Designation: F1884 – 04 (Reapproved 2011)

Standard Test Methods for Determining Residual Solvents in Packaging Materials[1]

This standard is issued under the fixed designation F1884; 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 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 F151.

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 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 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:*[2]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E260 Practice for Packed Column Gas Chromatography

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F151 Test Method for Residual Solvents in Flexible Barrier Materials (Withdrawn 2004)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *ream*—3000 ft^2 = 278.7 m^2 = 27.87×10^6 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.

[1] This test method is under the jurisdiction of ASTM Committee F02 on Flexible Barrier Packagingand is the direct responsibility of Subcommittee F02.15 on Chemical/Safety Properties.

Current edition approved April 1, 2011. Published April 2011. Originally approved in 1998. Last previous edition approved in 2004 as F1884 – 04. DOI: 10.1520/F1884-04R11.

[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 last approved version of this historical standard is referenced on www.astm.org.

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.

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 E260 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.

TEST METHOD A

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 E260.

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. 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,* Teflon/Silicone. 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.* [4,5]

7.7 *Syringe*—2 mL gas tight with valve.[4,6] Store syringe in 90°C oven between uses.

7.8 *4-Heptanone.* [4,7]

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 E260 for guidance.

8.2 Set up autosampler, if used, to heat vials for 20 min at 90°C before autoinjection.

[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.

[5] The sole source of supply of the apparatus known to the committee at this time is Cat. No. 33280, Supelco Inc., Bellefonte, PA 16823.

[6] 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.

[7] 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.

9. Calibration Procedure

9.1 *Standard Curve:*

9.1.1 Prepare blanks by heating a sample of the packaging material of interest (enough sample can be prepared at one time for several analysis runs) in a vacuum oven at 90°C for 24 h. Remove the blanks and store in a closed container. Blanks should be cut to the same relative size as the sample prior to heating in the vacuum oven.

9.1.2 To prepare a calibration standard place a blank (cut to appropriate size) in the 20 mL headspace vial and add the appropriate amount of standard solvent mix to the vial. Immediately cap and crimp the vial with the Teflon side of the septum toward the vial. It is suggested that blanks be fortified at five different concentrations along with an unfortified blank be prepared for calibration. See Practice E260 for guidance.

9.2 *Manual Injection:*

9.2.1 If using a syringe and hot air oven, heat each vial for 20 min at 90°C. Ensure that the syringe is heated to at least 90°C before taking headspace samples from the vials for injection into the chromatograph.

NOTE 2—When handling the hot syringe be sure that hands are adequately protected. Fill the gas tight syringe with 1 mL of air, close valve and insert the needle through the septum into the preheated vial. Open valve, inject the air into vial. Draw ½ mL of gas from vial into syringe, inject back into vial. Repeat 2 times. Draw exactly 1 mL of gas into syringe and close valve. Insert needle into injector of GC and inject.

NOTE 3—Consistent technique from injection to injection of standards and sample is required. This step should take no more than 30 s.

9.3 *Automated Injection*—The recommended method of injecting the headspace gas into the GC is use of an automated headspace sampling system where the vials are heated to 90°C for 20 min and then the headspace of each vial is automatically injected onto the GC column.

9.4 Repeat the procedure for all five calibration standards and the blank.

9.5 Construct a standard calibration curve from the data obtained using standard techniques as defined in Practice E260.

NOTE 4—Longer heating times may be used if it is deemed necessary to ensure that the solvent in the headspace of the vial has totally equilibrated with the sample.

10. Sampling

10.1 Samples should be taken in such a manner as to represent the entire web. The analyst should cut several layers deep into a roll of packaging material, discarding the outer layers, to ensure the sampling is representative of the entire roll. Samples should be taken from the left, center and right side of the web.

NOTE 5—Consideration should also be given when sampling rolls within a production lot to ensure uniformity within the production run.

10.2 Samples should be taken and handled in such a way as to minimize loss of solvent from the sample between the time the sample is taken, cut and loaded into the sample vial. Taking samples at press side, cutting and loading into vials immediately is the preferred method. Alternately, full web samples can be collected at press side and placed in a sealed container (samples can also be wrapped tightly in foil) for transport to the lab for cutting and loading into vials.

10.3 When taking samples from roll stock, discard the first 8 to 10 layers before taking samples from the next 30 to 40 layers to ensure that the samples are representative of the entire roll.

10.4 When possible, samples should have 100 % ink coverage in the area selected for testing. Selecting an area with 100 % ink coverage will ensure that the testing will elucidate a worst case. Using a sample area with representative ink coverage may also be considered.

