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ABSTRACT

For mission critical electronics or Class III products, such as those used within the military, aerospace and medical industries, highest electronic reliability is a requirement as failure is not an option. Within the electronics industry, this means that residues, either ionic or non-ionic, must be fully removed. Partially removed or untouched residues can lead to component and product failures resulting from electrochemical migration, dendrite growth and electrical leakage currents.

The goal of this study was to identify and qualify an aqueous cleaning process capable of removing combinations of no-clean flux residues for Class III electronic assemblies. Teamed with a global electronic manufacturing service (EMS) provider supplying electronics to the aerospace and medical industry, the Design of Experiment (DOE) developed was executed in two phases. Initial testing was completed utilizing EMS boards and final testing was validated using IPC test coupons and standards.

The goals of each phase of the DOE were as follows:

Phase 1:

I. Determine optimum parameters to effectively clean flux residues from EMS board samples and verify cleanliness through visual inspection and ionic contamination analysis.

Phase 2:

- I. Using the optimum parameters from Phase 1 above, clean additional EMS boards and verify cleanliness using Ion Chromatography (IC) and Solvent Extraction Conductivity (SEC) analyses.
- II. Conduct Surface Insulation Resistance (SIR) and Electrochemical Migration (ECM) analyses on IPC test coupons cleaned using parameters defined in Phase 1.
- III. Verify compatibility of all critical components and materials used on the boards with the selected cleaning agent.

Through this DOE, the authors were able to identify and quantify the critical parameters impacting cleanliness for Class III electronic components as validated by numerous IPC assessment standards.

INTRODUCTION

Class III products are generally considered to be high performance electronic products. These products demand continued high performance throughout the product lifecycle; the end use environment may be harsh and the equipment must function when and as required, such as in life support equipment or other critical systems. They are typically used in medical, military, avionics, and automotive applications.

In this study, a global contract manufacturer received a contract to manufacture Class III devices for a large medical Original Equipment Manufacturer (OEM). Since these products are manufactured with no-clean flux, the OEM requested extensive testing, evaluation and validation to confirm that the selected cleaning process is capable of removing all flux residues and thereby ensuring the long term integrity and reliability that is required of Class III products.

Based on the OEM's product requirement, a DOE was developed and executed in two phases. The initial cleaning trials were conducted at the ZESTRON Technical Center employing spray-in-air cleaning equipment similar to one available at the EMS. Analytical analyses were conducted at ZESTRON, the EMS and several independent laboratories. Test vehicles used were EMS boards and IPC coupons. All necessary ESD precautions were followed throughout this study.

During Phase 1 of the DOE, visual inspection and ionic contamination analyses were conducted at the ZESTRON Technical Center as the optimum inline cleaner operating parameters were developed. Once the optimized cleaning parameters were identified, Phase 2 was executed. IC and SEC tests were completed using the EMS boards validating the recommended cleaning parameters identified in Phase 1. The IC test was conducted at a certified laboratory and the SEC test at the EMS location. For Phase 2, SIR and ECM analyses were also conducted at a certified laboratory using IPC test coupons. Finally, compatibility testing was performed with the selected cleaning agent with all critical assembly components and materials.

Following the successful execution of the DOE, the recommended cleaning process was implemented at the OEM site and the cleaning results were validated.

DISCUSSION OF METHODOLOGY

The EMS used six (three each) solder pastes and wave fluxes within their manufacturing process and cleans all Printed Circuit Boards (PCBs) through an inline spray-in-air cleaner. Thus, for this study, the six solder pastes and liquid fluxes used were identified as Solder Paste A (leaded no-clean), Solder Paste B (lead-free no-clean), Solder Paste C (no-clean tacky flux) and Wave Flux D (no-clean leaded process), Wave Flux E (no-clean lead-free process) and Wave Flux F (no-clean lead-free process).

Upon considering wave flux process options within the DOE, the EMS requested for the cleaning process parameters, for dried flux (flux only) as well as activated flux (flux and solder) to be identified. Thus, it was decided to prepare boards with activated flux, dried flux and a combination of activated and dried flux. Although cleaning parameters were optimized for all combinations and verified using IC and SEC analyses, cleaning parameters for the activated and dried flux boards (labeled as ActDry) were considered the most challenging and therefore used as the basis for recommending optimal inline cleaning parameters.

Thus, wave solder fluxes were classified as:

- Activated flux (labeled as Act)
- Dried flux (labeled as Dry)
- Activated and dried flux (labeled as ActDry)

Prior to executing Phase 1 of the DOE, an aqueous cleaning agent was selected on the basis of its ability to solubilize the selected pastes and fluxes as well as its compatibility with the application cleaning equipment. As a result of this analysis, a micro phase cleaning agent, identified as Cleaning Agent A, was selected for all the cleaning trials within this DOE.

Phase 1: Test Protocol

The EMS initially provided fifty-nine (59) test boards for use in the initial DOE. The boards were categorized by paste and flux type. Of these, thirty-two (32) boards were used to determine optimum wash settings for the inline cleaner, including cleaning agent concentration, wash temperature and wash exposure time. Initial cleanliness assessments were made using visual inspection and ionic contamination analysis.

Visual inspection was conducted according to IPC-A-610E [1]. Ionic contamination analysis was conducted using test equipment with a 75% solution IPA in DI-water in accordance with the IPC-TM-650 Method 2.3.25 [2]. This evaluation was based on J-STD-001E [3].

The initial cleaning trials as well as the visual inspection and ionic contamination analyses were conducted at the ZESTRON Technical Center since this location had the same cleaning equipment as the contract manufacturer site.

Of the fifty-nine (59) boards provided by the EMS, they were categorized and processed as follows:

- Twenty-six (26) boards were reflowed using the three solder paste varieties and divided into three groups:
 - Seventeen (17) were used for defining inline process parameters
 - Six (6) were used for Ion Chromatography analysis
 - Three (3) were used for SEC analysis
- Thirty-three (33) boards were soldered with the three wave flux varieties and divided into three groups:
 - Fifteen (15) were used for defining inline process parameters
 - Twelve (12) used for Ion Chromatography analysis
 - Six (6) were used for SEC analysis

		Number of Boards Used						
	Process Development	Ion Chromatography	SEC	Total				
Paste / Board #								
Paste A / 1-9	6	2	1	9				
Paste B / 1-9	6	2	1	9				
Paste C / 1-8	5	2	1	8				
Sub Total Paste:	17	6	3	26				
Flux / Board #								
Flux D / 1-6 (Act)	3	2	1	6				
Flux D / 1-3 (Dry)		2	1	3				
Flux D / 1-2 (ActDry)	2			2				
Flux E / 1-6 (Act)	3	2	1	6				
Flux E / 1-3 (Dry)		2	1	3				
Flux E / 1-2 (ActDry)	2			2				
Flux F / 1-6 (Act)	3	2	1	6				
Flux F / 1-3 (Dry)		2	1	3				
Flux F / 1-2 (ActDry)	2			2				
Sub Total Flux:	15	12	6	33				
Grand Total:	32	18	9	59				

Table 1: Board Categorization by Paste and Flux type

RESULTS DISCUSSION

Phase 1: Cleaning Process

During the process development trials using the initial thirty-two (32) boards, the cleaning agent concentration, wash temperature and inline conveyor belt speed were varied and the results were recorded as summarized in Table 2.

