

The Use of Ion Chromatography in the Printed Circuit Board Industry

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Abstract:

Cleanliness, specifically ionic cleanliness, is an extremely important aspect in the design, fabrication, and construction of a printed circuit assembly as the unsuccessful removal of these materials can cause havoc for the functionality of an assembly once it has been installed into its final application. The use of Ion Chromatography, in comparison to less detailed bulk conductivity/resistivity testing, simply provides better quantitative data about a sample's cleanliness which can help better understand the origin of detected "contaminants" and when used in combination with other analytical techniques, can shed significant light on process problems or even worse, application failures.

Introduction:

Ionic cleanliness is a crucial aspect in the production of a "reliable" printed circuit assembly (PCA). Small clearances and tight spaces are plentiful on the surface of a printed circuit board (PCB) and ionic contamination can find its way into many, many of these locations. If not limited in

no-clean applications or successfully removed in water or solvent-clean applications, ionic contaminants can cause major reliability issues.

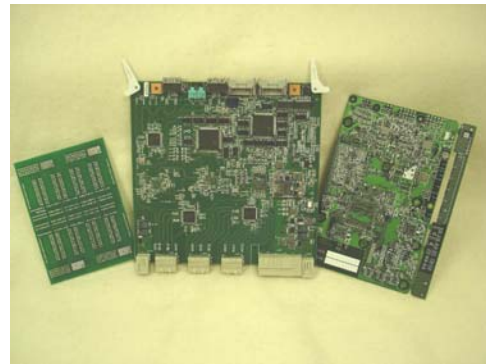


Image 1 – Representative photograph of printed circuit boards and assemblies

As a result, ionic cleanliness testing can be performed at various steps in the fabrication process to help qualify and quantify the presence of ionic contamination and help determine the possible sources of this contamination. Numerous test methods have been written and implemented over the years to help with this problem; however, as technology has advanced, ion chromatography (IC) has increasingly been used in this testing. Test methods have been developed by the military, the automotive industry, as well as, the printed circuit board industry itself to help manufacturers and their customers develop analytically "useful" tests for determining an assembly's cleanliness. This information can then be used in a proactive manner to help establish cleanliness guidelines and requirements that will hopefully help alleviate

application failures in the future. Methods are currently in place for analyzing everything from an unpopulated printed circuit board to fabrication materials such as surface mount adhesives and fluxes to the final assembled product.

Discussion:

Cleanliness is a concern in every step along the process of creating a product that contains printed circuit assemblies and as time passes and technology improves, the concern will grow. The reason that technological advances make cleanliness more important is because with fine-pitched components and small layouts for printed circuit boards, a smaller overall amount of actual contamination is required to cause problems.

The failure of a printed circuit assembly is usually detected by some kind of electrically based circuit not performing properly. Typically this electrical “failure” however has not been caused because of an electrical reason but because of a chemical or mechanical reason. For this discussion, we’ll focus on chemical reasons, which would include cleanliness and specifically ionic cleanliness.

Sources of contamination can come from anywhere and everywhere within the process of producing a printed circuit assembly. The raw materials used to construct the printed circuit board; laminates, glass fibers, resin, etc., must be “clean” initially or the problem will manifest itself from the start. During construction, the board must be cleaned after drilling, etching, etc., because the board’s surfaces must be “clean” before population with components. With that, the components themselves must also be

clean before placement on the board. This placement and attachment is typically completed using solder pastes and fluxes, which can leave organic and ionic residues on the surface of the board.

If the printed circuit assembly contains a significant amount of surface contamination, the product, upon power-up, might not function properly due to the presence of ionic residues in a “sensitive” area. These ionic and thus conductive residues reduce the dielectric spacing needed in a particular area causing an electrical issue in the form of decreased resistance in a specific circuit or an increase in voltage / current draw, possibly due to dendritic growth. Either way, the ionic contamination present has directly affected the performance of the assembly and most likely will not function as it was designed.

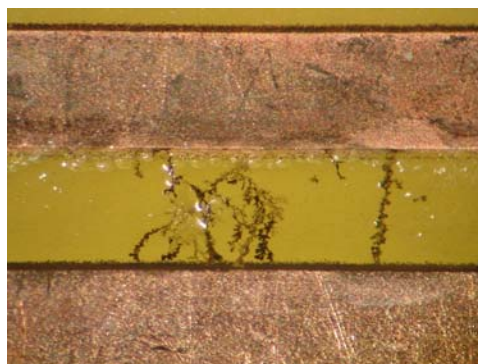


Image 2 – Dendritic growth due to contamination between parallel traces in the presence of opposing electrical polarities and moisture

The use of cleanliness testing can prevent this troubling issue from arising in the first place. If proper materials and cleaning procedures are used throughout the processing steps of a printed circuit assembly, along with appropriate ionic cleanliness testing, “failures” can



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hopefully be prevented or avoided. Testing for ionic cleanliness during manufacturing of an assembly can be a much more cost-effective endeavor than performing significantly more expensive root cause failure analysis testing once an assembly has made it into a product that is now failing in the field. Not to mention the cost of product recall and repair.