10.5 The sample size is dictated by the thickness of the sample and the ease of filling the vial. The sample size will vary from 5 to 50 $in.^2$ Typically, the vial will be less than 20 % full by volume. Alternately the ratio of the weight of the sample in grams to the volume of the vial in millilitres should not exceed 1 to 10. In the case of a 20-mL sample vial, the weight of the sample should not exceed 2 g.

10.6 The preferred method of cutting samples is the use of a punch press or die.

10.7 Add the appropriate amount of internal standard (if used) to the vial.

10.8 Immediately cap and crimp the vial with the Teflon side of the septa toward the vial.

11. Procedure

11.1 *Manual Injection:*

11.1.1 For those using the syringe, place the sample (vial) in a forced air oven at 90°C for 20 min.

NOTE 6—Longer heating times may be used if it is deemed necessary to ensure that the solvent in the headspace of the vial has totally equilibrated with the sample.

NOTE 7—When handling the hot syringe be sure that hands are adequately protected. Fill the preheated gas-tight syringe with 1 mL of air, close valve and insert the needle through the septum into the above conditioned vial. Open valve, inject the air into vial. Draw ½ mL of gas from vial into syringe, inject back into vial. Repeat 2 times. Draw exactly 1 mL of gas into syringe and close valve. Insert needle into injector of GC and inject.

NOTE 8—Consistent technique from injection to injection of standards and sample is required. This step should take no more than 30 s.

11.2 *Automated Injection:*

11.2.1 The recommended method of injecting the headspace gas into the GC is use of an automated headspace sampling system where the vials are heated to 90°C for 20 min and then the headspace of the vial is automatically injected onto the GC column.

NOTE 9—Longer heating times may be used if it is deemed necessary to ensure that the solvent in the headspace of the vial has totally equilibrated with the sample.

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

11.4 Run a blank and one calibration standard along with each sample set to ensure system integrity.

11.5 Sample sets should contain a minimum of three replicates per sample.

TABLE 1 Summary of Precision Parameters[A]

NOTE 1—Six labs are required before S_{lab} and S_R data are statistically significant.

Sample	Analyte	Average mg/ream	S_{Lab}	S_r	S_R	r $2.8*S_r$	R $2.8*S_R$
Poly Laminate	Isopropyl Acetate	909.7	270	109	291	305	815
Poly Laminate	n-Propyl Acetate	4184.0	1126	386	1190	1082	3333
Paper Laminate	Isopropanol	4529.8	700	573	904	1603	2536
Poly Laminate	Ethyl Acetate	4792	1336	535	1439	1498	4030

[A]where:
S_{lab} = the standard deviation between laboratories,
S_r = the repeatability within laboratories, and
S_R is the reproducibility between laboratories.

12. Calculation

12.1 Calculate the amounts of retained solvents as follows:

12.1.1 Measure the area of the analyte peak and compare to the area with that from the standard curve and determine the concentration of the analyte in mg/ream of retained solvent. Normalize the analyte peak area with that of the internal standard peak area if the internal standard method is used before calculating the retained solvent concentration.

NOTE 10—The above methodologies are described in Practice E260.

12.2 Add each of the analyte concentrations together to yield a total retained solvent in mg/ream.

13. Report

13.1 Report the following information:

13.1.1 The identification of each known analyte peak observed,

13.1.2 Report the presence of unknown peaks, including the area of the peak in the report,

13.1.3 The average concentration of each known analyte, mg/ream,

13.1.4 The standard deviation for each analyte,

13.1.5 Total retained solvents (sum of all identified solvents), mg/ream total retained solvents,

13.1.6 Report the type of sample, collection location and storing method and any other information that might impact the results (press side, roll, slab, returned roll, wrapped in foil, etc.), and

13.1.7 Report the dates and times of manufacture, sampling and testing.

14. Precision and Bias

14.1 The round robin was conducted in 1995 in accordance with Practice E177 and Practice E691. Each sample consisted of three sub-samples taken from a given packaging material to represent the right, center and left side of the printed film roll. Laboratories were instructed to analyze each sub-sample in triplicate. Five laboratories reported data.

14.2 Each sample contained seven analytes, however because of the differences in testing equipment only four of the analytes were reported by all five labs. The bulk samples were prepared by one laboratory and placed in sealed aluminum foil pouches to minimize loss of solvents. The individual test specimens were prepared at the laboratory conducting the testing. Data reported consists of the average of three individual determinations on each sub-sample.