At the conclusion of each trial with each board type, visual inspection and ionic contamination analyses were performed to assess board cleanliness until the optimum inline cleaner operating parameters were identified.

Cleaning Process: Initial Parameters				
Cleaning Agent Concentration:	10% to 15%			
Wash Temperature:	125°F to 145°F			
Conveyor Belt Speed:	2.0 to 2.5 ft/min			

Table 2: Cleaning Process Initial Parameters

Phase I: Cleaning Process Results

The inline cleaner process parameters evaluated as well as the ionic contamination test results are detailed in Table 3 and Table 4. The operating parameters yielding the lowest ionic contamination value for each solder and flux type are highlighted in each table. For this analysis, the ionic contamination threshold was set at $10.06 \mu g/inch^2$.

Test #	Paste / Board #	Cleaning Agent A Conc. (%)	Wash Temp. (°F)	Belt Speed (FPM)	Rinse Temp. (°F)	Ionic Values (µg/inch ²)
1	Paste B / 8	15%	135°F	2.0	140°F	0.00
2	Paste A / 8	15%	135°F	2.0	140°F	2.60
3	Paste C / 4	15%	135°F	2.0	140°F	0.00
4	Paste A / 9	15%	135°F	2.0	140°F	0.00
5	Paste B / 9	12%	125°F	2.0	140°F	0.60
6	Paste A / 3	12%	125°F	2.0	140°F	0.30
7	Paste C / 5	12%	125°F	2.0	140°F	0.06
8	Paste B / 1	10%	125°F	2.0	140°F	0.70
9	Paste A / 4	10%	125°F	2.0	140°F	0.50
10	Paste C / 6	10%	125°F	2.0	140°F	0.08
11	Paste B / 2	10%	135°F	2.0	140°F	0.34
12	Paste A / 2	10%	135°F	2.0	140°F	0.12
13	Paste C / 7	10%	135°F	2.0	140°F	0.04
14	Paste B / 3	15%	135°F	2.5	140°F	0.79
15	Paste A / 1	15%	135°F	2.5	140°F	0.59
16	Paste C / 8	15%	135°F	2.5	140°F	0.00

Table 3: Cleaning Results of the Post-Reflow Boards

Solder paste substrate pictures before and after cleaning: Figures 1 – 6



Figure 1: Paste A Before Cleaning



Figure 3: Paste B Before Cleaning



Figure 2: Paste A After Cleaning



Figure 4: Paste B After Cleaning



Figure 5: Paste C Before Cleaning



Figure 6: Paste C After Cleaning

	Table 4: Cleaning Results of the Post Wave-Solder Boards									
Test #	Flux / Board #	Cleaning Agent A Conc. (%)	Wash Temp. (°F)	Belt Speed (FPM)	Rinse Temp. (°F)	Ionic Values (μg/inch ²)				
1	Flux D / 2 (ActDry)	15%	135°F	2.0	140°F	0.70				
2	Flux E / 2 (ActDry)	15%	135°F	2.0	140°F	0.60				
3	Flux F / 2 (ActDry)	15%	135°F	2.0	140°F	0.57				
4	Flux D / 1 (ActDry)	15%	145°F	2.0	140°F	0.08				
5	Flux E / 1 (ActDry)	15%	145°F	2.0	140°F	0.07				
6	Flux F / 1 (ActDry)	15%	145°F	2.0	140°F	0.09				
7	Flux D / 6 (Act)	15%	135°F	2.0	140°F	0.07				
8	Flux E / 1 (Act)	15%	135°F	2.0	140°F	0.08				
9	Flux F / 3 (Act)	15%	135°F	2.0	140°F	1.00				
10	Flux E / 5 (Act)	15%	145°F	2.0	140°F	0.80				
11	Flux F / 2 (Act)	15%	145°F	2.0	140°F	0.27				
12	Flux D / 5 (Act)	10%	135°F	2.0	140°F	1.30				
13	Flux E / 6 (Act)	10%	135°F	2.0	140°F	0.08				
14	Flux F / 1 (Act)	10%	135°F	2.0	140°F	0.15				

 Table 4: Cleaning Results of the Post Wave-Solder Boards

Wave flux substrate pictures before and after cleaning: Figures 7 – 12



Figure 7: Wave Flux D (ActDry) Before Cleaning

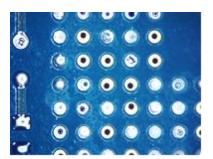


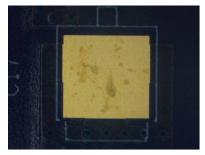
Figure 9: Wave Flux E (ActDry) Before Cleaning

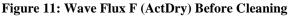


Figure 8: Wave Flux D After Cleaning



Figure 10: Wave Flux E (ActDry) After Cleaning





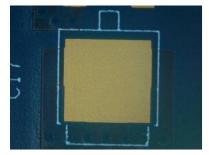


Figure 12: Wave Flux F (ActDry) After Cleaning

Phase 1: Cleaning Process Results Summary

Through the process development phase of this study, the authors identified the optimum cleaning parameters required in order to fully remove all post-soldered flux residues. However, since the EMC was seeking a single process condition capable of cleaning all flux residues, the authors used this test data as the basis for the optimum process recommendation. These optimized cleaning process parameters were then employed throughout the remainder of the DOE. These findings are detailed in Table 5.

