# **PWB Contamination & Reliability DOE**

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#### **EXECUTIVE OVERVIEW**

#### Background

When searching for common industry-accepted test methods to determine the cleanliness of printed wiring boards and assemblies, most manufacturer's turn to IPC (Association Connecting Electronic Industries) for guidance. It is common to use IPC-A-610 (Acceptability of Electronic Assemblies) as well as IPC TM 650 (Test Methods of Electronic Assemblies) and it's methods to benchmark the cleanliness or contamination of a process and product. Unfortunately, a chart listing contamination levels for a specific flux and process does not exist.

Contamination can lead to electromigration risk, dendrite growth and subsequent product reliability issues. There are several common, accepted techniques (R.O.S.E. (Resistivity of Solvent Extract)) and modified R.O.S.E. test, SIR test, Ion Chromatography, water-drop-across-trace test, and others). However, there is little consensus as to which test is the best measure of PWB or assembly contamination or cleanliness as well as the test result correlation to long-term reliability.

In the 1980s, prior to the Montreal Protocol and subsequent phase-out of CFCs, manufacturers could rely on CFCs and vapor phase reflow to meet soldering requirements. The eventual move away from RMA fluxes, replacement of vapor phase soldering with reflow and the continual miniaturization of electronics has made the focus on contamination and its link to product reliability more important than ever.

Dozens of pastes, fluxes, and cleaning materials, as well as continued miniaturization of packaging and designs further complicate matters.

#### Goals

This report hopes to achieve several goals:

- 1. Determine the link between bare PWB contamination, soldered assembly contamination and long-term product reliability.
- 2. Establish measurable limits for bare PWB cleanliness as well as process control limits for both an aqueous as well as a water-soluble soldering process.
- 3. Determine whether there is any correlation between common, industry-accepted rose/modified rose (omegameter/ionograph type) testing and long term product reliability.
- 4. Determine the effect PWB plating finish (HASL, Immersion Silver and Cu OSP) has on both bare PWB contamination as well as soldered assembly in a no clean and water soluble process.

- 5. Determine whether there is a link between percentage of saponifier used to wash soldered assemblies and long-term reliability.
- 6. Establish a more cost-effective test for manufacturers to use as a process-monitoring tool.

#### **TEST BOARD DESIGN**

Design a board containing a mix of PTH and SMT (topside and bottom side), containing several SIR test patterns as well as some water-drop-test trace patterns. This would provide the vehicle to test bare PWB, total assembly as well as specific site testing for contamination.

The Umpire Test Board, provided by Terry Munson of CSL is shown in Figures 1 and 2. The Umpire Board BOM is shown in Table 1.

#### WHY THESE COMPONENTS

The ZIF socket is included because research has shown that this component often entraps water in an aqueous cleaning operation and flux residues in a no-clean assembly operation. The DIP patterns are for use by through-hole only manufacturers. Four of the six test patterns look for pin-topin resistance degradation. The remaining two patterns look for the presence of flux residues under DIP components.

The LCC, QFP, and BGA patterns each have an area with solder mask and an area without solder mask to allow for various comparisons and to test interactions between pastes and fluxes and solder masks. The LCCs and QFPs have alternating daisy-chained mounting pads to examine the effects of paste flux residues. Identical patterns exist with and without solder mask to test for interactions.

The LCCs and the QFPs also have a comb pattern under the center of the components to test for flux residues under components. The LCC layout is the same as the IPC-B-36 standard test assembly (68 I/O LCC).

A large square of metal is included on the top to do solderability testing, such as SERA. The voltage supply and current-return lands are separated for better SIR performance. The inputs and outputs to the board are made via shroud connectors, which can, themselves serve as flux entrapment sites, as can PWAs.

The solder side of the board contains a Bellcore 25/50 test pattern with the striped mask to demonstrate Bellcore compliance and flux/solder mask interactions. Similarly, the boards contain several half-size IPC-B-24 test comb patterns for comparison to J-STD-004 test data. One comb has no solder mask to test the effects of flux only. One comb is completely masked to measure the effects of flux or preheats on the solder mask material. One comb has the striped mask to measure flux residue effects, similar to the Bellcore pattern. The two different striped patterns can be used to generate correlation factors. The solder side of the board also contains rows of 0805 and 1206 chips to look at wave soldering SMT devices.

