

## Method of Detecting Contamination

Minimizing contamination on a printed wiring board (PWB) is crucial to an assembly's performance and reliability. There are two classes of contaminants, ionic and non-ionic, that can be deposited during electronics manufacturing processes. The type of problem experienced by the customer determines the type of contaminant expected, which in turn determines the test method employed. Non-ionic contaminants are typically organic compounds, such as polymers, oils, or greases that are left on a printed circuit board (PCB) after production. An organic surface residue can cause problems with conformal coatings or may be an indication of an issue with the solder mask. Ionic contaminants can lead to corrosion and dendrite formation, both of which can cause electrical failures.

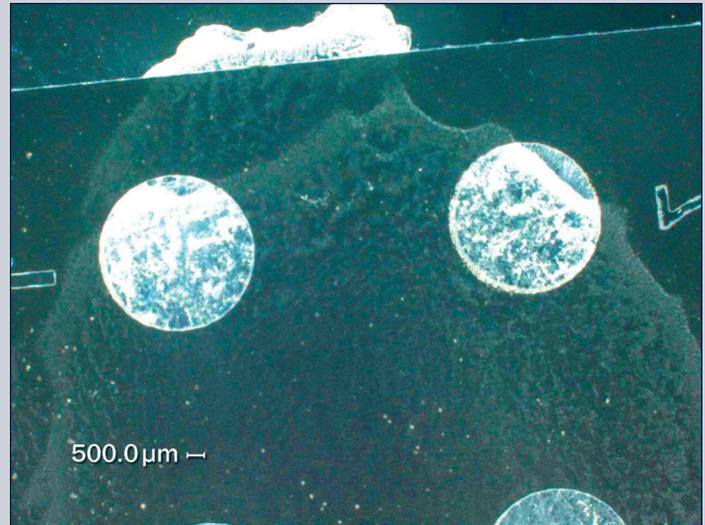
There are two IPC test methods for determining:

1. that an organic contaminant is present, in IPC-TM-650 2.3.38C, *Surface Organic Contaminant Detection Test*, and
2. the identity of the organic contaminant via Fourier Transform Infrared (FTIR) spectroscopy, in IPC-TM-650 2.3.39C, *Surface Organic Contaminant Identification Test (Infrared Analytical Method)*.

### *Surface Organic Contamination Detection Testing:*

In the "Surface Organic Contamination Detection Test," the PCB samples are rinsed in a dropwise fashion with 0.5 mL of acetonitrile onto precleaned aluminum coated glass slides. Using aluminum coated glass sample slides instead of glass-only slides reduces the background infrared absorption from the glass itself, increasing the signal to noise in the subsequent analysis. After the solvent has evaporated, more acetonitrile is rinsed over the PCBs and evaporated. This process is repeated until a total of 3.0 mL of acetonitrile has been deposited and evaporated from the slides. A control slide is used to verify that no residue was left by acetonitrile alone.

The slides are examined for visible signs of residue. When residue is observed, images of the slides are captured using a microscope and analyzed with the subsequent FTIR testing procedures. An example of



**Figure 1:** Image of residues obtained from rinsing a bare PCB.

residue observed with this method is shown in Figure 1.

### *Surface Organic Contamination Identification Testing:*

FTIR spectroscopy is a technique in which infrared energy is used to excite fundamental vibrational and associated rotational-vibrational modes of molecules in the mid-infrared, approximately 4000 to 400  $\text{cm}^{-1}$ . These vibrational modes correspond to molecular structures. Attenuated Total Reflectance (ATR) is a technique used with FTIR, which allows liquid and solid samples to be studied directly without further preparation. In ATR-FTIR, an infrared beam is directed through an optically dense crystal at a certain angle and internally reflects through the crystal, producing evanescent waves. When the crystal is pressed against an infrared active material, the infrared radiation from the evanescent waves, penetrates typically 1 to 4 micrometers into the sample.

In the "Surface Organic Contamination Identification Test (Infrared Analytical Method)," the slides with residues are examined with an ATR-FTIR microscope to attempt to identify the composition of the

residues. A background spectrum is run prior to capturing the spectra of all samples.