Optimum Inline Cleaning Process Parameters						
Post Reflow Flux						
Cleaning Agent Concentration:	15% (by volume)					
Wash Temperature:	135°F					
Belt Speed:	2.0 FPM					
Post Wave Flux						
Cleaning Agent Concentration:	15% (by volume)					
Wash Temperature:	145°F					
Belt Speed:	2.0 FPM					
Optimized Cleaning Process Parameters:						
Cleaning Agent Concentration:	15% (by volume)					
Wash Temperature:	140°F					
Belt Speed:	2.0 FPM					

Table 5:	Optimum	Inline	Cleaning	Process	Parameters

Phase 2: Test Protocol

To further validate the cleanliness results of the parameters identified in Table 5, SIR and ECM analyses were performed using IPC test coupons. Following these tests, compatibility testing with critical components and materials was also performed using the selected cleaning agent.

Regarding the SIR and ECM tests, eighteen (18) IPC B-24 and nine (9) IPC B-25A test coupons were sourced incorporating the matrix of paste and flux types required. Additionally, control coupons were included in order to confirm the accuracy of the tests. Excluding the control coupons, all were cleaned utilizing the optimized cleaning parameters identified in Table 5. Once cleaned, all of the boards were sent to an independent laboratory for analysis. The SIR tests were conducted per J-STD-004B [4] and IPC-TM-650, Method 2.6.3.7 [5]. The ECM tests were conducted per J-STD-004A [6] and IPC-TM-650, Method 2.6.14.1 [7].

Regarding material compatibility, separate tests were conducted using the EMS label material as well as a variety of critical components. For label compatibility trials, two (2) label types were evaluated; one used exclusively in the lead-free process and the other in the leaded process. The labels were passed through the inline cleaner five (5) times using Cleaning Agent A (15% concentration) at 140°F and 2.0 ft/min. As part of these trials, the labels were examined for adhesive and ink removal after each inline pass.

Material compatibility testing included short-term and long-term tests. For the short-term test, critical components were subjected to Cleaning Agent A at a concentration of 30% and 140°F for 15 minutes in a beaker placed on a hot plate with a magnetic stirrer. Upon inspection following the test, if the part was not compromised, the beaker test was extended to 24 hours as part of the long-term compatibility testing.

In additional to the beaker tests, the critical components were also passed through the inline cleaner five (5) times with the cleaning agent concentration at 15% and 140°F yielding a total exposure time of 13 minutes. The parts were then inspected for dimensional and cosmetic changes. The components subjected to material compatibility testing are detailed in Table 6. Separate material compatibility tests were conducted using the EMC label material as well as a variety of critical components.

	Table 0. Components for Compatibility Testing							
Item	Material	ID	Quantity	Description				
				• One part used for short-term and				
1	Diastia component	A1 A2 A2	3	long-term testing				
1	Plastic component	A1, A2, A3	5	 Second part used for inline testing 				
				• Third part for reference purposes				
2	Wine however	D	1	• Used for short-term and long-term				
2	Wire harness	В	1	testing				
3	Copper cables	С	1	• Used for inline testing				
4	Wire harness	D	1	• Used for inline testing				
				• One part used for short-term and				
5	Plastic component	E1, E2	2	long-term testing				
				• Second part used for inline testing				

Table 6: Components for Compatibility Testing

Phase 2: Ion Chromatography Results

For this test, eighteen (18) EMS boards as referenced in Table 1 were cleaned using the optimized parameters detailed in Table 5 and sent to an independent analytical laboratory for analysis.

The boards were divided into three groups consisting of six (6) samples each. These were Activated Flux samples, Dried Flux samples and Solder Paste samples. Ion Chromatography testing was conducted per IPC-TM-650, Method 2.3.28A [8]. All results are detailed in Tables 7, 8 and 9.

Based on the authors' experience with this test methodology, the maximum contamination level for all ionic species is indicated within each table. As can be seen, the contamination level determined in all tests was found to be well below the acceptable limit.

	Anions & WOA (Weak Organic Acids)							
	Maximum		Solder	Paste Typ	e / Board I	Number		
Ionic Species	Contamination Level (µg/in ²)	Paste A / 5 (µg/in ²)	Paste A / 6 (µg/in ²)	Paste B / 5 (µg/in ²)	Paste B / 6 (µg/in ²)	Paste C / 1 (µg/in ²)	Paste C / 2 (μg/in ²)	
Fluoride	3	0.23	0.21	0.11	0.09	0.14	0.14	
Chloride	4	0.30	0.28	0.12	0.12	0.26	0.25	
Nitrite	3	ND	ND	ND	ND	ND	ND	
Bromide	10	ND	ND	0.45	0.40	ND	ND	
Nitrate	3	0.05	ND	0.04	0.05	ND	0.07	
Sulfate	3	ND	ND	ND	ND	ND	ND	
Phosphate	3	ND	ND	ND	ND	ND	ND	
Acetate	3	0.50	0.50	0.31	0.26	0.28	0.26	
Formate	3	0.98	0.94	0.47	0.43	0.62	0.58	
Total WOA:	<25	2.11	2.23	1.18	1.11	1.17	1.18	
			Cations					
	Maximum		Solder	Paste Typ	e / Board I	Number		
Ionic Species	Contamination Level (µg/in ²)	Paste A / 5 (µg/in ²)	Paste A / 6 (µg/in ²)	Paste B / 5 (µg/in ²)	Paste B / 6 (µg/in ²)	Paste C / 1 (µg/in ²)	Paste C / 2 (µg/in ²)	
Lithium	3	ND	ND	ND	ND	ND	ND	
Sodium	3	0.39	0.33	0.28	0.15	0.11	0.15	
Ammonium	3	1.23	1.28	0.56	0.55	0.66	0.65	
Potassium	3	1.19	1.15	0.70	0.68	0.76	0.72	
Magnesium	1	ND	ND	ND	ND	ND	ND	
Calcium	1	0.41	0.38	0.30	0.29	0.22	0.18	

