analysis by running blanks.

7. Apparatus and Reagents

7.1 *Sample Cutter*—No. 14 cork borer.

7.2 *Glassware*—Wash all glassware thoroughly and dry in a 125°C air oven for a minimum of 4 h prior to using. Use no solvents.

7.2.1 *Vials*—40 mL.

7.2.2 *Culture Tubes*—10 by 75 mm.

7.3 *Vial Caps*—Screw caps for 7.2.1 vials.

7.4 *Vial Septa*—Polytetrafluoroethylene PTFE faced silicon backed septa, 22 mm diameter. Place septa into a vacuum oven at 135°C for 16 h prior to using.

7.5 *Volatile Adsorbent*—Refer to manufacturer's literature regarding physical, chemical, absorptive and desorptive characteristics of adsorbent.

7.5.1 *Adsorbent*—Tenax TA, 35/60 mesh.

7.5.2 *Conditioning*—Plug one end of a 14 cm long, 6.35 outside diameter by 5.3 mm inside diameter tube, premium grade 304 stainless steel with a plug of silanized glass wool. Fill tube with adsorbent, and plug other end with silanized glass wool. Connect the tube to the injection port outlet of the GC, set the UHP helium flow to 30 mL/min and condition adsorbent using the following program.

Injection temperature	250°C
Temperature 1	70°C
Time 1	30 min
Rate	10°C/min
Temperature 2	250°C
Time 2	60 min

7.5.3 *Storage*—Cap both ends of the tube after it cools, move to a chemical free area, uncap one end, remove glass wool, tap tube to transfer adsorbent to 40 mL glass vial, purge vial with UHP helium or argon for 1 min and seal with a conditioned PTFE/silicon septa (PTFE surface toward adsorbent). Exercise care in handling the adsorbent.

7.5.4 *Blanks*—The adsorbent should be tested for contamination prior to being used.

7.6 *Oil Bath*—Circulating bath capable of being heated to 250 ± 1°C. Use silicone oil to heat vials.

7.7 *Thermometers*—Capable of measuring up to 250°C. Calibrate thermometer with a NIST standardized thermometer to ensure its accuracy.

7.8 *GC/MS System:*

7.8.1 *Gas Chromatograph*—capable of temperature programming. The inlet carrier gas line should be equipped with a valve capable of being completely opened and closed within 1 s.

7.8.2 The injector should have a removable glass liner or insert, having a volume of at least 300 µL or 40 mg of adsorbent. The injector should have a closure that allows the liner/insert to be inserted and the injector sealed within 5 s. Modification of the injector may be required (**3**) through (**4**).

7.8.3 *GC Column*—60M Stabilwax, 0.25 mm ID, 0.5 µm df.

7.8.4 *Mass Spectrometer,* capable of scanning from 35 to 300 amu every 2 s or less when mass spectral data are obtained in the electron—impact ionization mode at a nominal electron energy of 70 eV.

7.8.5 *Data System*—An interfaced data system (DS) is required to acquire, store, reduce and output mass spectral data. The computer software must allow searching of any GC/MS data file for ions of a specific nominal mass and plot its abundance versus time or scan number. This type of plot is defined as an extracted ion current profile (EICP).

7.9 *Performance Volatile Standard for GC/MS System:*

7.9.1 *Stock Volatile Mixture*—Pipet in accordance with Table 1 the appropriate volume into a 100 mL volumetric flask which has been half filled with hexane. After all compounds have been added, fill to mark with hexane and mix well. Alternate compounds may be substituted. Refrigerate mixture at 4°C until needed.

7.9.2 *Performance Volatile Standard*—Dilute stock volatile mixture in step 7.9.1 1:1000 with hexane. Alternate dilutions may be made. Refrigerate standard at 4°C until needed.

7.10 *Susceptor Blank*—Obtain a representative sample of susceptor material to be tested. Bake in an air oven overnight at 105°C to remove any volatile extractables present. Store in a clean, sealed glass container (for example, desiccator) until needed.

TABLE 1 Stock Volatile Mixture—Preparation and Characteristic Ions, m/z, for Each Volatile

Compound	Volume Pipetted, mL[A]	Characteristic Ions, *m/z*
2-Methyl furan	1.7	82, 81, 53
Benzene	1.7	78, 77, 52
n-Propyl acetate	1.7	73, 43
Trichloroethylene	1.0	130, 95
Hexanal	2.0	56, 72, 82
n-Butyl alcohol	2.0	43, 41, 56
n-Butyl acrylate	1.7	55, 73, 85
Dodecane	2.0	57, 71, 85
Styrene	1.7	104, 103, 78
1,4-Dichlorobutane	1.5	55, 90
N,N-Dimethylformamide	1.5	73, 44, 42
Furfural	1.5	95, 96
Benzaldehyde	1.5	106, 105, 77
Pentanoic acid	1.5	73, 60
2-(2-Butoxyethoxy)-ethanol	1.5	45, 57, 75

[A] Pipet into 100 mL volumetric flask which has been half filled with hexane.

