1 Scope

1.1 This infrared spectrophotometric analysis test method is for use in identifying the nature of nonionic organic contaminants present on printed wiring board surfaces or on the contaminated microscope slide used in the solvent extraction procedure defined in IPC-TM-650, Test Method 2.3.38, by use of the Multiple Internal Reflectance (MIR) Method. This test should be performed only by an experienced spectroscopist.

2 Applicable Documents

IPC-TP-383 Organic Surface Contamination – Its Identification, Characterization, Removal, Effects on Insulation Resistance and Conformal Coating Adhesion
2.3.38 Surface Organic Contaminant Detection Test (In-House Method)

3 Test Specimens

3.1 A bare printed wiring board or test coupon with a surface area of at least 35 cm².

3.2 The contaminated microscope slide used in the solvent extraction procedure defined in IPC-TM-650, Test Method 2.3.38.

4 Apparatus & Materials

4.1 An infrared spectrophotometer capable of scanning the from 2.5 µm [98.4 µin] to at least 15.0 µm [591 µin] range.

4.2 A multiple internal reflectance (MIR) attachment with a KRS-5 or ZnSe plate. Other techniques such as Attenuated Total Reflectance (ATR) or reflection absorption using micro FTIR can be used in lieu of the MIR techniques (see 6.10).

4.3 The test fluid is Spectro or High Pressure Liquid Chromatography (HPLC) grade acetonitrile. Other appropriate solvents may be used as agreed upon by user and vendor.

4.4 Disposable glass medicine dropper with rubber squeeze bulb or 2 ml capacity glass syringe.

5 Procedure

5.1 Clean an MIR plate by moistening a soft tissue with test fluid, then gently wiping the surface of the plate until all residues have been removed. Since the KRS-5 plate scratches easily, stubborn stains may be removed by ultrasonic cleaning in acetone.

5.2 Obtain the contaminated microscope slide specimen prepared in Test Method 2.3.38, or the printed board specimen.

5.3 Test

Hold the test specimen by the edges at an angle above the clean MIR plate. The specimen should not touch the plate.

5.3.1 Transfer the residue from the test specimen. Slowly drip 0.25 ml - 0.50 ml of test fluid onto the contaminated test specimen, allowing it to wash across the surface and drip onto the MIR plate (see Figure 1).

5.3.2 Evaporate the test fluid with a gentle stream of dry, oil-free air or nitrogen in a well-ventilated fume hood.

5.3.3 Place the MIR plate in the MIR attachment. Generate an infrared spectrum of the residue according to the instrument manufacturer’s recommended procedure. Remove and clean the MIR plate.
5.4 Evaporate the same amount of test fluid on clean MIR plate to obtain a control specimen.

5.5 Evaluation

5.5.1 Compare the test and control spectra for evidence of organic contamination.

5.5.2 The chemical class for the contaminant may be determined from the major bands in the spectrum in Table 1.

Table 1 Organic Contaminant Class Identification by Major Infrared Spectrum Bands

<table>
<thead>
<tr>
<th>Major Contaminant Class</th>
<th>Infrared Spectrum Bands (expressed in microns)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ether, Aliphatic</td>
<td>8.8-9.1</td>
</tr>
<tr>
<td>Ether, Aryl</td>
<td>7.8-8.0</td>
</tr>
<tr>
<td>Carboxylic Acid</td>
<td>3.2-4.1</td>
</tr>
<tr>
<td></td>
<td>5.8-5.9</td>
</tr>
<tr>
<td></td>
<td>6.9-7.1</td>
</tr>
<tr>
<td></td>
<td>10.4-10.9</td>
</tr>
<tr>
<td>Carboxylic Acid Salts</td>
<td>6.2-6.4</td>
</tr>
<tr>
<td></td>
<td>7.1-7.4</td>
</tr>
<tr>
<td>Ester</td>
<td>5.7-5.8</td>
</tr>
<tr>
<td></td>
<td>8.0-8.5</td>
</tr>
<tr>
<td>Amide</td>
<td>2.8-3.3</td>
</tr>
<tr>
<td></td>
<td>5.9-6.5</td>
</tr>
<tr>
<td>Nitrile (cyano)</td>
<td>4.4-4.5</td>
</tr>
<tr>
<td>Alcohol (includes hydroxyl glycols, polyols, etc.)</td>
<td>2.8-3.1</td>
</tr>
<tr>
<td></td>
<td>8.7-9.7</td>
</tr>
</tbody>
</table>

5.5.3 See Figures 3 and 4, comparative examples of spectrum graphics.

6 Notes

6.1 A Perkin Elmer Model 283 Infrared Spectrophotometer was used to develop this test method. Equivalent instruments from other manufacturers should be satisfactory if they have provision for a multiple internal reflectance (MIR) attachment.

6.2 This test may also be performed using Fourier Transform Infrared Spectroscopy (FTIR) instrumentation.

6.3 The test fluid Fisher Scientific Co. High Pressure Liquid Chromatography (HPLC) grade acetonitrile was used to develop this test method. Equivalent material from other suppliers may be used, provided no residue remains after evaporation as described in this test method. Other solvents may be required to dissolve specific residues.

6.4 Fisher Scientific Co. straight medicine droppers, catalog number 13-700, were used to develop this test. Equivalent droppers or disposable pipettes may be used.

6.5 The American Conference of Governmental and Industrial Hygienists has adopted a 40 ppm (v/v) Threshold Limit Value (TLV) for acetonitrile. It is recommended that the application and evaporation of acetonitrile be carried out in a well-ventilated fume hood. Rubber gloves and safety glasses should be provided for person(s) running the test.

6.6 Modified procedures permit detection and identification of contaminants residues containing carboxylic acid, carboxylic acid salts, ester, hydroxyl, amide, or nitrile (cyano) functional groups. For example, dicyandiamide (“dicy”), dehydroabietic acid, unpolymerized bisphenol-A type epoxy resins, resin and long chain amides have also been identified on printed wiring surfaces.

6.7 Although the test fluid is also capable of dissolving very small amounts of various inorganic compounds, their presence would generally be masked by the much higher levels of the organic contaminants.
6.8 Rapid evaporation of the acetonitrile must be avoided to prevent evaporative cooling of the MIR plate and subsequent moisture condensation from the air onto the plate. Application of sufficient heat to evaporate the water may volatilize part or all of the residue and invalidate the results. The present limit of detection of aryalkyl polyether residues by this method is 10 micrograms/cm².

6.9 The maximum organic surface contamination levels that will still permit reliable end-use operation of printed wiring assemblies of differing component densities and conductor line spacings have not been established for the various contaminants.

6.10 The present limit of detection can be easily extended by an order of magnitude using more sophisticated instrumentation and computer enhanced spectra (see Figures 3 and 4).

6.11 The KRS-5 plate is very toxic; it should be handled only with gloved hands, and should be polished with recommended polishing compound to minimize generation of hazardous dust.
Figure 4  Typical Spectrum Comparison