#### **Figure 1 – Component Side**



#### Figure 2 – Solder Side



Table 1 – Umpire Board BOM

Item	Qty	Description			
1	2	LCC, 68 pin, no die			
2	1	TQFP, 80 pin, 0.5 mm pitch			
3	1	BGA, 272 pin, 1.27mm pitch			
4	1	PTH pin grid array			
5	1	PTH 5 pin header			
6	2	PTH 14 pin dip sockets			
7	13	PTH axial diodes			
8	2	PTH 3 pin header			
9	1	PTH 16 pin header			
10	1	PTH 2 pin header			
11	26	SMT 0805 chip resistors			
12	17	SMT 1206 chip resistors			
13	1	PTH radial capacitor			

The board contains two rows of 40 mil diameter holes and two rows or 20 mil diameter holes to examine characteristics such as hole fill. The board also contains room for a large electrolytic capacitor, which can be significant flux entrapment sites in assemblies.

Process	Machine		
Screen Print	DEK 248		
Component Placement	Mydata TP 9		
Solder Reflow	Heller 1500		
Axial Insertion	Universal 6285		
Wave Solder	Dover Soltec Delta		
Aqueous Cleaner	Technical Devices		

 Table 2 - Process Equipment Summary

#### **Table 3 - Screen Print Parameters**

Factor	Setting			
Stencil Thickness	6 mil			
Aperture Ratio	90%			
Stencil Fabrication Method	Laser Cut/ Electro-Polish			
Paste Chemistry	No-Clean and WS			
Paste Formula	63/37 eutectic			
Blade Length	12"			
Print Force	12 lb			
Print Speed	2.0"/sec			
Wipe Frequency	Every 10th Print			
Blade Lift Off Height	1.0"			
Snap Off	zero			
Blade Type	metal			

Reflow and wave solder settings and profiles were selected to meet paste and flux manufacturer's guidelines.

	Avg Cl	Avg BR	Total
HASL 1	1.63	0.52	2.15
HASL 2	3.05	11.17	14.22
HASL 3	6.16	14.49	20.76
Imm Ag	0.49	0.00	0.49
Cu OSP	1.39	0.74	2.13

All units in ug/ in.2

#### **Table 5 – Contamination DOE Matrix**

	<b>Process Number of Units</b>								
PWB Type/ Finish	WS No Saponifier	WS Typical Saponifier	No Clean						
HASL1	10	10	10	10					
HASL2	10	10	10	10					
HASL3	10	10	10	10					
Imm Ag	10	10	10	10					
Cu OSP	10	10	10	10					

WS – Water Soluble

### **TEST PROCEDURES**

#### **Preparation and Handling**

All of the test boards were handled at all times either using non-contaminating gloves or by the edges only. The boards were separated into coupon boards for ion chromatography (IC) testing and those for surface insulation resistance (SIR) testing. The boards for IC testing went directly to that test procedure. The unprocessed controls had no connectors on them since they were not part of the assembly process. For these controls, the test connections were made by hand soldering the Teflon coated leads to the circuit boards. During the hand soldering, the remainder of the board was covered with clean aluminum foil to guard the test patterns from flux spattering during soldering. We used Kester 44 RMA cored wire solder with no postsoldering cleaning. Our research has shown that this attachment method does not adversely affect SIR performance. The design of the Umpire board, with the voltage supply and current return portions of the board separated, ensures that there was no electrical leakage due to residual flux.

The SIR data acquisition system had a nominal 1 megohm resistor (1.0 E 6) in each circuit pathway. The one megohm value is relatively common in the SIR test field. These current-limiting resistors serve three primary purposes:

- To preserve dendritic formations that grow during the test
- To protect the data acquisition system from large currents
- To prevent a short circuit on one pattern from "robbing" the current from the remainder of the board.

#### **SIR Measurements**

Initial measurements were taken at  $25^{\circ}$ C and 50% RH with a measurement voltage of 100 volts DC and an electrification time of 60 seconds. For the unprocessed controls, because they had no components, all test patterns on the boards were measured.

After the initial measurements were complete, a temperature humidity profile was initiated. Measurements were made sequentially, board by board, until complete. Each measurement set took approximately 3 hours. Following the completion of the measurements, the electrical bias was reapplied to all test patterns on all boards.

