



A Comparison of Three Ionic Test Methods

Comparison of Ionic Contamination Test Methods To
Determine Their Ability to Reliably Predict Performance Risks

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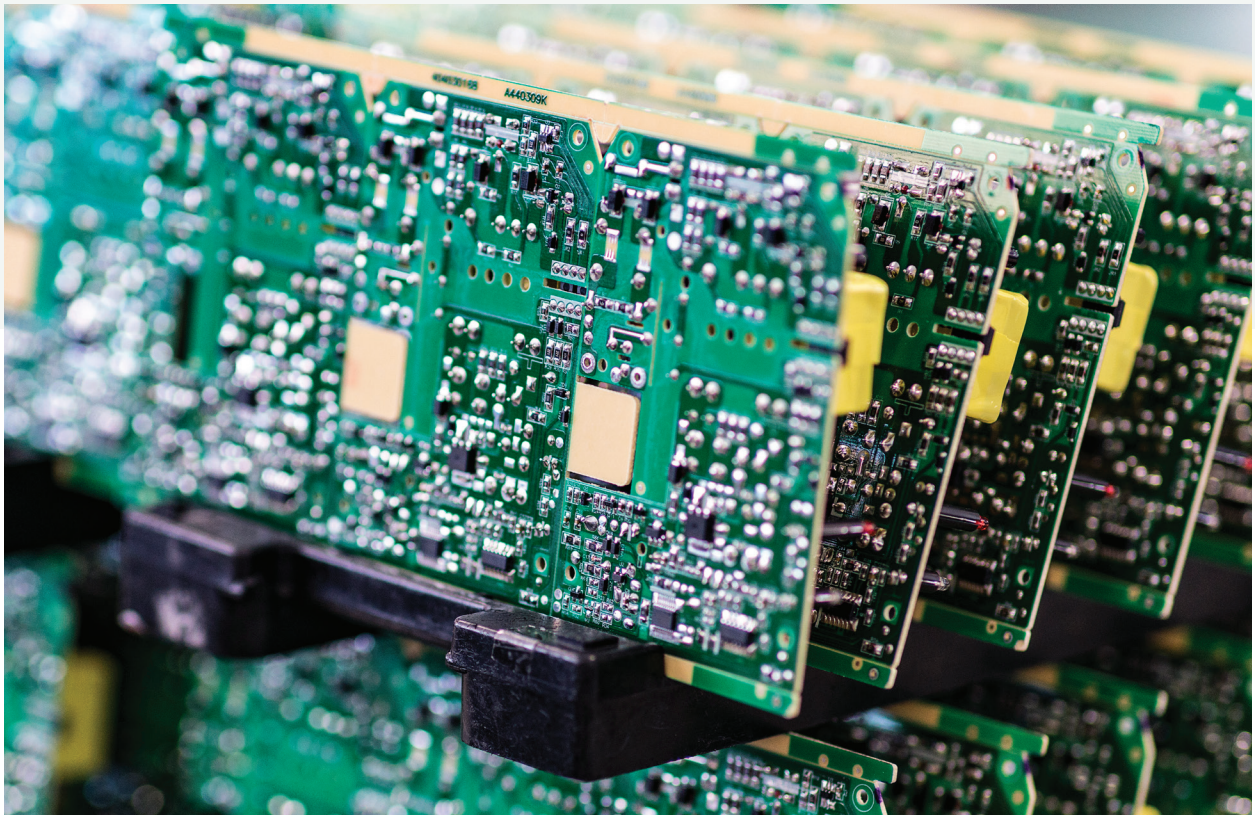
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ABSTRACT

In the Electronic Manufacturing Services Industry, one of the known failure mechanisms is caused by the presence of ionic contamination. Ionic contamination leads to electrochemical migration and dendritic growth. Through the years, there have been various methods employed to verify the ionic cleanliness of electronic components, printed circuit boards and their assemblies. The accepted industry standard test is Surface Insulation Resistance, SIR, testing. Although this is recognized as the test of merit, it can take weeks to prepare the test vehicles, run the test and analyze the results. By the time this is complete, the product has shipped. If failures are found in test, it is too late to be of practical use. The industry is looking for a test which can be run quickly and is representative of the product currently in production so that decisions, and potential corrective actions can be implemented prior to shipping product to customers.