TEST METHOD B

15. Apparatus

15.1 *Container, rigid,* capable of being sealed vacuum-tight. The volume of this container should be approximately 1000 mL. This container should be fitted with a lid that will give a vacuum-tight seal and have a septum device through which head space vapors can be sampled.

15.1.1 *FID Detector,* compatible with capillary columns.

NOTE 11—A glass mason jar is recommended.

15.1.2 *Lids*—two different types are recommended as follows:

15.1.2.1 A stainless steel lid equipped with a standard gas chromatography septum held in place by a high pressure fitting.

15.1.2.2 A standard Mason jar dome lid with a 3.97-mm (5⁄32-in.) hole punched in the middle, equipped with a 5-mm sleeve type rubber stopper.

NOTE 12—Take care with both lid types that no extraneous components are added to the head space from the lid gasketing material.

15.2 *Syringes*

15.2.1 *Syringe,* 5-mL, gastight, equipped with a metal Luer valve and a chromatographic-type needle.

15.2.2 *Syringe,* 1.0-µL, to be used for introducing calibration solvents into the gas chromatograph.

15.2.3 *Syringe,* 100-µL.

15.3 *Large Tongs,* to be used in handling the heated sample container.

15.4 *Template,* for cutting samples. The dimensions of this template shall be 203 by 915 mm (8 × 36 in.) with a total area of 0.186 m^2 (2 ft^2).

15.5 *Foil,* aluminum free of any extraneous materials. This can be checked by running the foil through the GC test as a blank.

NOTE 13—Foil may be cleaned by heating in an oven at 340°C for 5 min.

15.6 *Vacuum Pump or Aspirator,* with vacuum gage, rubber hose, valve, and large bore hypodermic needle for evacuating sample containers.

16. Safety Precautions

16.1 Exercise care when handling any syringe to avoid the danger of puncture wounds from hypodermic needles.

16.2 Use gloves and tongs when handling the heated sample container to avoid burns.

16.3 If a glass sample container is used, exercise care to prevent implosion when evacuated or explosion when heated.

A perforated cylindrical metal shield may be used to eliminate any hazard to the operator.

17. Test Specimens

17.1 Cut duplicate specimens from each sample using the specified template. Whenever possible, orient the template at a 45° angle to the direction of the flexible packaging material, so that the analysis will reflect an average value for the sample.

18. Preparation of Apparatus

18.1 *Gas Chromatograph*—Follow the manufacturer's instruction in setting up the instrument. Allow sufficient time for all temperatures in the instrument to stabilize. Check the recorder base line stability at the highest sensitivity to be used during analysis. The base line should be free from excessive drift and noise.

18.2 *Sample Containers*—Scrub sample containers with a detergent and dry them in an oven before each use. Check the sample containers frequently for extraneous materials, which might interfere with the analysis, by carrying them through the analytical procedure as a blank.

NOTE 14—Avoid detergents that contain perfume.

18.3 *Syringes*—The 5.0-mL syringes must be gastight and checked often to see that they maintain this condition. In order to check for proper delivery, inject an air sample with the syringe into a gas chromatograph equipped with a thermal conductivity detector. Use the detector response for the air peak as a check on volume delivery. Syringes must also be carefully cleaned to avoid injecting extraneous materials.

19. Procedure

19.1 *Determination of Optimum Heating Time:*

19.1.1 Since variations in oven type, sample container, temperature control, sample characteristics, and so forth, will cause variations in the rate and level of solvent equilibration in the head space, it is necessary that each laboratory determine the optimum heating time to achieve equilibrium for each packaging material to be analyzed.

19.1.2 Set the oven to a suitable, selected temperature for the material being tested.

NOTE 15—For cellophane, 150°C has been found to be a suitable temperature. However, some flexible packaging materials may be chemically degraded at an oven temperature of 150°C. In this case, lower oven temperatures should be tried and a temperature determined at which solvent equilibration is obtained without serious degradation. Polyethylene, for example, is normally run from 70 to 80°C. The temperature that is used should be given in the detailed material specifications.

19.1.3 Place a swatch of aluminum foil in the bottom of each of several sample containers. Using the specified template, cut specimens of the packaging material to be analyzed. Cut each specimen into strips about 25.4 mm (1 in.) wide, put the strips into a container, and seal.