Following the final ramp down step, a final measurement set was taken one hour after the chamber stabilized at ambient conditions.

Following the SIR test, all test specimens were removed from the chamber and visually examined for signs of corrosion or metal migration using top lighting and back lighting.

#### Ion Chromatography Procedure

The ion chromatography (IC) test method can be found in IPC-TM-650.

# IC Analysis -- Residue Summary

For this project, ion chromatography detected the following primary anion residues: chloride (Cl - ), bromide (Br - ), and weak organic acids (WOAs). These residues are typical for printed wiring boards and assemblies.

Weak organic acid residues are generally benign unless applied in volumes so high that adequate thermal activation cannot occur.

# **Residue Findings by IC**

The ion chromatography data is expressed as micrograms of the residue species per square inch of extracted surface, unless otherwise noted. This measure should not be confused with micrograms of sodium chloride equivalent per square inch, which is the common measure for most ionic cleanliness (ROSE) test instruments.

# Bromide

For epoxy-glass laminates, bromide levels range anywhere from 0 to 7 micrograms per square inch  $(ug/in^2)$  depending on the amount of bromide fire retardant the laminate manufacturer has added. Exposure to reflow conditions tends to increase the porosity of the laminate and mask and so bromide levels can go as high as 10-12 micrograms per square inch with several exposures to reflow conditions.

For bromide residues, CSL recommends the following:

For bare boards fabricated with a cold plating process, such as silver or palladium, we recommend bromide levels under 10 micrograms per square inch.

Using these levels as guidelines, we see the bromide levels for the unprocessed controls as typical for FR-4 laminate with a UL 94 V-0 rating. This level of bromide can be completely attributed to the fire retardant material in the laminate and mask. As a completely complexed (chemically) material, the bromide is not detrimental and poses little electrochemical risk to assemblies. The relatively low level of bromide indicates that the fabricator did not use a brominated HASL flux.

# Chlorides

Chloride is one of the more detrimental materials found on printed circuit assemblies. Chlorides can come from a variety of sources, but is most often attributable to flux residues. Chlorides will generally initiate and propagate electrochemical failure mechanisms, such as electrical leakage, metal migration and electrolytic corrosion, when combined with water vapor and an electrical potential. The amount of allowable chloride on a bare board is difficult to assess. If the boards will go into an assembly process, which incorporates cleaning, then a higher level of chloride can be tolerated. If the bare board is intended for a no-clean assembly process, then a lower chloride level is recommended.

# **CSL Chloride Guidelines for Assemblies**

These recommended maximums do not presently appear in any nationally accepted specifications or standards, but are based on our failure analysis efforts with numerous customers. The amount of chloride that can be tolerated on an assembly depends on the flux chemistry being used. Assemblies processed with highsolids rosin fluxes (RA, RMA) can tolerate higher levels of chloride due to the encapsulating nature of the rosin.

# Weak Organic Acids

Weak organic acids, such as adipic or succinic acid, serve as activator compounds in many fluxes, especially no-clean fluxes. WOAs are typically benign materials and are therefore not a threat to long term reliability. In order to avoid formulation disclosure difficulties with flux manufacturers, we group all detected weak organic acid species together and refer to them collectively as WOAs.

Fully reacted and therefore benign WOAs act as insulators that, even at levels as low as  $10 \text{ g/in}^2$ , can potentially create a high resistance contact-to-contact resistance problem on devices such as switches.

# Test Procedure – ROSE (Omegameter 600SMD)

The resistivity of solvent extract (ROSE) testing was accomplished using an Omegameter 600SMD. The Omegameter was chemically calibrated prior to testing. The test samples were tested according to IPC-TM-650, method 2.3.26.1 using a 10 minute test time, pin grid array area in 100 ml of solution, and a solution concentration of 75% isopropanol/25% deionized water.

#### **Modified ROSE**

The modified-ROSE test is still in the experimental stage. It has received a tentative designation from the IPC as IPC-TM-650, method 2.3.25.1. The test solution used for this test was the remaining 50 ml of extract solution remaining from the ion chromatography procedure.

#### Water Drop Electrochemical Migration Procedure

Using the test boards after processing without the DIP package in sites U7 and U8.