One alternative method is Resistivity of Solvent Extract, ROSE,¹ testing. This method is primarily aimed at product which has gone through a cleaning process just prior to the test. However, in printed circuit board assemblies, PCBAs, there has been a shift from water wash fluxes followed by cleaning, to a process that utilizes no-clean flux with no cleaning. If ROSE testing is used in conjunction with no-clean flux, it often will lead to false fails, because no-clean fluxes are known to contain ionic residues.

Another more recent test is the Critical Cleanliness Control, C3,² test, which is designed to test specific regions on a PCBA that may be prone to ionic contamination related failures. In this paper, a direct comparison will be made between ROSE testing, SIR testing and C3 testing. The test results will be augmented by a detailed visual inspection, Ion Chromatography, IC, testing, and other tests, as required.

Key Words: No-clean, Flux, Process, Fails, SIR, ROSE, C3 and IC

Introduction:

The purpose of this paper is to explore alternate, accelerated test options to determine if the ionics present on PCBA's are deleterious to the projected field life of the product. The respected industry standard test is the SIR test.³ Due to product design and limitations, SIR testing is rarely performed on actual product, instead it is performed on test vehicles, TV's. The TV's are subjected to accelerating parameters, in an attempt push any excessive ionics to fail.

Another industry standard test is ROSE testing. In this test the PCBA is immersed in a solution designed to dissolve any ionic materials which may be present, quantify the total ions in solution, and calculate the number of ions per area of the PCBA or targeted components. ROSE testing has limited potential application in the testing of no-clean flux.

A relatively new test is C3, localized ionics testing. The localized ionics tester is designed to test a specific confined region for the amount of mobile ions which may be present.⁴ In this method, high risk regions can be specifically tested, and TV's or actual PCBA's can be tested. This test is calibrated to test no-clean flux, although it could easily test water wash flux as well.

This paper will compare these three analysis techniques, noting the advantages and disadvantages of each test method.

Test Vehicle:

The test vehicle contains 16 different SIR test site conditions and one breakaway QFP test site. The SIR test site conditions are described in table 1. Figure 1 also shows the top side of the TV.

Table 1: Test Vehicle Test Site Description

Pin Number	Test Site Description
1	QFN's
2	Header
3	Header
4	Header
5	PQFP, Outer Ring

6	Under PQFP
7	B24 Comb
8	LCC, Outer Ring
9	BGA
10	Under LCC
11	B24 Comb
12	B24 Comb
13	No Part
14	No Part
15	DIP Leads
16	Y-Pattern

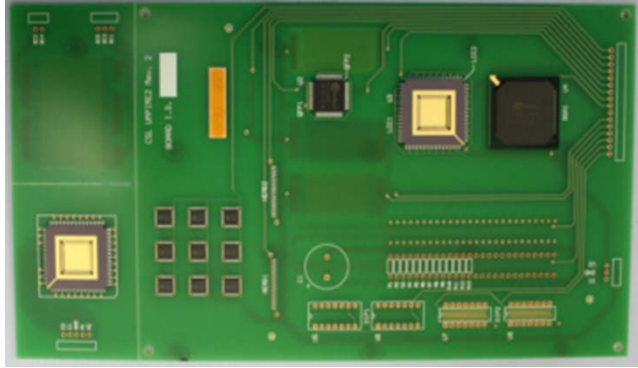


Figure 1: TV Topside Layout

Test Plan:

The TV assemblies were all built by one contract manufacturer. To increase variability of the analysis, the assemblies were built according to table 2. Three different types of solder paste were used, creating 3 different sample groups. In addition, there was variability in the fluxes used for plated through holes and rework sites. Group A was analyzed by Foresite Inc, while Group C was analyzed by IBM Rochester. Group B was held back for potential regression analysis, if necessary. The TV assemblies and breakaway coupons were used to compare different ionic residue testing methods (SIR, Local Ionics Testing and ROSE testing). There were 11 assemblies in each group. Each sample was subjected to at least one ionic contamination test method, except for samples “A-11” and “C-11” which were held as control samples.