 Table 7: Ion Chromatography Results – Solder Paste Samples

ND= None Detected

	Anions & WOA (Weak Organic Acids)							
			Flu	ıx Type / B	oard Num	ber		
Ionic Species	Maximum Contamination Level (µg/in ²)	Flux D / 1 (Act) (μg/in ²)	Flux D / 2 (Act) (μg/in ²)	Flux E / 2 (Act) (μg/in ²)	Flux E / 3 (Act) (µg/in ²)	Flux F / 4 (Act) (µg/in ²)	Flux F / 5 (Act) (µg/in ²)	
Fluoride	3	0.16	0.12	0.14	0.15	0.13	0.23	
Chloride	4	0.16	0.06	0.31	0.18	0.30	0.21	
Nitrite	3	ND	ND	ND	ND	ND	ND	
Bromide	10	0.15	0.17	0.16	0.18	0.22	ND	
Nitrate	3	ND	ND	0.06	0.04	0.04	0.14	
Sulfate	3	ND	0.03	0.10	0.06	ND	0.07	
Phosphate	3	ND	ND	ND	ND	ND	ND	
Acetate	3	0.32	0.28	0.22	0.2	0.29	0.3	
Formate	3	0.67	0.49	0.54	0.39	0.71	0.7	
Total WOA:	<25	1.53	1.4	1.24	1.52	2.46	2.51	
		•	Cations					
	Maximum		Flu	ıx Type / B	oard Num	ber		
Ionic Species	Contamination Level (µg/in ²)	Flux D / 1 (Act) (µg/in ²)	Flux D / 2 (Act) (µg/in ²)	Flux E / 2 (Act) (µg/in ²)	Flux E / 3 (Act) (µg/in ²)	Flux F / 4 (Act) (µg/in ²)	Flux F / 5 (Act) (µg/in ²)	
Lithium	3	ND	ND	ND	ND	ND	ND	
Sodium	3	1.51	0.48	0.28	0.15	0.72	1.04	
Ammonium	3	1.58	0.95	0.83	0.8	1.26	1.31	
Potassium	3	1.05	0.88	1.17	0.77	1.37	1.46	
Magnesium	1	0.09	0.04	0.03	0.03	0.06	0.05	
Calcium	1	0.2	ND	ND	0.07	0.13	0.09	

 Table 8: Ion Chromatography Results – Activated Flux Samples

ND= None Detected

	Anions & WOA (Weak Organic Acids)							
	Maximum		Flu	ıx Type / B	oard Num	ber		
Ionic Species	Contamination Level (µg/in ²)	Flux D / 1 (Dry) (µg/in ²)	Flux D / 2 (Dry) (µg/in ²)	Flux E / 1 (Dry) (µg/in ²)	Flux E / 2 (Dry) (µg/in ²)	Flux F / 1 (Dry) (µg/in ²)	Flux F / 2 (Dry) (μg/in ²)	
Fluoride	3	0.21	0.28	0.21	0.17	0.37	0.46	
Chloride	4	0.21	0.19	0.15	0.09	0.20	0.22	
Nitrite	3	ND	ND	ND	ND	ND	ND	
Bromide	10	0.04	0.04	0.06	0.10	0.04	0.03	
Nitrate	3	0.05	0.03	0.08	0.05	0.03	0.07	
Sulfate	3	0.32	0.26	0.03	0.35	0.41	0.48	
Phosphate	3	ND	ND	ND	ND	ND	ND	
Acetate	3	0.42	0.39	0.59	0.59	0.53	0.60	
Formate	3	0.89	0.80	0.89	0.88	1.05	0.96	
Total WOA:	<25	2.86	2.09	2.38	1.56	2.36	2.07	
			Cations					
	Maximum		Flu	ıx Type / B	oard Num	ber		
Ionic Species	Contamination Level (µg/in ²)	Flux D / 1 (Dry) (µg/in ²)	Flux D / 2 (Dry) (µg/in ²)	Flux E / 1(Dry) (µg/in ²)	Flux E / 2 (Dry) (µg/in ²)	Flux F / 1 (Dry) (µg/in ²)	Flux F / 2 (Dry) (µg/in ²)	
Lithium	3	ND	ND	ND	ND	ND	ND	
Sodium	3	1.2	0.92	0.18	0.96	1.60	0.99	
Ammonium	3	1.36	1.06	1.00	1.58	1.91	1.15	
Potassium	3	1.14	1.06	0.98	1.47	1.30	1.68	
Magnesium	1	0.09	0.07	0.03	0.07	0.11	0.07	
Calcium	1	0.36	0.18	0.05	0.12	0.48	0.22	

 Table 9: Ion Chromatography Results – Dried Flux Samples

ND= None Detected

Phase 2: SEC Results

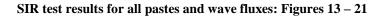
For this test, nine (9) EMC boards as referenced in Table 1 were cleaned using the optimized parameters detailed in Table 5 and returned to the EMC for SEC analysis as per J-STD-001E [3]. SEC test results are detailed in Table 10.

Table 10: SEC Results							
Paste / Flux	Board Number	(µg/in ²)					
Paste A	7	0.02					
Paste B	7	0.05					
Paste C	3	0.07					
Flux D (Act)	3	0.05					
Flux E (Act)	4	0.02					
Flux F (Act)	6	0.06					
Flux D (Dry)	2	0.02					
Flux E (Dry)	3	0.01					
Flux F (Dry)	3	0.02					

All of the values obtained from the SEC tests as well as Ion Chromatography were well below the specifications set forth by the EMS.

Phase 2: SIR and ECM Results

For all wave flux types, the SIR and ECM tests were conducted on both the Act and Dry test coupons only. With regard to the SIR analysis (7 day, $85^{\circ}C/85^{\circ}$ RH), all test coupons met the minimum requirement (>10⁸). With regard to the ECM analysis (500 hours, $65^{\circ}C$, 85° RH), all test coupons met the minimum requirement (Final>Initial/10).





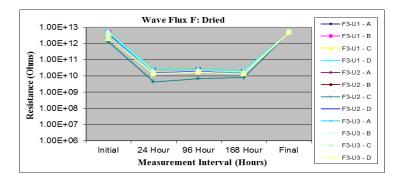


Figure 21: Wave Flux F – Dried

ECM test results for all pastes and wave fluxes: Figures 22 - 30

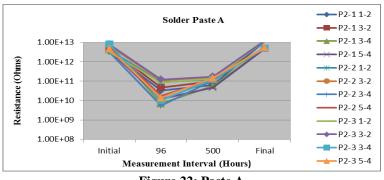
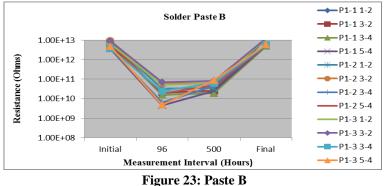
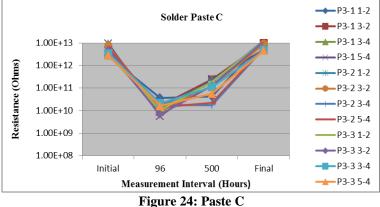


Figure 22: Paste A Minimum Requirement: 2.75E+09 (Pass)



Minimum Requirement: 1.88E+09 (Pass)