- 1. Both Y patterns are electrified at 10 volts for 30 seconds, then one drop of D.I. water (approximately 10ul) is placed in the center of the Y Pattern.
- 2. Boards are placed under a microscope and viewed at 60x for viewing of dendrite growth; Tests can be repeated on the remaining Y pattern.
- 3. A stopwatch is used to determine the time to migration. The watch is started at the time the water is applied to the energized Y pattern and stopped when the first dendrite contacts the opposite trace. This data is recorded then the power is stopped and the assembly is moved aside.

### **Test Procedure – Water Drop Test**

Y patterns are electrified at 10 volts for 30 seconds, then one drop of D.I. water is placed in the center of the comb. Current recorded as a function of time up to a maximum current of 150 mA, dry-out of the drop or 5 minutes. The test is repeated for the remaining Y pattern on the first coupon and the 4 combs on the second coupon. Coupons examined microscopically after electrical testing for dendrites etc.

#### Surface Insulation Resistance An Overview

Surface insulation resistance (SIR) is a material characteristic of a system of materials, such as laminate, solder mask, and metal conductors. The testing of SIR is generally done under conditions of high temperature and high humidity, with an applied electrical potential. The specific levels of temperature, humidity and potential chosen depend largely upon what specification is driving the testing, and what the goal of the testing may be. The acceptable levels of resistance demonstrated throughout the testing also depend largely upon what specification or test method is used, along with the geometry of the test patterns.

For a more in-depth understanding of SIR testing in general, and the many factors contain in analyzing SIR data, the reader is referred to IPC-9201, The SIR Handbook. This Handbook is available from the IPC (www.ipc.org).

For the SIR test professional who wishes to test many of these factors, there are several different standards, specifications, and test methods to choose from. In the United States, the three most commonly encountered SIR tests can be found in the following documents:

- ANSI J-STD-001, used by high reliability (Class 2 and 3) assemblers
- ANSI J-STD-004, used by flux vendors for materials characterization

Both these methods have an applied potential during humidity exposure of 50 volts DC and a measurement voltage of 100 volts DC.

### **SIR Data Comparisons**

SIR data is considered presently to be dependent upon the geometry of the test pattern and it is not a recommended practice to directly compare resistance readings from two or more SIR patterns that have significantly differing geometry. In other words, the data from a comb pattern should not be directly compared with the data from a Y pattern. In this study, we compare similar patterns only.

Correlation Combination IC vs. Test X	tion Combination C vs. Test X Coeffic	
	No Clean	W.S.
Cl vs. Modified Rose	-0.856	-0.65
Cl vs. Omega Meter	-0.314	-0.84
Cl vs. Water Drop	-0.964	-0.84
Br vs. Modified Rose	-0.57	-0.5
Br vs. Omega Meter	-0.147	-0.74
Br vs. Water Drop	-0.952	-0.58
Cl + Br vs. Modified Rose	-0.661	-0.55
Cl + Br vs. Omega Meter	-0.195	-0.55
Cl + Br vs. Water Drop	-0.983	-0.8