7.11 *Helium*—ultra high purity (UHP).

7.12 *Calibrated Oven*—see Test Method F1317.

8. Instrument Set-up

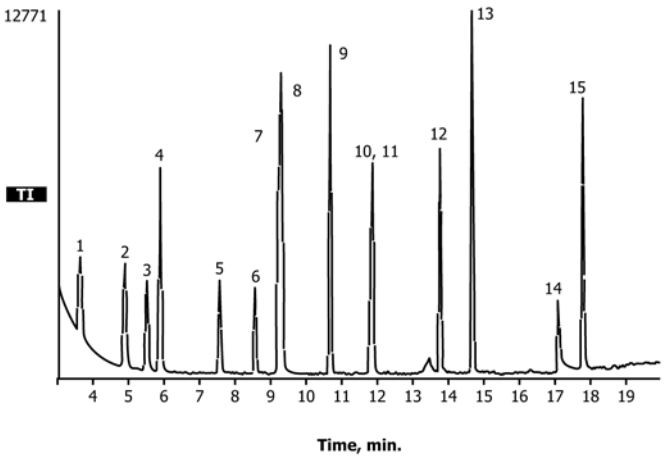
8.1 Setup the GC/MS/DS to meet the following criteria. Alternate conditions can be used to resolve unidentified volatile compounds.

Temperature 1	40°C
Time 1	3 min
Rate	10°/min
Temperature 2	210°C
Time 2	15 min
Carrier gas flow	10 mL/min UHP helium
Injector Temperature	250°C
Detector	electron multiplier
Interface temperature	250°C
Mass scanned	35–250 amu
Ionization voltage	70 eV

9. Daily GC/MS Performance Check

9.1 Tune the mass spectrometer in accordance with the instrument manufacturer's procedure.

9.2 Inject 1 µL (approximately 14 to 18 ng of each volatile is injected) of the performance volatile standard (see 7.9.2) to verify chromatographic retention times and mass spectra produced using conditions in Section 8. A typical chromatogram is shown in Fig. 1. As a minimum, the ions listed in Table 1 should be present and in their expected ratios for each volatile listed.



Number	Compound
1	2-Methyl Furan
2	Benzene
3	*n*-Propyl Acetate
4	Trichloroethylene
5	Hexanal
6	*n*-Butyl Alcohol
7	*n*-Butyl Acrylate
8	Dodecane
9	Styrene
10	1,4-Dichlorobutane
11	N,N-Dimethylformamide
12	Furfural
13	Benzaldehyde
14	Pentanoic Acid
15	2-(2-Butoxyethoxy)-Ethanol

FIG. 1 Total Ion Chromatogram of Performance Volatile Standard

9.3 Repeat 9.1 and 9.2 until these conditions are met prior to running any sample.

10. Sampling

10.1 Microwave susceptor sample selected for extraction should be representative of the entire susceptor.

10.2 Sample should be undamaged, that is, lamination intact, uncreased (unless this is its normal configuration), and unaltered.

10.3 Carefully cut a 0.75-in. diameter circular portion from the susceptor using a cork borer. Carefully trim away any frayed edges before extracting.

11. Procedure

11.1 Insert sample from 10.3 carefully into 40 mL vial.

11.2 Place enough conditioned Tenax-GC (approximately a volume of 250µ L or 40 mg) into a 10 by 75 mm culture tube and place it in vial with susceptor.

11.3 Immediately place septa over vial (PTFE side toward sample) and cap.

11.4 Place vial in an oil bath maintained at 218 ± 1°C (425 ± 2°F) for 5 min. The oil bath temperature should be verified using a calibrated thermometer. The temperature and time the sample is to be heated can be established using Test Methods F874 and F1317. Alternately Test Method F1308 can be used for heating the susceptor.

11.5 After heating, remove vial from oil bath and place in a 35°C oven for 16 hours.

11.6 Pour the Tenex GC from the culture tube into a GC injection port liner (see 7.8.2). A small funnel equipped with a short piece of plastic tubing will aid in the transfer. Place a plug of silanized glass wool into the other end of the liner to retain the Tenax.

11.7 Turn off the carrier gas to the GC by using the inlet toggle valve.

11.8 As quickly as possible, remove the cap from the injector, place the liner in the injector port, replace the cap and turn the carrier flow on.

11.9 Activate the GC program.

11.10 Chromatograph the sample using the conditions given in Section 8.

11.11 A vial containing only the Tenax in a culture tube should be carried through the entire procedure to identify potential artifactual peaks (**2**).

12. Volatile Extractable Identification

12.1 From the data obtained from Section 11, obtain a mass spectrum for the volatile extractable of interest. A background spectrum should be taken just before or after each volatile extractable elutes and subtracted from the volatile extractable spectrum to minimize mass spectral interferences.

12.2 Using a suitable reference library (**5**), search and find the best match for the volatile extractable mass spectrum in question.