Minimum Requirement: 1.33E+09 (Pass)

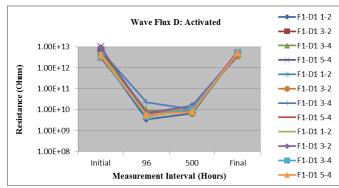


Figure 25: Wave Flux D – Activated Minimum Requirement: 6.41E+08 (Pass)

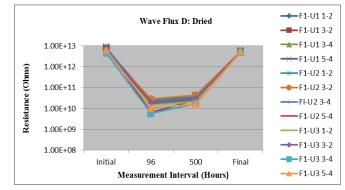


Figure 26: Wave Flux D – Dried Minimum Requirement: 1.57E+09 (Pass)

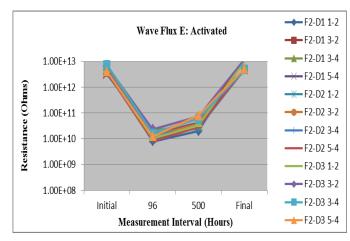


Figure 27: Wave Flux E – Activated Minimum Requirement: 1.32E+09 (Pass)

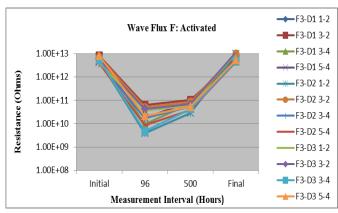


Figure 29: Wave Flux F - Activated Minimum Requirement: 1.86E+09 (Pass)

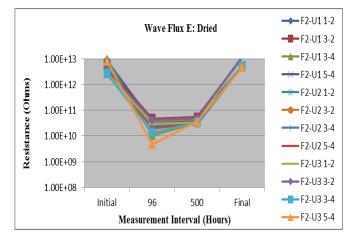


Figure 28: Wave Flux E – Dried Minimum Requirement: 2.24E+09 (Pass)

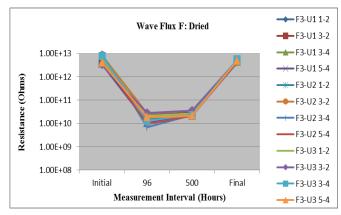


Figure 30: Wave Flux F - Dried Minimum Requirement: 1.68E +09 (Pass)

Phase 2: Compatibility Results

The lead-free label was found to be completely compatible with the cleaning agent under all test conditions. However, the top coat of the leaded label included a varnish base and began to peel during the inline wash process. The top coat was completely removed from the label at the conclusion of the inline trials. The authors recommended the label supplier use the same top coat for the leaded label as used for the lead-free label. The new labels were re-examined and met the EMS's specifications.

Figures 31 and 34 represent the control label that is currently used by the EMS for the lead-free process. This label was baked by passing three times through a reflow oven prior to conducting the compatibility trials.





Figure 31: Before Cleaning

Figure 32: After Cleaning - Maintained Ink Integrity

Figures 33 and 34 represent the label used in the leaded process which included a varnish coating. This label was also baked by passing three times through a reflow oven prior to conducting the compatibility trials.



Figure 33: Before Cleaning



Figure 34: After Cleaning - Varnish removed after five passes through the inline cleaner

There were no significant changes observed with regard to the material compatibility of the components. All the materials were found to be intact with no swelling, brittleness, or cracks when exposed to the cleaning process for a prolonged period.

Tables 11 and 12 detail the results of both the short term (15 minutes of continuous exposure to Cleaning Agent A) and long term (24 hours of continuous exposure to Cleaning Agent A) exposure testing.

ID	Exposure	Chemistry	Conc.	Pre-Test	Post-Test	Change	Remarks
	Time		(%)	Measurement	Measurement	Observed (%)	
A1	15 minutes	Cleaning Agent A	30	67.95 grams	68 grams	+ 0.07	No changes observed
В	15 minutes	Cleaning Agent A	30	2.61 grams	2.63 grams	+ 0.76	No changes observed.
E1	15 minutes	Cleaning Agent A	30	1.44 grams	1.44 grams	0.00	No changes observed

ID	Exposure	Chemistry	Conc.	Pre-Test	Post-Test	Change	Remarks
	Time		(%)	Measurement	Measurement	observed (%)	
A1	24 hours	Cleaning Agent A	30%	67.95 grams	68.12 grams	+ 0.25	No changes observed
В	24 hours	Cleaning Agent A	30%	2.61 grams	2.60 grams	- 0.38	Changed from purple to grey. No other changes observed.
E2	24 hours	Cleaning Agent A	30%	1.48 grams	1.48 grams	0.00	No changes observed

 Table 12: Long Term Compatibility Results – Weight Difference

However, the color of the wire harness changed color from purple to grey during the 24 hour test. This would never be the case in a production environment since these components could not be continuously exposed to a cleaning agent for 24 consecutive hours. Both the EMS and OEM confirmed these test results.

FINAL CONCLUSION & SUMMARY

When building Class III products, it is essential to thoroughly understand the cleaning process and be assured that it has been optimized based on board design, cleaning equipment type, cleaning agent selected, operating parameters used, paste and flux types used and residues generated. The OEM for which this DOE was developed understood the importance of the cleaning process and sought empirical data to design and verify the cleaning process.

Using the EMS's boards and paste and flux details, a DOE was developed enabling ZESTRON to select the most suitable cleaning agent and implement a testing program with cleaning equipment similar to that used by the EMS. Cleaning equipment parameters were optimized and excellent cleaning results were obtained as indicated by visual inspection and ionic contamination analyses. The cleaning process efficiency was verified using EMS boards through Ion Chromatography and SEC analyses. Finally, additional verification regarding the effectiveness of the cleaning process was obtained through the use of SIR and ECM analyses employing IPC test coupons.

Once both the OEM and EMC were assured that the recommended cleaning process met their Class III cleanliness requirements, material and component compatibility testing was conducted. Critical board components were found to be completely compatible; however, the label supplier was required to make a top coat change on one of the two labels used.

Based on the results of this DOE, the cleaning process was implemented at the EMS site and cleaning results were subsequently validated as meeting the OEM cleanliness specifications.