#### **Table 6 – Correlation Among Tests**

Greater than abs. 0.75 implies strong correlation

		Bare PWB		Soldered Assembly		Failures				
Finish	Group	Cl	BR	Cl + BR	Cl	BR	Cl + BR	SMT	РТН	Bottom
HASL1/WSF Water Cleaned (0% Sap)	1	1.63	0.52	2.15	9.65	6.90	16.55	84%	23%	20%
HASL2/WSF Water Cleaned (0% Sap)	2	3.05	11.17	14.22	7.07	10.38	17.44	58%	23%	33%
HASL3/WSF Water Cleaned (0% Sap)	3	6.06	14.49	20.76	7.21	12.00	19.21	68%	23%	47%
Imm Ag/WSF Water Cleaned (0% Sap)	4	0.49	0	0.49	7.26	6.63	13.89	48%	40%	43%
CU OSP/WSF Water Cleaned (0% Sap)	5	1.39	0.74	2.13	6.97	5.99	12.96	36%	40%	37%
HASL1/WSF Water Cleaned (3% Sap)	6	1.63	0.52	2.15	3.47	3.92	7.39	4%	0%	0%
HASL2/WSF Water Cleaned (3% Sap)	7	3.05	11.17	14.22	4.60	7.79	12.39	0%	0%	0%
HASL3/WSF Water Cleaned (3% Sap)	8	6.16	14.49	20.76	4.44	7.22	11.67	0%	0%	0%
Imm Ag/WSF Water Cleaned (3% Sap)	9	0.49	0	0.49	3.90	3.63	7.53	0%	0%	0%
CU OSP/WSF Water Cleaned (3% Sap)	10	1.39	0.74	2.13	4.44	4.19	8.63	0%	0%	0%
HASL1/WSF Water Cleaned (10% Sap)	11	1.63	0.52	2.15	5.94	3.44	9.38	0%	0%	0%
HASL2/WSF Water Cleaned (10% Sap)	12	3.05	11.17	14.22	5.53	10.34	15.87	0%	3%	0%
HASL3/WSF Water Cleaned (10% Sap)	13	6.16	14.49	20.76	4.92	10.09	15.01	30%	23%	20%
Imm Ag/WSF Water Cleaned (10% Sap)	14	0.49	0	0.49	4.38	3.62	8.00	0%	0%	0%
CU OSP/WSF Water Cleaned (10% Sap)	15	1.39	.74	2.13	5.31	4.03	9.34	0%	0%	0%
HASL1/No Clean Process	16	1.63	0.52	2.15	1.80	5.90	7.69	2%	3%	0%
HASL 2/No Clean Process	17	3.05	11.17	14.22	5.30	17.83	23.13	80%	57%	67%
HASL3/No Clean Process	18	6.16	14.49	20.76	5.81	11.69	17.50	74%	43%	57%
Imm Ag/No Clean Process	19	0.49	0	0.49	2.32	5.61	7.93	0%	0%	0%
CU OSP/No Clean Process	20	1.39	.74	2.13	2.72	5.50	8.22	0%	0%	0%

Table 7 – Bare PWB vs. Soldered Assembly Contamination

# Passed SIR Test

The above no clean processed units show real promise for implementation, from a contamination perspective. The groups HASL2/WSF Water Cleaned and HASL3 WSF Water Cleaned show excessive bromide contamination and follow-up discussion with the board supplier will hopefully expose some corrective action opportunity.

In most cases bromide levels and chloride levels will rise as a result of the thermal excursions that reflow and/or wave represent.

### RESULTS

The acceptance limits prior to running the experiment were 2.0 ug/in<sup>2</sup> for Cl and 3.0 ug/in<sup>2</sup> for Br. for a bare PWB and 5.0 ug/in<sup>2</sup> for Cl for a soldered assembly.

Water Soluble Flux with 0% Saponifier @ Wash None of the units in this group passed SIR testing. Some saponifier was considered a must as a result of this data.

#### Water Soluble Flux with 3% Saponifier @ Wash

Using a final assembly limit of 6.0 ug/in<sup>2</sup> Cl, groups 6 (HAS 1), 7 (HAS 2), 8 (HASL 3), 9 (Imm Ag) and 10 (CuOSP) met specification. Group 6 is the control group for board finish (HASL <2 ug/in<sup>2</sup>), process and flux type. This supports a reduction in percentage of saponifier to 3% concentration.

#### Water Soluble Flux with 10% Saponifier @ Wash

Groups 12(HASL 2), 13 (HASL 3), and 14 (Imm Ag) met the 6.0 ug/in.<sup>2</sup>. Chloride limit but all had bromide levels above 5.0 ug/in.<sup>2</sup>. Only Group 14 had bromide levels below 9.0 ug/in.<sup>2</sup>. Group 15 (Cu OSP) met the 9.0 limits.

### No Clean Flux (No Wash)

Groups 16, 19 and 20 met the 2.5 ug/in.<sup>2</sup> limits for Chloride. Only group 16 met both the limits for Chloride and Bromide. Groups 19 and 20 were very close to the limit for bromide.

#### FINAL ASSEMBLY CONTAMINATION VERSUS BARE PWB CONTAMINATION (PROCESS DELTA)

#### Water Soluble Process

On average, soldering (processing) a bare PWB into an assembly added up to 8 ug/in.<sup>2</sup> of chloride and up to 6 ug/in.<sup>2</sup> of bromide. Amount of added contamination decreased as saponifier was increased. However, the level of weak organic acid (WOA) jumped a factor of ten (ranging between 39 and 55 ug/in.<sup>2</sup>) with 10% saponifier, as compared to zero or 3% of the typical percentage.