12.2.1 Note that if several volatile extractables are present in the sample and coelute, the resulting spectrum will represent a composite. Alternate techniques may be needed to get a suitable mass spectrum of the volatile extractable of interest.

12.3 Using the same instrumental conditions that were used to analyze the sample, collect the mass spectra of authentic reference compounds.

12.4 Compare the mass spectra of the reference compounds to the mass spectra of the unknown volatile extractables to confirm the initial library search match.

12.5 Compare the retention times of the authentic reference compound and tentatively identified volatile extractable. If the retention time of the volatile extractable is within ± 1 % of the retention time of the reference compound, the two compounds may be considered the same.

13. Absorbent Efficiency

13.1 Three independent laboratories ran a collaborative study using this method to determine the effectiveness of the absorbent to adsorb and desorb volatile extractables reproducibly and accurately.

13.2 Each laboratory prepared aqueous standards of isopropyl alcohol, dibutyl ether, and toluene so that spiked susceptor samples containing approximately 10 ng of each of these compounds could be analyzed.

13.3 A 1 $in.^2$ sample of a vacuum dried, laminated product, was spiked with an aqueous standard on the paper side of the susceptor, placed in a PTFE sealed 40 mL vial, and held for 16 h at 35°C for equilibrium prior to analyses.

14. Reporting

14.1 All reports should include test conditions, especially the susceptor maximum temperature and time held at this temperature.

15. Precision and Bias

15.1 This is a qualitative method which requires that substances must be adsorbed, then desorbed and identified. The data acquired in Section 13 and displayed in Table 2 showing the collection and identification of substances of different polarities. The published data in the Refs **1**, **6**, **7**, and **2** show the utility and viability of this test method.

16. Keywords

16.1 characteristic mass; diffusion trapping; extractables, volatile; gas chromatography/mass spectrometry; mass spectrometry; microwave; microwave heating; microwave oven; microwave susceptors; qualitative analysis; susceptor ; susceptors, microwave

TABLE 2 Tenax GC Adsorption/Desorption Efficiency Study

NOTE 1—Vacuum-dried susceptor materials were spiked with 0.01 µg/10 $in.^2$ of susceptor material.

Compound	Positive Identification		
	Laboratory 1	Laboratory 2	Laboratory 3
Isopropyl Alcohol	positive ID	positive ID	positive ID
Sample No. 1	yes	yes	yes
Sample No. 2	yes	yes	yes
Sample No. 3	yes	yes	yes
Sample No. 4	yes	yes	yes
Sample No. 5	yes	yes	yes
Toluene	positive ID	positive ID	positive ID
Sample No. 1	yes	yes	yes
Sample No. 2	yes	yes	yes
Sample No. 3	yes	yes	yes
Sample No. 4	yes	yes	yes
Sample No. 5	yes	yes	yes
Dibutyl Ether	positive ID	positive ID	positive ID
Sample No. 1	yes	yes	yes
Sample No. 2	yes	yes	yes
Sample No. 3	yes	yes	yes
Sample No. 4	yes	yes	yes
Sample No. 5	yes	yes	yes

REFERENCES

(1) Booker, J. L., "Collecting Volatile Compounds by Simple Diffusion," *Journal of Chromatography Science*, Vol 23, 1985, p. 415.

(2) MacLeod, G., and Ames, J. M., "Comparative Assessment of the Artifact Background on Thermal Desorption of Tenax-GC and Tenax-TA," *Journal of Chromatography Science*, Vol 355, 1986, p. 393.

(3) Pankow, J. F., and Isabelle, L. M., "Adsorption Thermal Desorption as a Method for the Determination of Low Levels of Aqueous Organics," *Journal of Chromatography Science*, Vol 237 , 1982, p. 25.

(4) Nunez, A. J., Gonzalex, L. F., and Janak, F.," Pre-Concentration of Headspace Volatiles for Trace Organic Analysis by Gas Chromatography," *Journal of Chromatography Science*, Vol 300, No. 1, 1984, p. 127.

(5) The Royal Society of Chemistry," Eight Peak Index of Mass Spectra," third edition 1983, published by the Mass Spectrometry Data Centre, *The Royal Society of Chemistry*, The University, Nottingham, NG7 2RD, UK.

(6) Eiceman, G. A., Field, L. R., and Sievers, R. E.," Gas Chromatographic Injector Attachment for the Direct Insertion and Removal of a Porous Polymer Sorption Trap," *Analytical Chemistry*, Vol 50, No. 14, 1978, p. 2152.

(7) Lawrence, A. H., and Elias, L., "A Single-Stage GC Injector Apparatus for Thermal Desorption of Sorbent Tubes," *American Laboratory*, July 1989, p. 88.

(8) Pankow, J. F., Isabelle, L. M., and Kristensen, T. J., "Tenax-GC Cartridge for Interfacing Capillary Column Gas Chromatography with Adsorption/Thermal Desorption for Determination of Trace Organics," *Analytical Chemistry*, Vol 54, 1982, p. 1815.