19.1.4 Using the vacuum pump or aspirator, evacuate each sample container to 380 mm (15 in.) mercury.

19.1.5 Heat one sample container in the oven for 5 min ± 10 s.

19.1.6 Remove the sample container from the oven using tongs and withdraw a sample of the head space vapors with a 5.0-mL gastight syringe as follows:

19.1.6.1 Open the valve on the syringe and push the syringe plunger all the way in. Close the valve.

19.1.6.2 Insert the needle into the septum.

19.1.6.3 Open the valve and draw the gas sample into the syringe.

19.1.6.4 Flush the syringe back into the container and pull another sample.

19.1.6.5 Close the valve and withdraw the needle from the septum.

19.1.6.6 Insert the needle into the septum of the gas chromatograph.

19.1.6.7 Open the valve and inject the sample into the chromatograph.

19.1.7 Perform the sampling operation immediately after removing the container from the oven because condensation of solvent vapors takes place. Condensation can be delayed by placing the container in an insulated sleeve immediately after removal from the oven. Also, heat the disassembled syringe, plunger, and barrel separately in the oven along with the sample container in order to minimize temperature drop.

19.1.8 Record the chromatogram of the vapor sample as previously described in the main method.

19.1.9 Heat another sample container in the oven for 10 min ± 10 s.

19.1.10 Remove the container from the oven and withdraw a vapor sample. Inject the sample into the gas chromatograph in accordance with 19.1.6.

19.1.11 Repeat the procedure for the remaining samples using heating times of 15, 20, 25, and 30 min ± 10 s successively.

19.1.12 For each solvent peak in the chromatograms, construct a graph of peak area, versus, heating time.

19.1.13 From these graphs, determine the heating time necessary to reach maximum peak area, for all solvents in the sample. Add 1 min to this to obtain heating time.

NOTE 16—Solvents will vary in their equilibration rate, depending on boiling point and degree of chemical "attraction" to the material being analyzed. If solvents with a wide boiling point range are being analyzed quantitatively it may be necessary to establish heating time for each range. It is more desirable, however, to find a maximum heating time to extract all solvents of interest.

19.1.14 If an optimum heating time is not reached within 30 min.; analyze additional samples at longer heating times.

19.2 *Adjusting Vacuum Level:*

19.2.1 After an optimum heating time is established, adjust the initial vacuum level in the sample container to leave a slight negative pressure in the container after heating. Since cellophane and paper samples contain moisture, the initial vacuum will have to be greater than for other materials such as polyethylene film.

19.3 *Determination of Recovery of Volatilized Solvents:*

19.3.1 Loss of solvent vapors from the sample container or from the gastight syringe causes large analytical errors. Therefore, check containers and syringes initially and at periodic

intervals for solvent loss. Measurement of solvent recovery is one way in which this can be done.

19.3.2 Make up two to three solutions of a spectro-grade solvent, (for example, toluene in a higher boiling solvent such as Cellosolve acetate (ethylene glycol monoethyl ether acetate) in the concentration range from 3 to 20 mg toluene/g solution).

19.3.3 Using a 1.0 µL-syringe, inject 1.0 µL of solution directly into the chromatograph. Record the chromatogram, noting the attenuations used.

19.3.4 Place a swatch of aluminum foil in the bottom of a clean sample container. Close the container and evacuate to the vacuum level established in 19.2.

19.3.5 Using a 100-µL syringe, inject 200 µL of the same solution into the container. Heat it at 150°C for the optimum heating time. Withdraw 5.0 mL of head space vapors and inject them into the chromatograph in accordance with 19.1.6. Record the chromatogram, noting the attenuations used.

19.3.6 Since 5.0 mL of head space vapors will contain the same mass of toluene as 1.0 µL of liquid solution, the response factor, RI, for both liquid and vapor samples should be equal if there are no leaks in the container or syringe.

19.3.7 Repeat the procedure with other solutions in the 3 to 20 mg toluene/g solution range.

19.4 *Method of Analysis:*

19.4.1 *Calibration:*

19.4.1.1 Place a swatch of aluminum foil in the bottom of a clean sample container. Close and evacuate the container to the vacuum level determined in 19.2.

19.4.1.2 Using a 1.0-µL syringe, inject 1.0 µL of pure solvent into the container.

19.4.1.3 Heat the container in the oven at 150°C for the optimum heating time.

19.4.1.4 Remove the container from the oven, immediately withdraw a 5.0-mL sample of head space vapors, and inject it into the gas chromatograph in accordance with 19.1.6. Record the chromatogram noting the attenuations used.

19.4.1.5 Run duplicate determinations at other volumes that will produce solvent concentrations in the head space in the same range as one expects to encounter in the printed or coated materials.

19.4.1.6 Repeat the above procedure for each solvent to be analyzed.

19.4.2 *Analysis of Flexible Packaging Material:*

19.4.2.1 Place a swatch of aluminum foil in the bottom of a clean sample container. Cut a test specimen of the flexible packaging material to be analyzed using the specified template (0.186 m^2).

