The Institute for Interconnecting and Packaging Electronic Circuits
2215 Sanders Road • Northbrook, IL 60062-6135

IPC-TM-650
TEST METHODS MANUAL

Number
2.3.37

Subject
Volatile Content of Adhesive Coated Dielectric Films

Date	Revision
5/98	**B**

Originating Task Group
Flex Peel Strength Test Methods Task Group (D-13A)

1 Scope The purpose of the two test methods is to provide means for determining the volatile content of adhesive coated dielectric films used in the manufacture of flexible printed wiring. Method A is a weight loss procedure. Method B is a headspace gas chromatography procedure.

2 Applicable Documents None

3 Test Specimen

3.1 Method A The test specimens shall be squares of adhesive coated dielectric film. The specimens shall be 10 cm x 10 cm.

This method is to be used for non-aqueous solvent systems only.

3.2 Method B Three specimens shall be prepared: One specimen shall be cut from the center of the material and one each from each edge of the material. Specimens shall be cut no closer than 25.4 mm from the edge of the material.

4 Equipment/Apparatus

4.1 Method A

4.1.1 Analytical Balance Analytical balance capable of weighing to the nearest milligram (0.001 gram).

4.1.2 Test Chamber Circulating air oven maintained at 150°C ± 2.8°C.

4.2 Method B[1]

4.2.1 Analytical Balance Analytical balance capable of weighing to the nearest 0.1 milligram (0.0001 gram).

4.2.2 Sample Vials It is essential that the septa are placed with the Teflon® side toward the sample and that the vials are sealed tightly enough that it is not possible to turn the lid by hand.

4.2.3 Test Device A gas chromatograph fitted with a Head Space Sampler and a data acquisition/manipulation system capable of recording and quantitating gas chromatograms.

4.2.4 Chromatography Conditions

Tenax column 6.35 mm O.D. 1.8 m long.
Injector temperature: Product dependent
Oven temperature: Product dependent
Isothermal
Flame Ionization Detector:
Carrier gas is Nitrogen at kg/cm^2

Adjust equipment conditions for specific product being tested.

4.2.5 Headspace Conditions Temperature and dwell time dependent on product tested.

5 Procedure Method A

5.1 Preparation

5.1.1.1 Condition each specimen at 50% ± 5% relative humidity (RH) and 23°C ± 2°C for a minimum of three hours.

5.1.1.2 Weigh each specimen to the nearest milligram (0.001 gram) [W_1].

5.1.1.3 Subtract out substrate weight by calculating the area x the density of the substrate under test (W_s).

5.1.1.4 Hang each specimen from a metal hook in the circulating air oven at 150°C ± 2.8°C for 15 ± 1 minutes.

5.1.1.5 Remove each specimen from the chamber and place in a stabilization environment of 50% ± 5% RH and 23°C ± 2°C for a minimum of three hours.

5.1.1.6 Reweigh each specimen to the nearest milligram [W_F].

5.1.1.7 Subtract out substrate weight by calculating the area x the density of the substrate under test (W_s).

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[1] Method B requires proprietary information regarding the constituents of the material.

5.1.2 Evaluation The volatile content of each specimen is calculated as follows:

$$\text{Volatile Content (\%)} = \frac{(W_I - W_S)(W_F - W_S)}{(W_I - W_S)} \times 100$$

5.1.3 Report The results should be documented and contain the following:

1. Certification that the test was performed in accordance with this test method.
2. Identification of specimens tested.
3. Volatile content % for each specimen tested and the average.

Procedure Method B

5.2 Calibration

5.2.1 Prepare two liquid calibration mixtures as directed per vendor instructions. The mixtures do not need to have these exact amounts but the amount of each ingredient should be measured precisely to within 0.0002 grams. The mixture should be stored in a suitable container with a resealable septa lid. Calibration mixtures should be appropriate for volatiles present in product tested.

5.2.1.2 Make sure the mixtures are each well mixed, then determine the density of the mixtures by any acceptable method.

5.2.1.3 From each of the mixtures inject a minimum of four different amounts (0.5 to 3.0 microliters each) into separate, previously prepared, empty, sealed vials.

5.2.1.4 Allow each vial to equilibriate in the headspace unit, then inject each into the chromatograph and obtain an area for each sample.

5.2.1.5 Plot the areas vs. the amount (in milligrams) of each ingredient. The eight points should all lie on the same straight line.

5.2.1.6 Determine the response factor Rf (milligrams/area unit) for each ingredient. If the plot above is not linear or does not pass through the origin, then it might be necessary to have different Rf at different amounts.

5.2.2 Sample

5.2.2.1 Cut and weigh a 25.4 mm square sample of material to be tested. Place into sample vial and seal tightly. Equilibrate sample in the headspace unit and then inject onto the chromatography column. Allow the chromatogram to run for eight minutes.

Note 1: *Appropriate time and temperature dependent on product tested.*

5.2.2.2 Calculate the amount of each calibrated peak using the following equation.

Volatile % (i) = (Area (i) * Rf (i) * 100)/(Wt * F)

where: Area (i) = area generated for ith calibrated peak
Rf (i) = response factor for ith peak
Wa = weight of sample (not including substrate—subtract out substrate weight by calculating the area x the density of the substrate under test (W_s) and subtracting the value from the total sample weight (Wt). Wt −Ws = Wa)
F = fraction of ingredient in film that reaches the chromatography relative to the same fraction of pure liquid.

5.2.3 Determination of F

5.2.3.1 Determination of F__liq Prepare a mixture of ingredients to be tested. Inject 1 to 2 microliters of the mixture into a sealed vial. Equilibrate in the head space unit, then inject into the chromatograph and obtain the area for each peak of interest.

5.2.3.1.1 Immediately after the injection is complete, vent the vial by means of a small syringe needle and replace the vial to allow it to equilibrate again. Again after equilibration inject the vial into the chromatograph.

5.2.3.1.2 Vent the vial a second time and equilibrate and inject again. There should have been three injections, each preceded by an equilibration and two ventings, one immediately after the first injection and the other immediately after the second injection.

5.2.3.1.3 Calculate F__ret__liq and F__liq for each ingredient as follows.

F__ret__liq = (square root (area3/area2) + area2/area1)/2

F__liq = 1 − F__ret__liq

5.2.3.2 Determination of F__film Prepare a film sample in a vial as standard. Make three injections with venting between injections and equilibration before each one, just as in the case of the liquid. The F__ret__film and F__film are calculated in exactly the same way as F__ret__liq and F__film, except that the areas used are generated by the film sample.

5.2.3.3 F__liq and F__film are determined at least eight times, preferably over a period of several days. Approximately the same number of liquids as films are tested each day. The values of F__liq and F__film are averaged separately.

5.2.3.5 Once determined, F should not change unless the ingredients change or unless the equilibrium conditions change.

5.2.3.6 Different materials will have different values of F.

6 *Notes*

Multiple Headspace Extraction—A Procedure for Quantitative Analysis of Volatile Compounds in Solid Samples, Kolb, Auer, and Pospisil, ANGEWANTE CHROMATOGRAPHIE—APPLIED CHROMATOGRAPHY, January, 1981.