Figure 2 contains an example of an FTIR spectrum. The spectrum is then compared to an in-house commercial database and commercial database on the internet. Figure 3 shows that the FTIR spectra of the unknown residue and a known polyester resin adhesive compound closely match one another.

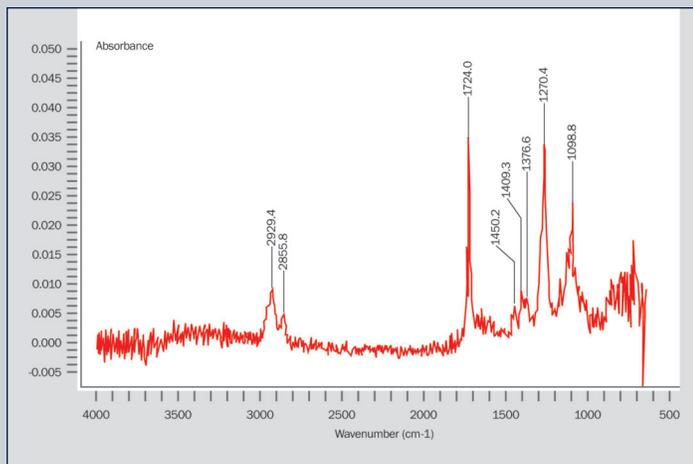


Figure 2: FTIR spectrum of residue rinsed from a PCB.

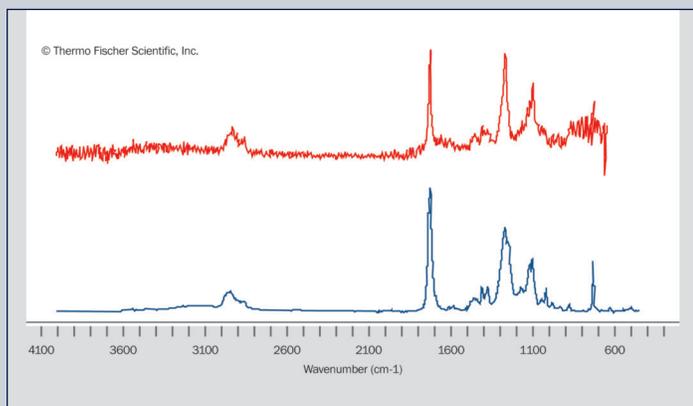


Figure 3: FTIR spectra compared for the residue from the PCB (top, red) with a known adhesive compound (bottom, blue).

If a known compound is not found to be a match, the peaks in the spectrum are identified by the frequency and intensity. Table 1 shows how the peaks correlate to chemical functional groups. From the information, it is suggested that the compound is an aliphatic ester with some ether functionality. This structural information agrees with the polyester resin result obtained from the spectral library search.

Frequency (cm <sup>-1</sup> )	Intensity	Peak Assignment
2929.4	Medium to Weak	CH <sub>2</sub> & CH <sub>3</sub> Asymmetric & Symmetric Stretch
2855.8	Weak	
1724.0	Strong	C=O Stretch (Ester)
1450.2	Weak	CH <sub>2</sub> & CH <sub>3</sub> Bend
1409.3	Weak	
1376.6	Weak	CH <sub>2</sub> Rock
1270.4	Strong	C-O Stretch (Ester)
1098.8	Medium	C-O Stretch (Ether)

Table 1: Infrared Absorption Frequencies and Chemical Identification

In the case of this surface organic contamination, the residue is suggested to be causing a problem with a conformal coating. The resolution is to clean the board with more rigorous process conditions, such as a stronger surfactant or solvent. Ionic residues though, can lead to degradation of the electrical conductors via corrosion and/or dendrite formation, which cause problems with reliability.