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INFORMATION that INSPIRES INNOVATION

Cleanliness Assessment for Class III Lead-Free No-Clean Assemblies

2013

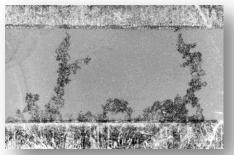
Presented by Umut Tosun, M.S.Chem.Eng. Application Technology Manager u.tosun@zestronusa.com

- Methodology Phase 1
- Results Review Phase 1
- Methodology Phase 2
- Results Review Phase 2
- Conclusion
- Questions & Answers

- Class III electronic products are high performance requiring highest reliability:
 - Medical
 - Military
 - Avionics
 - Automotive



- Residues, either ionic or non-ionic, must be fully removed
- Partially removed or untouched residues can lead to component and product failure from:
 - Dendrite growth
 - Electrochemical migration
 - Electrical leakage currents





- Electronic Manufacturing Service (EMS) provider had an opportunity to manufacture Class III boards for an Original Equipment Manufacturer (OEM) within the medical industry
- OEM requested testing to confirm that the selected cleaning process used by the EMS is capable of removing all flux residues
- EMS teamed with ZESTRON to develop a Design of Experiment (DOE) to validate and qualify the EMS cleaning process

- DOE design included two phases:
 - Phase 1:
 - Determine optimum parameters to effectively clean flux residues from EMS board samples
 - Verify cleanliness:
 - · Visual inspection
 - · Ionic contamination test



- Phase 2:
 - Using the optimum parameters to clean additional EMS boards
 - Using IPC standards, verify cleanliness:
 - Surface Insulation Resistance (SIR)
 - Electrochemical Migration (ECM)
 - Verify compatibility of all critical components and materials used on the boards

- Methodology Phase 1
- Results Review Phase 1
- Methodology Phase 2
- Results Review Phase 2
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- Three (3) solder paste types:
 - Solder Paste A (leaded no-clean)
 - Solder Paste B (lead-free no-clean)
 - Solder Paste C (no-clean tacky flux)
- Three (3) wave flux types:
 - Wave Flux D (no-clean leaded process)
 - Wave Flux E (no-clean lead-free process)
 - Wave Flux F (no-clean lead-free process)



- Wave fluxes classified:
 - Activated flux (Act)
 - Dried flux (Dry)
 - Activated and dried flux (ActDry)

2013

Cleaning agent selection:

- Based on the micro phase cleaning technology
- Chosen on the basis of its:
 - Ability to solubilize the selected pastes and fluxes
 - Compatibility with PCB materials and components

2013

Phase 1 Test Protocol:

- EMS provided 59 boards:
 - 32 boards were used to determine:
 - > Optimum wash settings including:
 - Cleaning agent concentration
 - Wash temperature
 - Wash exposure time
- Cleanliness assessments:
 - Visual inspection
 - Ionic contamination testing

2013

Phase 1 Test Protocol:

- EMS provided 59 boards:
 - 26 boards were reflowed using the three solder pastes:
 - > 17 were used for defining inline process parameters
 - > 6 were used for Ion Chromatography analysis
 - > 3 were used for SEC analysis
 - 33 boards were soldered with the three wave fluxes:
 - > 15 were used for defining process parameters
 - > 12 used for Ion Chromatography analysis
 - > 6 were used for SEC analysis

2013

20)

	Number of Boards Used				
	Process Development	Ion Chromatography	SEC	Total	
Paste / Board #					
Paste A / 1-9	6	2	1	9	
Paste B / 1-9	6	2	1	9	
Paste C / 1-8	5	2	1	8	
Sub Total Paste:	17	6	3	26	
Flux / Board #					
Flux D / 1-6 (Act)	3	2	1	6	
Flux D / 1-3 (Dry)		2	1	3	
Flux D / 1-2 (ActDry)	2			2	
Flux E / 1-6 (Act)	3	2	1	6	
Flux E / 1-3 (Dry)		2	1	3	
Flux E / 1-2 (ActDry)	2			2	
Flux F / 1-6 (Act)	3	2	1	6	
Flux F / 1-3 (Dry)		2	1	3	
Flux F / 1-2 (ActDry)	2			2	
Sub Total Flux:	15	12	6	33	
Grand Total:	32	18	9	59	



32 boards used for process development trials

Cleaning Process: Initial Parameters				
Cleaning Agent Concentration:	10% to 15%			
Wash Temperature:	125°F to 145°F			
Conveyor Belt Speed:	2.0 to 2.5 ft/min			

- Methodology Phase 1
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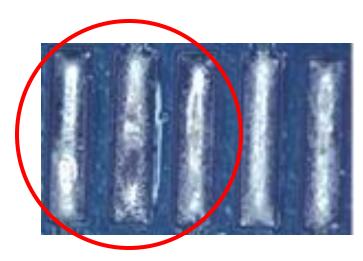
2013

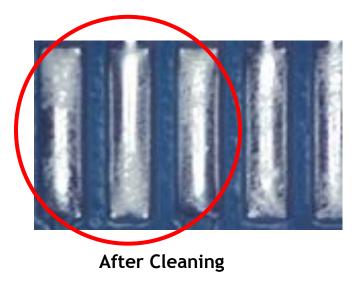
• The cleaning system operating parameters yielding the lowest ionic contamination value for each solder paste:

Test #	Paste / Board #	Cleaning Agent A Conc. (%)	Wash Temp. (°F)	Belt Speed (FPM)	Rinse Temp. (°F)	Ionic Values (μg/inch ²)
1	Paste B / 8	15%	135°F	2.0	140°F	0.00
3	Paste C / 4	15%	135°F	2.0	140°F	0.00
4	Paste A / 9	15%	135°F	2.0	140°F	0.00



Best results Post Reflow Boards Paste A:





Before Cleaning



Best results Post Reflow Boards Paste B:



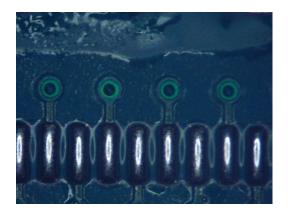
Before Cleaning



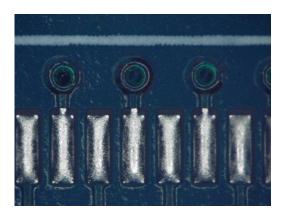
After Cleaning



Best results Post Reflow Boards Paste C:



Before Cleaning



After Cleaning

2013

• The cleaning system operating parameters yielding the lowest ionic contamination value for each wave flux:

