



Designation: F 151 – 86 (Reapproved 1997)

Standard Test Method for Residual Solvents in Flexible Barrier Materials¹

This standard is issued under the fixed designation F 151; 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 provides an index for comparing the level of solvents retained in flexible barrier materials of the same construction which result from casting, coating, printing, or laminating operations.

1.2 This test method does not yield absolute quantitative measurements of solvents retained in flexible barrier materials.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8 and Note 6.

2. Referenced Documents

2.1 A general guide for the application of gas chromatography (GC) to the analysis of vaporizable organic and inorganic mixtures is given in the following ASTM standards:

2.2 *ASTM Standards:*

E 260 Practice for Packed Column Gas Chromatography²

E 355 Practice for Gas Chromatography Terms and Relationships²

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

3. Terminology Definitions

3.1 *peak intensity, PI*—the response of the gas chromatographic detector to a liquid converted to an attenuation of 1:

$$PI = \text{peak height above base line} \times \text{attenuation}$$

NOTE 1—Peak area may be used in place of peak intensity.

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 March 27, 1986. Published May 1986. Originally published as F 151 – 72. Last previous edition F 151 – 72.

² *Annual Book of ASTM Standards*, Vol 14.02.

3.2 *response factor, RF*—the response (peak intensity) of the detector to a given mass of liquid:

$$RF = [(\text{microlitres of liquid}) (\text{density of liquid g/cm}^3 (10^3))] / PI$$

4. Summary of Test Method

4.1 Specimens of flexible barrier materials are placed in a closed container and heated to vaporize the retained solvents into the head space. A specimen of the head space vapors in the container is taken with a gastight syringe and injected into a gas chromatograph for analysis.

4.2 The recovery of volatilized solvents is determined by comparing the response factors, *RF*, of several different solutions with those for the same solutions vaporized in the test containers and the specimen then taken with a gastight syringe for injection into the gas chromatograph.

5. Significance and Use

5.1 Solvents retained in flexible barrier materials can cause odor, off-taste, ink offset, ghosting, and blocking problems. An accurate rapid method for comparing the level of retained solvents is valuable in quality control, process development, and complaint handling, particularly when the index has been correlated with the results of organoleptic tests, or with a total extraction method.

5.2 Round-robin testing has shown that this test method has a coefficient of variation between laboratory means of approximately $\pm 15\%$.

6. Apparatus

6.1 *Gas Chromatograph*, having a flame ionization detector with enough sensitivity to detect solvent vapors in a 5.0-mL air specimen in the range from 10 to 10 000 ppm. This chromatograph must be equipped with a partitioning column that will separate the solvents being analyzed from any other materials that might be injected with the specimen of head space vapors.

6.2 *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.

NOTE 2—A glass mason jar is recommended.

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

6.2.1.1 A stainless steel lid equipped with a standard gas chromatography septum³ (Fig. 1) held in place by a high pressure fitting.

6.2.1.2 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 3—Take care with both lid types that no extraneous components are added to the head space from the lid gasketing material.

6.3 Syringes:

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

6.3.2 *Syringe*, 1.0- μ L, to be used for introducing calibration solvents into the gas chromatograph.

6.3.3 *Syringe*, 100- μ L.

6.4 *Oven*, mechanically convected capable of maintaining a constant temperature in the range from 50 to 200 $\pm$ 2°C.

6.5 *Gloves*, cloth, leather, or asbestos, to protect the analyst's hands when handling the heated sample container.

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

6.7 *Template*, for cutting samples. The dimensions of this template shall be 203 by 915 mm (8 by 36 in.) with a total area of 0.186 m² (2 ft²).

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

NOTE 4—Foil may be cleaned by heating in an oven at 340°C for 5 min.

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

7. Reagents

7.1 Solvents of spectro-grade or equivalent purity shall be used for calibration purposes.

8. Safety Precautions

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

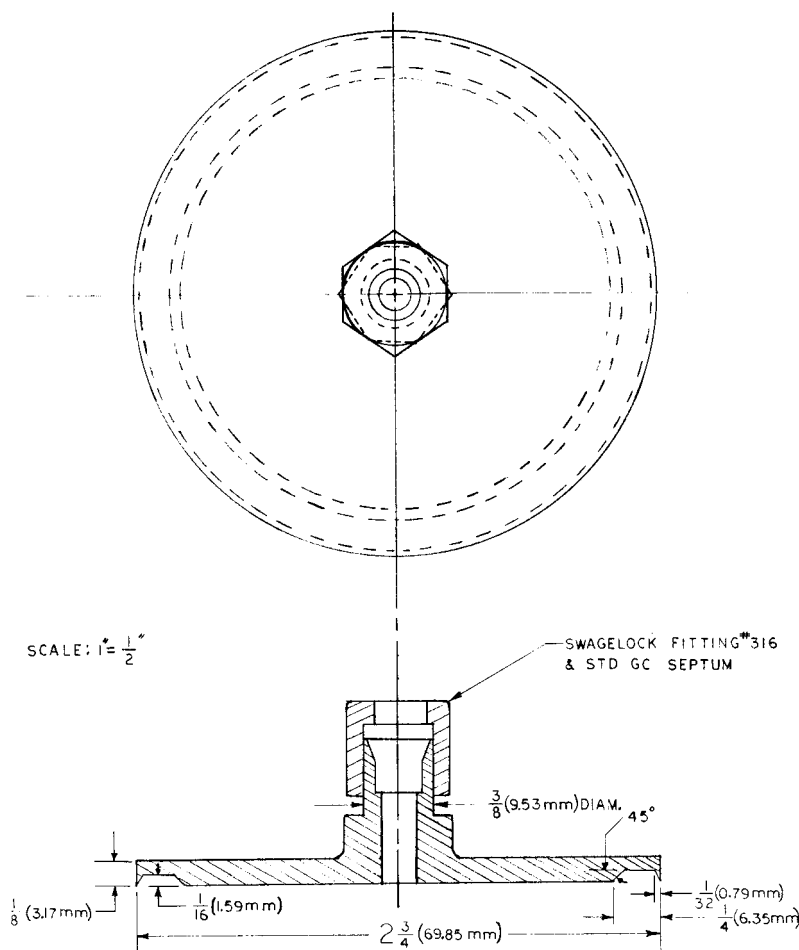


FIG. 1 Stainless Steel Lid with Standard Gas Chromatography (GC) Septum