Table 2: Test Plan

Card Group	Type of Flux (No-Clean Fluxes only)		
	SMT Lead free SAC 305	PTH SAC 305	Rework, No Solder
Group A	Solder paste A	Flux D	Flux E
Group B	Solder paste B	Flux D	Flux E
Group C	Solder paste C	Flux D	Flux E

The processing, treatment, and testing conditions for each Group A Assembly are outlined in Tables 3, 4, and 5.

Table 3: Assembly Build Parameters and Group A Test Conditions

Pin	Primary solder	Rework if done	What is measured	Localized Cleaning
1	Solder Paste Reflow	None	Ground to pads	None
2	Kiss (solder fountain)	None	SMT pads with flux	None
3	Hand Solder	Yes liquid flux iron	SMT pads with flux	IPA and a Brush
4	Control (Bare Board Conditions)	None	SMT pads no flux	None
5	Solder Paste Primary Reflow	None	SMT pad to pad	None
6	Solder Paste Primary Reflow	None	Flux entrapment of QFP	IPA and a Brush
7	Hand Solder	Yes liquid flux iron	20 mil spaced comb	None
8	Solder Paste Primary Reflow	None	SMT pad to pad	IPA and a Brush
9	Solder Paste Primary Reflow	Yes IR Rework liquid	Ball to ball	None
10	Solder Paste Primary Reflow	None	Flux entrap of LCC	None
11	None	None	20 mil spaced comb	None
12	Hand Solder	Yes Liquid flux iron	20 mil spaced comb	IPA and a Brush
13	Kiss (solder fountain)	None	Bottom side SMT	None
14	Kiss (solder fountain)	None	Bottom side SMT	IPA and a Brush
15	Kiss (solder fountain)	None	Trapped flux under DIP	None
16	Kiss (solder fountain)	None	PTH Lead to lead	None
3 & 16	Kiss (solder fountain)	None	None	Sap / steam cleaned

Table 4: Group A Breakaway Coupon Build Conditions

Breakaway LCC and comb	Primary Reflow Solder paste	None	ROSE	IPA and a Brush
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Table 5: Group A Test Conditions

Pin	SIR 40/90	Additional Testing	
1	SIR testing	Not Tested	
2	SIR testing	LOCAL IONICS	IC Testing
3	SIR testing	LOCAL IONICS	IC Testing
4	SIR testing	LOCAL IONICS	IC Testing
5	SIR testing	LOCAL IONICS	IC Testing
6	SIR testing	Not Tested	
7	SIR testing	Not Tested	
8	SIR testing	LOCAL IONICS	IC Testing
9	SIR testing	LOCAL IONICS	IC Testing
10	SIR testing	LOCAL IONICS	IC Testing
11	SIR testing	Not Tested	
12	SIR testing	LOCAL IONICS	IC Testing
13	SIR testing	LOCAL IONICS	IC Testing
14	SIR testing	LOCAL IONICS	IC Testing
15	SIR testing	Not Tested	
16	SIR testing	LOCAL IONICS	IC Testing
3 & 16	Not Tested (Breakaway Site)	Not Tested (Breakaway Site)	

The analysis treatment combinations for Group C Assemblies can be seen in table 6 and table 7, where table 6 has the main TV and table 7 the breakaway coupons which were removed from the main assemblies.