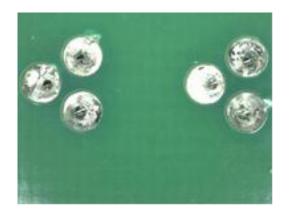
Test #	Flux / Board #	Cleaning Agent A Conc. (%)	Wash Temp. (°F)	Belt Speed (FPM)	Rinse Temp. (°F)	Ionic Values (μg/inch ²)
4	Flux D / 1 (ActDry)	15%	145°F	2.0	140°F	0.08
5	Flux E / 1 (ActDry)	15%	145°F	2.0	140°F	0.07
6	Flux F / 1 (ActDry)	15%	145°F	2.0	140°F	0.09



Best results Post Reflow Boards Wave Flux D (ActDry):



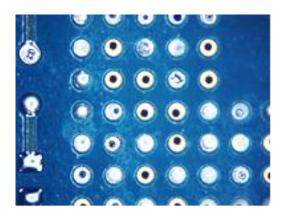
Before Cleaning



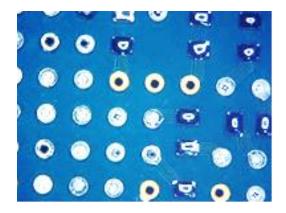
After Cleaning



Best results Post Reflow Boards Wave Flux E (ActDry):



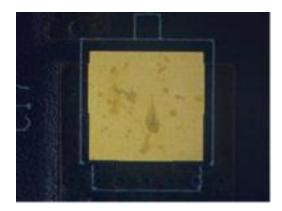
Before Cleaning



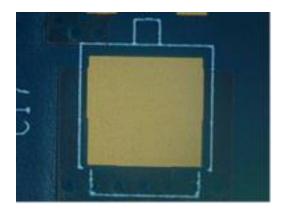
After Cleaning



Best results Post Reflow Boards Wave Flux F (ActDry):



Before Cleaning



After Cleaning

2013

Optimum Inline Cleaning Pr	rocess Parameters
Post Reflow Flux	
Cleaning Agent Concentration:	15% (by volume)
Wash Temperature:	135°F
Wash Exposure Time:	2.6 minutes (2.0 FPM)
Post Wave Flux	
Cleaning Agent Concentration:	15% (by volume)
Wash Temperature:	145°F
Wash Exposure Time:	2.6 minutes (2.0 FPM)
Optimized Cleaning Process Parameters:	
Cleaning Agent Concentration:	15% (by volume)
Wash Temperature:	140°F
Wash Exposure Time:	2.6 minutes (2.0 FPM)

Introduction

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Methodology

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Phase 2 Test Protocol

- IPC test coupons for SIR and ECM analyses:
 - Using optimized parameters:
 - > 18 IPC B24
 - > 9 IPC B25A
- Material compatibility tests:
 - Beaker test:
 - > 30% concentration, 140°F
 - > 15 minutes and 24 hours
 - Inline cleaner test: 13 minute exposure time
 - > 15% concentration, 140°F

Methodology

2013

Phase 2 Test Protocol: Components used for compatibility

Item	Material	ID	Quantity	Description
1	Plastic component	A1, A2, A3	3	 One part used for short-term and long-term testing Second part used for inline testing Third part for reference purposes
2	Wire harness	В	1	• Used for short-term and long-term testing
3	Copper cables	С	1	• Used for inline testing
4	Wire harness	D	1	• Used for inline testing
5	Plastic component	E1, E2	2	 One part used for short-term and long-term testing Second part used for inline testing

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Ion Chromatography - Paste Samples

	A	nions & W	OA (Weak C	Organic Acid	ls)		
	Maximum		Solde	r Paste Type	e / Board Nu	ımber	
Ionic	Contamination	Paste	Paste	Paste	Paste	Paste	Paste
Species	Level	A/5	A / 6	B / 5	B / 6	C / 1	C / 2
	(µg/in ²)	$(\mu g/in^2)$	$(\mu g/in^2)$	(µg/in ²)	(µg/in ²)	$(\mu g/in^2)$	$(\mu g/in^2)$
Fluoride	3	0.23	0.21	0.11	0.09	0.14	0.14
Chloride	4	0.30	0.28	0.12	0.12	0.26	0.25
Nitrite	3	ND	ND	ND	ND	ND	ND
Bromide	10	ND	ND	0.45	0.40	ND	ND
Nitrate	3	0.05	ND	0.04	0.05	ND	0.07
Sulfate	3	ND	ND	ND	ND	ND	ND
Phosphate	3	ND	ND	ND	ND	ND	ND
Acetate	3	0.50	0.50	0.31	0.26	0.28	0.26
Formate	3	0.98	0.94	0.47	0.43	0.62	0.58
WOA:	<25	2.11	2.23	1.18	1.11	1.17	1.18

ND= None Detected

Ion Chromatography - Paste Samples

	Cations										
	Maximum		Solde	r Paste Type	e / Board Nu	ımber					
Ionic	Contamination	Paste	Paste			Paste	Paste				
Species	Level	A/5	A / 6	B / 5	B / 6	C / 1	C / 2				
	(µg/in ²)	$(\mu g/in^2)$	$(\mu g/in^2)$	$(\mu g/in^2)$	(µg/in ²)	$(\mu g/in^2)$	$(\mu g/in^2)$				
Lithium	3	ND	ND	ND	ND	ND	ND				
Sodium	3	0.39	0.33	0.28	0.15	0.11	0.15				
Ammonium	3	1.23	1.28	0.56	0.55	0.66	0.65				
Potassium	3	1.19	1.15	0.70	0.68	0.76	0.72				
Magnesium	1	ND	ND	ND	ND	ND	ND				
Calcium	1	0.41	0.38	0.30	0.29	0.22	0.18				

ND= None Detected

Ion Chromatography - Activated Flux Samples

	A	nions & W	OA (Weak C	Organic Acid	ls)						
			F	lux Type / B	oard Numb	ber					
Ionic	Maximum	Flux	Flux	Flux	Flux	Flux	Flux				
Species	Contamination	D / 1	D / 2	E / 2	E / 3	F / 4	F / 5				
species	Level (µg/in ²)	(Act)	(Act)	(Act)	(Act)	(Act)	(Act)				
		$(\mu g/in^2)$	(µg/in ²)	(µg/in ²)	(µg/in ²)	(µg/in ²)	$(\mu g/in^2)$				
Fluoride	3	0.16	0.12	0.14	0.15	0.13	0.23				
Chloride	4	0.16	0.06	0.31	0.18	0.30	0.21				
Nitrite	3	ND	ND	ND	ND	ND	ND				
Bromide	10	0.15	0.17	0.16	0.18	0.22	ND				
Nitrate	3	ND	ND	0.06	0.04	0.04	0.14				
Sulfate	3	ND	0.03	0.10	0.06	ND	0.07				
Phosphate	3	ND	ND	ND	ND	ND	ND				
Acetate	3	0.32	0.28	0.22	0.2	0.29	0.3				
Formate	3	0.67	0.49	0.54	0.39	0.71	0.7				
WOA:	<25	1.53	1.4	1.24	1.52	2.46	2.51				

