



Designation: F 1884 – 98

Standard Test Method for Determining Residual Solvents in Packaging Materials¹

This standard is issued under the fixed designation F 1884; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the amount of residual solvents released from within a packaging material contained in a sealed vial under a given set of time and temperature conditions and is a recommended alternative for Test Method F 151.

1.2 This test method covers a procedure for quantitating volatile compounds whose identity has been established and which are retained in packaging materials.

1.3 The analyst should determine the sensitivity and reproducibility of the method by carrying out appropriate studies on the solvents of interest. The analyst is referred to Practice E 260 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 suitable techniques in conjunction with this test method.

1.5 Sensitivity of this test method in the determination of the concentration of a given retained solvent must be determined on a case by case basis due to the variation in the substrate/solvent interaction between different types of samples.

1.6 This test method does not address the determination of total retained solvents in a packaging material. Techniques such as multiple headspace extraction can be employed to this end. The analyst is referred to the manual supplied with the GC-Autosampling system for guidance.

1.7 The values stated in SI units are to be regarded as the standard.

1.8 *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:

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods²

E 260 Practice for Gas Chromatographic Procedures²

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method²

F 151 Test Method for Residual Solvents in Flexible Barrier Materials³

3. Terminology

3.1 Definitions:

3.1.1 *ream*— $3000 \text{ ft}^2 = 278.7 \text{ m}^2 = 27.87 \times 10^6 \text{ cm}^2$.

3.1.2 *retained solvents*—those chemical species, which are retained by packaging material and can be detected in the headspace of sealed sample vials under conditions of elevated temperature.

4. Summary of Test Method

4.1 Retained volatile organic solvents are determined by subjecting the packaging material to elevated temperatures in a headspace sampling system with subsequent gas chromatography of the headspace and detection using a suitable detection device such as a flame ionization detector (FID).

4.2 Volatile components can then be quantified by comparison with standards of known concentration.

4.3 Qualitative analysis may be carried out on a gas chromatograph (GC) coupled to an appropriate detector capable of compound detection / identification, such as a mass spectrometer or infrared detector.

5. Significance and Use

5.1 This test method is intended to measure volatile organic compounds that are emitted from packaging materials under high-temperature conditions.

5.2 This test method may be useful in assisting in the development and manufacture of packaging materials having minimal retained packaging ink/adhesive solvents.

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

¹ This test method is under the jurisdiction of ASTM Committee F-2 on Flexible Barrier Materials and is the direct responsibility of Subcommittee F02.30 on Test Methods.

Current edition approved May 10, 1998. Published March 1999.

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 15.09.

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 chemical species being quantitated include retention time comparisons using alternate GC conditions or using an alternate GC column. Good judgment in the interpretation of chromatographic results is always important. Refer to Practice E 260 for guidance.

6.2 *Apparatus*—Because this method is designed for detecting trace quantities of organic compounds, contaminants can lead to misinterpretation of results. Preparing apparatus properly and carrying out blank determinations is essential to minimize this possibility.

7. Apparatus and Reagents

7.1 Gas chromatograph equipped as follows:

7.1.1 *FID Detector*, compatible with capillary columns.

7.1.2 *Injector*, split/split-less compatible with capillary columns.

7.1.3 *Column*, DB-5, 30m, 0.25 mm ID, 1 μm film thickness, Cat. No. 122-5033, or 0.32 mm, Cat. No. 123-5033.⁴ A short piece of deactivated fused silica column may be placed between the injector and the column to serve as a guard column.

7.1.4 *Peak Area Integration System*, compatible with GC system in use. Alternately, a chart recorder and hand integration can be used.

7.1.5 Auto sampler is recommended.

7.2 *Standard Solutions*, consisting of the organic solvent mixture of interest, at concentrations that simulate the expected retention levels. 4-Heptanone may be added to the solutions for use as an internal standard as described in Practice E 260.

7.2.1 An example of a working standard is listed below. The standard used will vary based on the solvents present in the sample to be tested. The quantities shown in the table will result in roughly equivalent size peaks due to differences in detector response. If the solvents are mixed neat, adding 1 μL per gram of material in the headspace vial provides a good starting point for calibration.

7.2.2 If desired, water may be used as the diluent for the standard. The solvents are diluted in 1 L of water, typically 2 mL of the resulting solution is added per gram of sample in the headspace vial for calibration. 2 mL of 20 μL/L of 4-heptanone containing solution in water can be used as an internal standard.

NOTE 1—Water will change the partition coefficient between the sample and retained solvents.

Solvent	μL/L	μg/mL
Methanol	120	94.96
Ethanol	80	63.14
2-Propanol	60	47.13
n-Propanol	60	48.21
Methylethyl ketone	40	32.20
Ethylacetate	40	36.08
2-Propylacetate	20	17.08
Benzene	10	8.76
Methylisobutylketone	20	16.02
Toluene	10	8.70
Heptanone	20	16.42

7.3 *Vials*, 20 mL.⁵ 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*.⁶

7.5 *Septa*, Teflon/Silicone.⁷ To ensure that the septa are free of volatiles, condition the septa in a vacuum oven at 130°C for 16 h.

7.6 *Crimping Tool for Vials*.⁸

7.7 *Syringe*—2 mL gas tight with valve.⁹ Store syringe in 90°C oven between uses.

7.8 *4-Heptanone*.¹⁰

7.9 *For Manual Injection Only*—Hot air oven and heat resistant gloves.

8. Instrument Setup

8.1 Set up the gas chromatographic system per the manufacturer's recommendations and as follows:

8.1.1 *Injector Temperature*—250°C.

8.1.2 *Detector Temperature*—250°C.

8.1.3 *Column Temperature*:

8.1.3.1 Initial 40°C for 4 min.

8.1.3.2 *Program*—Adjust temperature program to give a retention window of at least 15 min to ensure optimum separation of solvents.

8.1.4 Attenuation or sensitivity, or both, set to give a detector response of 40 % or more of full scale on the recorder or integrator of the expected internal standard and standard sample response. See Practice E 260 for guidance.

⁵ Cat. No. 6676601, available from Shamrock Glass Co., 220 N. Delaware Ave., Seaford, DE or Cat. No. 237-502 Chemical Research Supplies, PO Box 888, Addison, IL 60101, or equivalent, have been found suitable for this purpose.

⁶ Cat. No. 778704, available from Shamrock Glass Co., 220 N. Delaware Ave., Seaford, DE or Cat. No. 07675-20625, available from Hewlett Packard Computer Supplies Operation, PO Box 62124, San Francisco, CA 94162, or equivalent, have been found suitable for this purpose.

⁷ Cat. No. 778183A, available from Shamrock Glass Co., 220 N. Delaware Ave., Seaford, DE or Cat. No. 5080-8726, available from Hewlett Packard Computer Supplies Operation, PO Box 62124, San Francisco, CA 94162, or equivalent, have been found suitable for this purpose.

⁸ The sole source of supply of the apparatus known to the committee at this time is Cat. No. 33280, Supelco Inc., Bellefonte, PA 16823. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁹ The sole source of supply of the apparatus known to the committee at this time is Cat. No. 050034, Alltech, 2051 Waukegan Rd., Deerfield, IL 60015. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

¹⁰ The sole source of supply of the apparatus known to the committee at this time is Cat. No. 10, 174-5, Aldrich, 940 W. St. Paul Ave., Milwaukee, WI 53233. If you are aware of the alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁴ The sole source of supply of the apparatus known to the committee at this time is J. and W. Scientific, Cat. No. 122-5033 and Cat. No. 123-5033. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.