Surface Insulation Resistance (SIR) Test:

A standard SIR test was used to test the cleanliness of the TV assemblies. Eight Group A test vehicles were subjected to this test at a temperature of $40^{\circ} \pm 2^{\circ}$ C and a relative humidity of $90.0 \pm 3.0\%$, while eight Group C test vehicles were subjected to this test at a temperature of $50^{\circ} \pm 2^{\circ}$ C and a relative humidity of $80.0 \pm 3.0\%$. The targeted locations had a maximum of 15 V bias limited by a 1.0 M Ω resistor. An initial insulation resistance reading was taken at ambient conditions, then under test conditions at times 0, 24, 50, 100, 150, 200, 250 and 300 hours. Additionally, a

final measurement was taken after the samples had returned to ambient conditions.

Table 6: Group C Treatment by Assembly

Board	SIR	Local Ionics	ROSE	IC, 10 locations
C-1	✓	✓		✓
C-2	✓	✓		
C-3	✓	✓		
C-4	✓			
C-5	✓			
C-6	✓			
C-7	✓			
C-8	✓			
C-9		✓		✓
C-10			✓	
C-11	Control			

Table 7: Group C Breakaway Coupon Test Plan

Board	Local Ionics	ROSE	IC, 1 Location
C-1		✓	
C-2		✓	
C-3		✓	
C-4		✓	
C-5			
C-6			
C-7			
C-8	✓		
C-9	✓		✓
C-10		✓	
C-11	Control		

The threshold for card assemblies is 10 M Ω ($1.0 \times 10^7 \Omega$); if the in-situ reading is below 10 M Ω , visual inspection is required. Visual inspection is necessary to determine if any dendritic growth or electrochemical migration occurred.

Local Ionics Test:

An electronics cleanliness tester was used to measure ionic contamination and provide an indication of local spot cleanliness. This local ionics test was investigated as an alternative or supplement to the traditional SIR testing. Removal of ionic residue is achieved through a heated delivery system with 3 stages:

1. Heat the extraction solution and deliver to the extraction site.
2. Soak to allow dissolution of ionic species.
3. Aspirate the solution into a collection cell.

The process has nine cycles to ensure full removal of the residue. The test is performed over an area of approximately 0.1 in². The system produces an output of “clean” or “dirty” which provides an indication of the localized cleanliness. An electrode, immersed in the extracted solution, measures the electrical conductivity. A 10 volt bias is applied in order to measure the leakage current. The cleanliness is determined by a parameter of 500 μ A and 120 seconds. If

the current though the eluent exceeds 500µA within 120 seconds, the localized region is considered dirty. The time parameter for cleanliness is based off of the theory that corrosive ionics will cause leakage more rapidly, whereas, insulative species cause leakage after a longer time duration⁸.

Table 8: Results of Local Ionics Test vs SIR Test for Group C

Card	Pin	Local Ionics	SIR
C-1	1	P	P
	2	F	F
	3	P	SHORT*
	4	P	P
	5	P	P
	7	P	P
	8	F	F
	9	F	P
	13	P	P
	14	P	P
	15	F	F
16	F	F	
C-2	1	F	P
	2	P	P
	3	P	P
	4	P	P
	5	P	P
	7	F	P
	8	F	p
	9	P	P
	13	F	P
14	F	P	
C-3	1	P	P
	2	F	P
	3	P	P
	4	P	P
	5	P	P
	7	F	SHORT*
	8	F	F
	9	F	P
	13	P	P
14	P	P	

*Note: Solder Short from the initial assembly.

The electrode and cartridge were changed between each test to prevent contamination. Ten different locations were tested on each TV assembly, as well as one location on each breakaway coupon. Multiple locations were tested to indicate localized cleanliness on a variety of component types. A summary of the localized extraction and SIR results are presented below in table 8.

Table 9: Correlation of Local Ionics and SIR Test; Pass/Pass, Pass/Fail, Fail/Pass & Fail/Fail

PP	PF	FP	FF
17	0	7	5
PP+FF	PF+FP		
76%	24%		

The results above indicate that localized ionics and SIR testing have a 76% correlation when both tests have a matching pass or fail result.