19.4.2.2 Cut the test specimen into small strips about 25.4 mm (1 in.) wide, and put them into the sample container. Immediately close the container, and evacuate it to vacuum level established in 19.2.

19.4.2.3 Place the container in the oven at 150°C, and heat it for the optimum heating time.

19.4.2.4 Remove the container from the oven, and immediately sample the head space in accordance with 19.1.6.

19.4.2.5 Record the chromatogram of the head space vapors noting the attenuations used.

19.4.2.6 Repeat the above procedure using a duplicate sample.

20. Calculation

20.1 Calculate the amounts of retained solvents as follows:

20.1.1 Measure the area of the analyte peak and compare to the area with that from the standard curve and determine the concentration of the analyte in mg/ream of retained solvent. Normalize the analyte peak area with that of the internal standard peak area if the internal standard method is used before calculating the retained solvent concentration.

NOTE 17—The above methodologies are described in Practice E260.

20.2 Add each of the analyte concentrations together to yield a total retained solvent in mg/ream.

21. Report

21.1 Report the following information:

21.1.1 The identification of each known analyte peak observed,

21.1.2 Report the presence of unknown peaks, including the area of the peak in the report,

21.1.3 The average concentration of each known analyte, mg/ream,

21.1.4 The standard deviation for each analyte,

21.1.5 Total retained solvents (sum of all identified solvents), mg/ream total retained solvents,

21.1.6 Report the type of sample, collection location and storing method, and any other information that might impact the results (press side, roll, slab, returned roll, wrapped in foil, and so forth), and

21.1.7 Report the dates and times of manufacture, sampling and testing.

22. Precision and Bias

22.1 Table 2 and Table 3 are based on a round robin conducted in 1982 involving four materials tested by ten laboratories. Each test result was based on three individual determinations. Each laboratory obtained one test result for each material[8].

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1003.

TABLE 2 Statistical Data

Material	Mean mg/m^2	S_r	S_R	I_r	I_R
1[A]					
A Tetrahydrofuran	3.385	0.557	1.954	1.576	5.530
B Toluene	3.147	0.411	2.071	1.163	5.861
2[A]					
C Tetrahydrofuran	1.491	0.257	0.509	0.727	1.440
D Toluene	0.902	0.182	0.248	0.515	0.702
3[B]					
E Tetrahydrofuran	1.013	0.089	0.367	0.252	1.039
F Toluene	8.461	0.982	2.836	2.779	8.026
4[B]					
G Tetrahydrofuran	0.999	0.096	0.355	0.272	1.005
H Toluene	7.994	0.726	2.640	2.055	7.471

[A] Saran-coated cellophane.
[B] Saran-coated polyester film.

TABLE 3 Means and Relative Standard Deviations

Material[A]	Mean mg/m^2	Relative Standard Deviation	
		Within Laboratory, %	Between Laboratory, %
A	3.4	16.5	57.7
B	3.2	13.1	65.8
C	1.5	17.2	34.1
D	0.9	20.2	27.5
E	1.0	8.8	36.2
F	8.5	11.6	33.5
G	1.0	9.6	35.5
H	8.0	9.1	33.0

[A] See Table 2.

22.2 In Table 2 and Table 3, for the materials indicated:

S_r = is the within-laboratories standard deviation of the mean, when the mean is the average of 3 specimens;
S_R = is the total among-laboratories standard deviation of the mean, when the mean is the average of 30 specimens;
I_r = 2.83 S_r (see 15.3); and
I_R = 2.83 S_R (see 15.4) of Table 2.

22.2.1 Other materials may give somewhat different results.

22.3 *Repeatability*—In comparing two averages (of three specimens each) for the same material, obtained by the same operator using the same equipment on the same day, the averages should be judged not equivalent if they differ by more than the I_r value for that material and condition.

22.4 *Reproducibility*—In comparing two averages (of three specimens each) for the same material, obtained by different operators using different equipment on different days (in the same laboratory or in different laboratories), the averages should be judged not equivalent if they differ by more than I_R value for that material and condition.

22.5 The judgments resulting from 15.3 and 15.4 will be correct in approximately 95 % of such comparisons.

22.6 For further information on the methodology used in this section, see Practice E691.

22.7 No statement can be made about the bias of this test method, because there is no standard reference material or reference method that is applicable.

NOTE 18—Precision and Bias statement values are in mg/m^2. To convert from mg/m^2 to mg/ream multiply the value by 278.71.

23. Keywords

23.1 gas chromatography; head space analysis; packaging; residual solvents; retained solvent; volatile organic compounds