Cleanliness testing has evolved over the years from simple “bulk” conductivity type testing using a conductivity or resistivity meter to ion specific analysis using ion chromatography. “Bulk” conductivity test methods have been used for years and still are to this day. These methods are used to test PCB’s and PCA’s to evaluate them for overall cleanliness. Samples can be tested either in a “static” form or “dynamic” form. For “static” evaluations, the PCB / PCA sample is rinsed with solvent solution which is collected and then measured for conductivity. This conductance value is then converted to a sodium chloride (NaCl) equivalent per sample surface area ($\mu\text{g NaCl} / \text{cm}^2$) value by using a developed calibration curve along with the rinse volume and sample surface area used. In comparison, “dynamic” testing is performed in a similar way except that the PCB / PCA sample is continuously rinsed with solvent solution that is continuously monitored for conductance as a function of time. Once the conductance of the solution drops back to the initial or baseline value, the conductivity curve is integrated for a total “bulk” conductivity value, which is converted to the same “NaCl equivalent per surface area” value type described above.

Basically, the difference between these two methods is simply the fact that for the “static” test the sample is rinsed one time, while when using the “dynamic” version, the sample is rinsed until “clean”. Either way, the result found for both of these tests provides only a single value for cleanliness. If this value is believed to be too high for a PCB moving on to a population process or for reliable operation of a PCA, no specific information is provided to help in determining the source of the contamination. These “rinse” tests are generally referred to as Resistivity of Solvent Extract (ROSE) methods.

For ionic cleanliness testing using ion chromatography, PCB’s and PCA’s can be analyzed along with many other types of samples that alone can give more information about possible contaminating sources, such as the components attached to the PCA and even the attachment materials (solder pasts, fluxes, surface mount adhesive, etc.). More importantly however is that ion chromatography gives specific values for the levels of individual ions present as opposed to a single value for the overall cleanliness, which is the only information obtained when performing ROSE testing.

Methodology:

Ion chromatography based test methods for cleanliness have been published by different organizations in order to assess different areas of the industry. IPC, an organization focused mostly on the printed circuit board industry, the military, and Delphi Delco Automotive Systems, a group focused on testing for the automotive industry; have all written



documents for use by manufacturers, OEM's, and the like.

IPC has published two (2) methods under IPC-TM-650 to address the use of ion chromatography in testing PCB's, PCA's, fluxes, solder pastes, and cored solder wire. The first, test method 2.3.28, titled "Ionic Analysis of Circuit Boards, Ion Chromatography Method", uses a 75% isopropyl alcohol (IPA) / 25% deionized (DI) water solution for extraction of a PCB / PCA sample. The sample is placed, along with the extract solution, in a bag and then sealed and extracted in a water bath at 80°C for one (1) hour. After an hour, the sample is removed from the bag and the extract solution is analyzed using IC. Per this test method, the suggested ions to analyze for are: anions – bromide, chloride, fluoride, nitrate, nitrite, phosphate, and sulfate; cations – ammonium, calcium, lithium, magnesium, potassium, and sodium; weak organic acid anions – acetate, adipate, formate, glutamate, malate, methane sulfonate, succinate, and phthalate.

The second, test method 2.3.28.1, titled "Halide Content of Soldering Fluxes and Pastes", is used for determining ionic levels present in solder fluxes used to populate printed circuit boards. Procedures for the extraction of fluxes, solder pastes, and cored solder wire are described. For each, sample preparation techniques are given to extract the flux in a given sample using a 10% IPA / 90% DI water solution. The extract solution is then analyzed using ion chromatography for the presence of halides, specifically bromide, chloride, and fluoride.

In support of the microelectronics industry, the military has specified the use of ion chromatography for the testing of polymeric materials in microcircuit applications in MIL-STD-883, method 5011.4 titled "Evaluation and Acceptance Procedures for Polymeric Materials". Polymeric materials are used in both electrically conductive and electrically insulative applications. These materials are used to attach materials within microelectronic components, attach components to printed circuit boards, and encapsulate surfaces. Sample preparation procedures describe a DI water reflux extraction for twenty (20) hours. After extraction, the solutions are analyzed by ion chromatography, specifically for chloride, fluoride, potassium, and sodium content. Along with the ion chromatography analysis described in this method, additional pH and electrical conductance measurements are also obtained for qualification purposes.