Resistivity of Solvent Extract (ROSE) Testing:

Card ionics testing was performed with an Alpha Metals Omegameter 600 SMD. The apparatus employs a heated isopropyl alcohol (75%) and deionized water (25%) solution with an aggressive spray action in order to remove and quantify ionics from the samples being tested. Raw printed circuit boards are generally extracted for 10 minutes, while printed circuit board assemblies are typically tested for 15 minutes. Ionic results are reported as “micrograms of NaCl equivalent per square inch”. However, the ionic species present on the samples cannot be identified as an output of ROSE testing. ROSE testing differs from local ionics testing, because this method tests cleanliness of the entire board and assumes a uniform distribution of ionics where local ionics testing, as the title implies, tests a specific location.

Prior to each usage of this instrument, a calibration test run was performed to verify that the system was functioning properly. The ROSE testing results are presented in table 10 below.

Table 10: ROSE Test Results of Group C

Sample	Omegameter Result (µg NaCl eq./in ²)*	Comments:
Calibration	3.8, 3.9	Calibration reading needs to be between 3.4-3.9 to indicate proper performance.
The TV Assembly: C-10	14.2	7000 mL extraction solution
Breakaway Coupon: C-10	59.0	4.5 in ² surface area (only including area of LCC), 15 minutes test time, 4500 mL extraction solution
Breakaway Coupon: C-1	44.5	

Breakaway Coupon: C-2	35.3	
Breakaway Coupon: C-3	42.8	
Breakaway Coupon: C-4	44.0	

*Note: IPC Standard: <6 µg NaCl eq./in² ⁵

All of the ROSE samples tested failed. All of the readings were significantly over the IPC standard upper limit. Yet, there is nothing unusual pertaining to these sites. The issue lies with the fact that no-clean fluxes leave ionic residues, causing them to fail ROSE testing. These residues, when properly processed, are safe⁶ because the ions are sufficiently immobilized such that there is no reliability issue. ROSE testing also provides no indication of the residue's chemical reliability⁷. For these reasons, ROSE testing is generally not applicable to no-clean flux residues, the ions in no-clean flux are not eliminated, they are just immobilized.

SIR vs Local Ionics Testing:

SIR and local ionics testing have common elements in that they both test the conductivity of ionic residue. However, with SIR, in order for the test to be meaningful, the residues must be between comb patterns or pads. SIR testing is limited to these predetermined locations and requires subjecting the samples to 168 hours, or more, of electrical biasing under elevated temperature and humidity. The SIR accelerating conditions may cause a phase change of the residues bringing in to question the outcome of the test. SIR testing requires the residue to absorb the test humidity. Then, with a 15 Volt bias, assess the surface insulation resistance of the residue. The local ionics test can assess any specific spot on either a test board or production board. The test creates a mini-chamber over an isolated area and provides heated DI water, 18 MΩ, 3x polished, to the board surface and allows it to dissolve the surface residue. This solubilizing effect of the residues will bring ionic and organic material into solution similarly to the high humidity effect. The local ionics extraction deposits drops 75 µl of hot DI water at a time and allows it to soak for 20 seconds. The test then repeats 9 times to collect a total of 2.2 ml of dissolved residue in DI water.

The major advantage of the local method is that it can be tested on any board in order to assess the residues of the top and bottom SMT processes, the bare board residues, the wave or selective wave residues on the surface of direct contact area and SMT, or top and bottom via areas next to the selective wave area where the flux is not contacted by the solder directly.