# **No Clean Process**

On average, soldering (processing) a bare PWB into an assembly added up to only 3 ug/in.<sup>2</sup> of chloride and up to 7 ug/in.<sup>2</sup> of bromide. The level of weak organic acid (WOA) was higher than all water-soluble units (ranging between 80 and 100 ug/in.<sup>2</sup>). This was well below the 220 ug/in.<sup>2</sup> level seen on production units tested during paste and flux validation.

# **Alternative PWB Finishes**

The Cu OSP finished PWBs exhibited less wetting as compared to HASL finishes.

# **Component Solderability / Wetting**

Initial experimentation with the 0402 chips placed between SMT pad patterns exhibited good wetting, but unfortunately included shorts as well as adhesive on the pad. Later, refinement to the pad design and adhesive dot was performed.

Wetting on all LCC castellations indicated a successful class 1 solder joint, however all LCC wetting was in the range of 50% of castellation height, indicating a marginal process for class 2 and class 3.

BGA wetting and alignment met the requirements for IPC 610 class 3. X Ray analysis confirmed alignment, but unfortunately daisy chained PWB vias hid any possible solder shorting. Voiding met the IPC-A-610 class 3 specification for less than 10% voiding.

# **OVERALL CONCLUSION**

The electrical product operating in a humid environment will fail due to high enough levels of ionic contamination on sensitive enough circuits. Electrical performance is directly correlated to SIR on sensitive circuitry and will create the same electrical effects on the assemblies. Ion Chromatography, SIR and Water Drop testing correlate with this data. Omega Meter and Modified ROSE run in the Omega Meter do not appear to correlate.

The incoming bare board cleanliness has great effects on electrical performance of boards cleaned with water and boards processed with no clean assembly techniques. Clean incoming boards showed low percentages of failures on no clean process. HASL2 and HASL3 incoming bare boards showed large percentage of electrical failures.

	No	Clean	Water Soluble			
	Bare PWB	Soldered Assembly	Bare PWB	Soldered Assembly		
CL	2.5	3.0	8.0	6.0		
Br	5.5	9.0	15.0	9		

All units in ug/in.<sup>2</sup>

\* These limits correlate to SIR pass/fail criteria used in this experiment, using specific pastes, fluxes, PWB materials, components and our processes. They may not apply to your process and your components and are offered as a reference only. Experimentation to characterize your process with your materials is strongly recommended.

The Alpha Silver PWB process and copper OSP provided incoming ionically clean assemblies that performed well with the no clean and saponified cleaning processes.

The areas of entrapment, such as the BGA, LCC and the TQFP all performed outstandingly. The need for a saponifier for the low stand off boards can be seen on the percentage of failures identified between the SMT and the PTH technology.

No-clean units with bare PWB CL below 2.5 ug and Br below 5.5 ug/in.<sup>2</sup> performed very well.

Saponifier washed units with Cl below 8.0 ug/in.<sup>2</sup> and Br below 15 ug/in.<sup>2</sup> also performed very well.

The 3% saponified group passed the numerical requirements and requirement for no metal migration outlined by the IPC ANSI J-STD 001 appendix B for Class Three hardware. The 10% saponified group showed three of the ten boards to have poor electrical performance due to the high levels of bromide on three boards. These failures are due to the incoming bromide levels of the dirty HASL bare board group.

There were some instances of metal migration on the Alpha Silver boards in the SIR test. There was rampant metal migration on the HASLed boards, and none on the clean bare copper boards. In some cases, there was an assignable cause for the metal migration, in others, there was not. There were also reaction products present (black and green) on the SIR test boards. With many of the failures on the SMT, electrical leakage was detected as the failure but no dendrite shorts were present. The resistors on both sides of nearly all assemblies (groups 6-10, 14, 15, 16, 19 and 20) showed great performance. Little voiding was detected in the glued resistors. All sizes of the resistor passed with flying colors (0402, 0805, 1206).

3% (2 gal/tank) saponifier concentration is at least as effective as 10% (6 gal/tank) concentration, from a product contamination perspective. Saponifier concentration at wash was reduced as a result of this experiment.

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