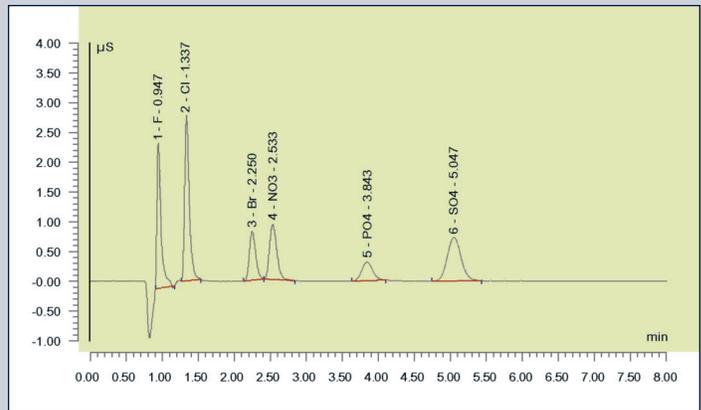
To determine if there are ionic contaminants present on the board, there are two methods that give different levels of information. The more general method follows IPC-TM-650 2.3.25C, *Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract (ROSE)*, in which a sample is immersed in an isothermal bath (at 35°C) containing a 3 to 1 solution of isopropyl alcohol to deionized water. Ionic contaminants extracted from the sample pass through a conductivity cell which continuously measures the conductivity of the solution. The conductivity values are integrated over the time of the extraction. The ionic material then passes through a deionization column before being recirculated back into the test chamber. As the ionic materials are extracted from the assemblies, the conductivity (and hence resistivity) of the solution will change dynamically until nearly all the extractable ionic material has been removed.

Results from dynamic extraction by Ionograph are reported in micrograms of NaCl equivalent per square inch. For assemblies soldered using rosin-based fluxes, the ionic cleanliness requirement per J-STD-001D is a maximum of 10.06 micrograms of NaCl equivalent per square inch (1.56 micrograms NaCl equivalent per square centimeter). There is no industry standard for acceptable bulk ionic levels, but it is better to have as low a value as possible. ACI recommends Ionograph results to not exceed a level of 2 to 3 micrograms of NaCl equivalent per square inch.

The more specific method of ionic contaminant analysis is IPC-TM-650 2.3.28A, *Ionic Analysis of Circuit Boards, Ion Chromatography Method*, in which the samples are sealed in a KAPAK bag with a 3 to 1 solution of isopropyl alcohol to water and heated in an 80 °C water bath for 1 hour to extract any ionic residues. The extract solution is analyzed against known standards to confirm the presence of and quantify each of the following anions: fluoride, chloride, bromide, nitrate, phosphate, and sulfate in units of  $\mu\text{g}/\text{mL}$ . The surface area is calculated from the board dimensions and the final results are reported in  $\mu\text{g}/\text{in}^2$ .

ACI's maximum recommended amounts of fluoride, chloride, bromide, nitrate, and sulfate for bare boards are 2, 4, 5, 1, and 3  $\mu\text{g}/\text{in}^2$ , respectively. The recommended levels of ionic contamination for populated assemblies will depend upon the application. However, for typical component packages on FR-4 or a like substrate, the maximum recommended amounts of fluoride, chloride, bromide, nitrate, and sulfate are 2, 9, 15, 1 and 10  $\mu\text{g}/\text{in}^2$ , respectively. Both sets of acceptance criteria were developed from experience and in conjunction with industry leaders.

An example of an ion chromatograph is shown in Figure 4. The anions in solution are separated from one another by their different rates of interaction with the quaternary ammonium groups in an ion-exchange column. A set of standards is run with the samples to compare elution times (how long it takes for an anion to leave the ion-exchange column) and corresponds to the identity of the anion. The peaks correspond to different anions passing through an electrochemical detector, which measures changes in conductivity resulting from the flow of ions in solution when moving through an electric field. The level of conductivity is directly proportional to the concentration of the anion, which is calculated by integrating the area under the peaks.



**Figure 4: Example of an ion chromatograph, featuring a standard solution containing fluoride, chloride, bromide, nitrate, phosphate, and sulfate anions.**

ACI's facilities are well equipped to assist with cleanliness testing for both organic and ionic contamination, in addition to the manufacturing factory floor capabilities, where testing of different cleaning chemistries and techniques can be used to optimize cleaning methods.

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