Ion Chromatography Test:

Ion chromatography (IC) was used to detect the presence of several cations and anions. The eluent was removed from the Local Ionics Tester and run through IC. IC performed both qualitative and quantitative analysis for 12 ionic species. IC is capable of detecting the presence of cations and anions on a part-per-billion scale⁸. The quantity was compared to established limits in µg/in². Table 11 details which anions and cations were present and which were over the proposed limits on assemblies A-1 through A-5. IC limits differ from ROSE testing limits; IC limits represent the amount of ions in a spot area, selected and tested specifically for the presence of ions which may conduct sufficient current to facilitate the growth of electrochemical migration or dendrites. On the other hand, the ion limits in ROSE testing are limits averaged over the entire PCBA surface area, whether there is flux, other conductive materials present, or not..

Table 11: Ion Chromatography Results of Group A

Ion	Present	Limit: µg/sq. in.	Over the High Limit
Fluoride	No	1	No
Acetate	Yes	3	Yes
Formate	Yes	3	No
Chloride	Yes	3.0	No
Nitrite	No	3	No
Bromide	Yes	6.0	No
Nitrate	Yes	3	No
Phosphate	Yes	3	No
Sulfate	Yes	3.0	No
Weak Organic Acid	Yes	25	Yes
Methyl Sulfonic Acid	No	1	No
Lithium	No	3	No
Sodium	Yes	3	Yes
Ammonium	Yes	3	Yes
Potassium	No	3	No
Magnesium	Yes	n/a	na
Calcium	Yes	n/a	na

There was 100% correlation between ions considered over the maximum limit and whether the Local Ionics test failed. Table 12 below provides a comparison between anion and cation limits along with localized ionics test results. Table 12 only lists the ions which were over the proposed ion limits.

Table 12: Correlation; Ion Chromatography vs Local Ionics Testing on Assemblies A-1 to A-5

Ions, all values in mg/in ²				Local Ionics Tester Results
Acetate	WOA	Sodium	Ammonium	
3*	25*	3*	3*	
2.07	22.13	2.41	0.26	Clean
2.87	20.09	2.69	0.35	Clean
4.06	51.63	7.89	3.62	Dirty
4.95	63.25	11.41	3.65	Dirty
5.69	74.54	8.54	2.11	Dirty
2.54	11.24	1.62	1.04	Clean
1.62	9.98	1.25	0.65	Clean
1.35	12.04	1.63	0.29	Clean
6.71	35.98	9.14	2.14	Dirty
1.62	11.32	1.54	1.66	Clean
1.94	7.98	1.25	0.65	Clean
2.84	21.04	2.41	1.21	Clean
2.69	22.67	2.65	1.30	Clean
11.24	34.71	11.42	5.64	Dirty
19.98	51.42	27.45	11.09	Dirty
12.35	39.91	18.54	5.14	Dirty
1.54	11.14	1.25	0.64	Clean
1.33	15.24	1.33	0.92	Clean
1.54	12.11	1.61	0.54	Clean
10.07	48.21	14.24	6.32	Dirty
0.87	10.19	1.07	0.24	Clean
1.62	11.04	1.51	0.65	Clean
1.88	19.98	2.04	0.54	Clean
1.97	20.04	2.65	1.21	Clean
21.35	39.27	16.35	4.98	Dirty
23.65	44.51	26.27	5.38	Dirty
15.94	31.30	20.15	4.55	Dirty
1.76	12.14	1.21	0.69	Clean
1.14	9.98	1.36	1.79	Clean
1.65	13.25	1.29	0.69	Clean
20.04	41.08	19.21	4.21	Dirty
1.04	11.74	2.11	0.36	Clean
1.05	9.54	1.13	0.65	Clean

1.91	20.67	2.58	0.98	Clean
2.57	22.15	2.67	1.65	Clean
27.31	63.54	33.26	5.24	Dirty
31.65	77.07	38.65	6.36	Dirty
18.84	42.95	21.95	10.97	Dirty
1.07	11.25	1.25	0.88	Clean
0.98	13.25	1.62	0.65	Clean
1.31	12.36	2.05	0.98	Clean
20.15	53.21	29.65	3.21	Dirty
1.41	13.67	1.21	0.81	Clean
1.05	8.91	1.25	0.68	Clean
0.95	18.24	2.51	1.37	Clean
0.64	21.31	2.19	1.22	Clean
41.05	61.25	30.21	4.65	Dirty
53.27	74.65	38.21	5.24	Dirty
20.95	37.21	20.67	3.51	Dirty
0.92	10.34	2.15	0.98	Clean
1.21	12.65	2.19	0.57	Clean
1.12	19.25	1.54	0.95	Clean
21.54	38.51	21.36	2.88	Dirty
0.49	12.04	1.46	0.44	Clean

*Note: Proposed Limits for those specific ions.