Similar to IPC, Delphi Delco Engineering Systems also has two (2) ion chromatography based test methods that are to be used for electronic devices associated with automotive applications. Each of these methods, Q-1000-119 and Q-1000-127, are titled "Surface Ion Contamination" and the two (2) are designed to handle two (2) different groups of samples. The Q-1000-119 method is designed for testing accessories related to PCB's and PCA's such as housings, leads, connectors, components, etc. Also similar to IPC, the test samples are extracted in a water bath at 80°C while sealed in a bag but instead with a 10% IPA / 90% DI water solution as opposed to a 75% IPA / 25% DI water solution. Also contained within this method is a solvent



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extract test method similar to the ROSE methodology described above. The Q-1000-127 method is almost identical to the Q-1000-119 except that this method is specifically designed for testing PCB's and PCA's. Ion chromatography and ROSE based methods are also stated within this method as in the Q-1000-119 method.

Overall, the five (5) test methods described above from IPC, the military, and Delphi Delco Automotive Systems, summarize the test methods available for ionic cleanliness testing within the printed circuit board industry which call for the use of ion chromatography. These documents are test methods and do not specifically address the idea of ionic level requirements, however there are some exceptions. Specifications are typically written to provide requirements for a test sample and would then reference a specific test method in which to analyze the given sample. Additionally, manufacturers and OEM's may specify certain ionic level requirements based on the processing involved in the creation of the product as well as on the application in which the product will be used; for example, aerospace, automotive, consumer, medical, military, etc. From a reliability standpoint, some of these applications may not need as strict of a cleanliness level as others, for example, consumer products vs. military hardware.

Observations:

Ion chromatography can be used as an analytical tool for "proactive" or "reactive" investigation of a product's ionic cleanliness. The decision on which approach to use is up for debate, however,

the "proactive" approach would seem to be potentially less expensive.

For "proactive" ionic cleanliness testing, manufacturers can simply institute some form of quality control process step. Daily, weekly, or monthly sample pulls can be obtained from different steps in the PCA construction process, in which through testing, the manufacturer can gain useful information about their process and whether the cleaning steps designed into the process are performing adequately. This "spot" check style should be extremely useful in determining a problem once it starts and potentially halting the production of numerous possibly contaminated products. Additionally, from a product conformance standpoint, ionic cleanliness testing can simply be included as part of the product's qualification program. With this, theoretically no product would make it out of the factory without at least one check for ionic cleanliness.

Without ionic cleanliness testing as part of a quality control program, testing of a much different nature could be needed if failures occur in the field during product use. The failure of product in the field is an expensive possibility not just due to the testing that will need to be performed to determine a root cause of failure but also because of the need to physically recall product and the possible loss of business due to customer inconvenience. Strictly from a testing standpoint, the costs associated with root cause failure analysis type testing will most likely be a more expensive and lengthy process than simply performing ion chromatography cleanliness testing during the manufacturing process.



Conclusions:

The use of ion chromatography as an analysis tool in the printed circuit board industry can provide a significant amount of information about a product's cleanliness as well as, a process's ability to clean such product. Simple questions such as: "Is my product clean?", "How clean is my product?", and "Is my product getting cleaner?"; can all be answered by using ion chromatography. The answering of these questions by ion chromatography would be a "proactive" type analysis, while the use of ion chromatography for "reactive" analysis is also possible to answer some different questions, such as: "Why did I fail?" and "What is the source of the detected contamination?".

Analysis using ion chromatography, as opposed to ROSE-style ionic cleanliness testing, gives specific ion results that can, most importantly, be used to identify sources of contamination. If samples meet specified requirements for cleanliness during processing then no problems are present and the product can move on, however, if "high" ionic levels are present, then determining the source of the "high" levels is paramount. ROSE style ionic analysis sheds no light on this predicament while testing via ion chromatography can. The presence of certain specific ionic species at elevated levels can indicate specific possible sources of contamination.

Residues from acid etches, residues from fluxes, handling issues, and contaminated rinses, along with other aspect of a production process, can all be sources of contaminants and analysis by ion chromatography can shed light on these sources. By obtaining specific ion data, adjustments can be made to process steps to help decrease the ionic levels present on a product.

Ultimately, as technological advances and PCA applications become more "micro" and even "nano", the benefits of using ion chromatography analysis will increase due to its ability to detect the presence of ionic species at ppb levels. This low of a detection limit is critical in smaller applications because with smaller dielectric spacings, less contamination is needed to cause electrical problems.

Ion chromatography's use in cleanliness testing for the printed circuit board industry has increased over the years as more members of the industry become aware of its benefits. Uses and sample preparation techniques are sure to change and adapt over time, but ion chromatography's use in the industry is here to stay as the benefits of the testing have already helped numerous manufacturers solve previously unknown contamination problems.

For more information concerning these topics or any other testing needs, please contact Trace Laboratories – East at (410) 584-9099 (traceeast@tracelabs.com). Visit us on the web at www.tracelabs.com.