Summary:

Printed Circuit Board Assembly Test Vehicles were built with a variety of solder pastes, rework fluxes and several different treatment combinations. The treatment combinations include sites that were subjected to rework and component attachment with several different component types, or sites with no rework or components. These groups of TV's were analyzed in two different locations, Rochester, Minnesota and Kokomo, Indiana. The results from each site were compared. Where the Rochester team compared SIR and ROSE testing to Local Ionics Testing, the Kokomo team compared the results from IC to Local Ionics testing. The findings from each site are comparable:

1. ROSE testing is not calibrated to perform ionic cleanliness testing on PCBA's assembled with no-clean solder pastes and fluxes. Localized ionic contamination tests is a suitable process control tool for no-clean solder pastes and fluxes⁷.
2. There was a 76% correlation between SIR test results and Local Ionics test results when both the pass/pass and fail/fail categories are combined. Local Ionics Test tends to be slightly more conservative in calling out fails that SIR testing does not fail.
3. IC testing on the eluent from the Local Ionics testing compared to Local Ionics test results

directly on the TV's correlate 100%. These results are 100% correlated when the IC calculation of ions per square inch is compared with whether or not 500 micro-amps is conducted through the collected eluent within 120 seconds.

4. SIR test almost always requires test vehicles to obtain meaningful results. Local Ionics Testing can be performed on either test vehicles or actual product assemblies.
5. SIR test takes 2-4 weeks, or longer, to prepare test vehicles, run them through the test conditions and analyze results. Local Ionics Test has the advantage that it takes minutes to perform the test and obtain meaningful results.
6. Local Ionics Testing is a serial test. Each location must be tested individually. SIR testing tests all of the test locations concurrently.
7. SIR testing requires opposing comb patterns to be a meaningful test.
8. Local Ionics Testing can test any spot location on the board, as long as the location is flat. Sometimes components need to be removed to use the Local Ionics Test.
9. Both tests provide go/no-go information as well as parametric information, if further evaluation is required.

¹ IPC TM-650, 2.3.25 Rev, D, "Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract (ROSE)," IPC Press, IPC Press, Bannockburn, IL.

² C3 is a registered trademark of the Foresite Company. Kokomo, Indiana.

³ IPC 9201A, "Surface Insulation Resistance Handbook." IPC Press, 2007, Bannockburn, IL.

⁴ Isaacs, P., Kobeda, E. and Zhang, J. "The Use of No-Clean Flux." 2015 SMTA Pan Pacific Conference Proceedings.

References:

Where SIR testing and Local Ionics testing may not be interchangeable tests, both tests are shown to provide a representative analysis of whether or not the condition under test may fail in the field. SIR Test has years of history and data behind it. In this study it was found that Local Ionics Test compares very well to SIR test results.

Acknowledgements:

The team acknowledges and thanks the expertise and work provided by the Rochester and Tucson IBM Materials and Processing Engineering Laboratories.

⁵ IPC J-STD 001, 8.3.6 "Requirements for Soldered Electrical and Electronic Assemblies," IPC Press, 2000, Bannockburn, IL.

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⁷ Interflux Electronics Technical Info Sheet, "Ionic Contamination Testing in a No-Clean Soldering Process."

⁸ Tam Tran Nguyen, M., "Reliability Assessment of Ion Contamination Residues on Printed Circuit Board," University of Florida Scholar Commons, 2013.