ND= None Detected

Ion Chromatography - Activated Flux Samples

	Cations										
	Maximum		F	lux Type / B	oard Numb	er					
Ionic	Contamination	Flux D /	Flux D /	Flux E / 2	Flux E / 3	Flux F / 4	Flux F / 5				
Species	Level	1 (Act)	2 (Act)	(Act)	(Act)	(Act)	(Act)				
	(μg/in ²)	$(\mu g/in^2)$									
Lithium	3	ND	ND	ND	ND	ND	ND				
Sodium	3	1.51	0.48	0.28	0.15	0.72	1.04				
Ammonium	3	1.58	0.95	0.83	0.8	1.26	1.31				
Potassium	3	1.05	0.88	1.17	0.77	1.37	1.46				
Magnesium	1	0.09	0.04	0.03	0.03	0.06	0.05				
Calcium	1	0.2	ND	ND	0.07	0.13	0.09				

ND= None Detected

Ion Chromatography - Dried Flux Samples

	A	nions & WO	OA (Weak C)rganic Acid	ls)		
	Maximum		F	lux Type / B	oard Numb	er	
Ionic	Contamination	Flux D /	Flux D /	Flux E / 1	Flux E / 2	Flux F / 1	Flux F / 2
Species	Levels	1 (Dry)	2 (Dry)	(Dry)	(Dry)	(Dry)	(Dry)
	(µg/in ²)	$(\mu g/in^2)$	(µg/in ²)	(µg/in ²)	$(\mu g/in^2)$	$(\mu g/in^2)$	(µg/in ²)
Fluoride	3	0.21	0.28	0.21	0.17	0.37	0.46
Chloride	4	0.21	0.19	0.15	0.09	0.20	0.22
Nitrite	3	ND	ND	ND	ND	ND	ND
Bromide	10	0.04	0.04	0.06	0.10	0.04	0.03
Nitrate	3	0.05	0.03	0.08	0.05	0.03	0.07
Sulfate	3	0.32	0.26	0.03	0.35	0.41	0.48
Phosphate	3	ND	ND	ND	ND	ND	ND
Acetate	3	0.42	0.39	0.59	0.59	0.53	0.60
Formate	3	0.89	0.80	0.89	0.88	1.05	0.96
WOA:	<25	2.86	2.09	2.38	1.56	2.36	2.07

ND= None Detected

Ion Chromatography - Dried Flux Samples

	Cations									
	Maximum		F	lux Type / B	oard Numb	er				
Ionic	Contamination	Flux D /	Flux D /	Flux E /	Flux E / 2	Flux F / 1	Flux F / 2			
Species	Level	1 (Dry)	2 (Dry)	1(Dry)	(Dry)	(Dry)	(Dry)			
	(µg/in ²)	$(\mu g/in^2)$	(µg/in ²)							
Lithium	3	ND	ND	ND	ND	ND	ND			
Sodium	3	1.2	0.92	0.18	0.96	1.60	0.99			
Ammonium	3	1.36	1.06	1.00	1.58	1.91	1.15			
Potassium	3	1.14	1.06	0.98	1.47	1.30	1.68			
Magnesium	1	0.09	0.07	0.03	0.07	0.11	0.07			
Calcium	1	0.36	0.18	0.05	0.12	0.48	0.22			

ND= None Detected

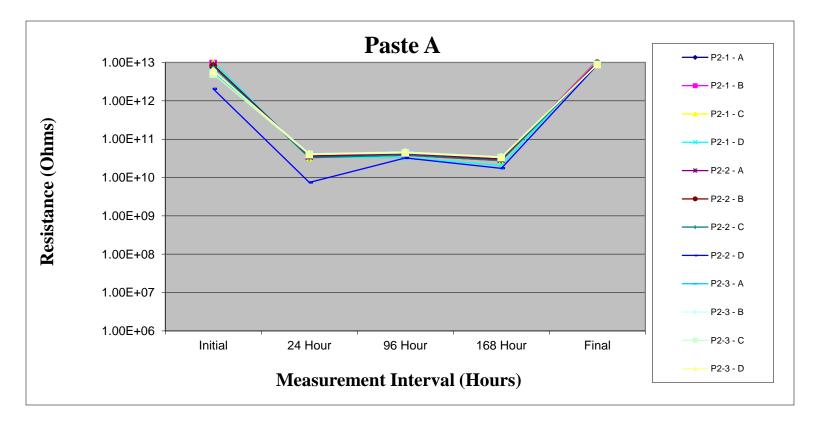
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SEC Results

Paste / Flux	Board Number	(μg/in ²)		
Paste A	7	0.02		
Paste B	7	0.05		
Paste C	3	0.07		
Flux D (Act)	3	0.05		
Flux E (Act)	4	0.02		
Flux F (Act)	6	0.06		
Flux D (Dry)	2	0.02		
Flux E (Dry)	3	0.01		
Flux F (Dry)	3	0.02		

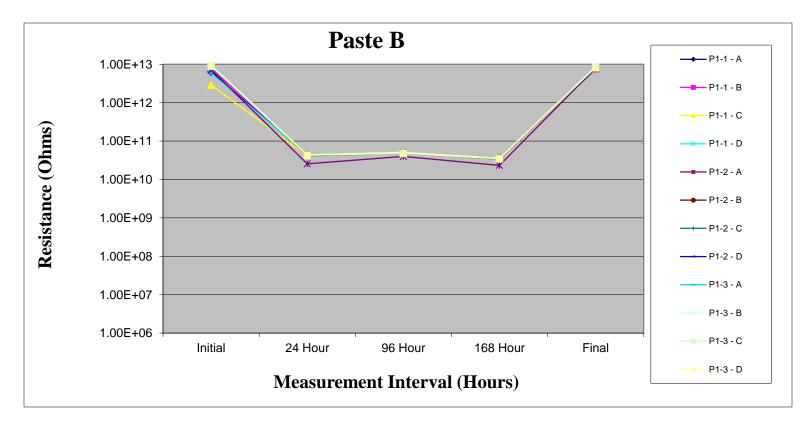


SIR - Solder Pastes



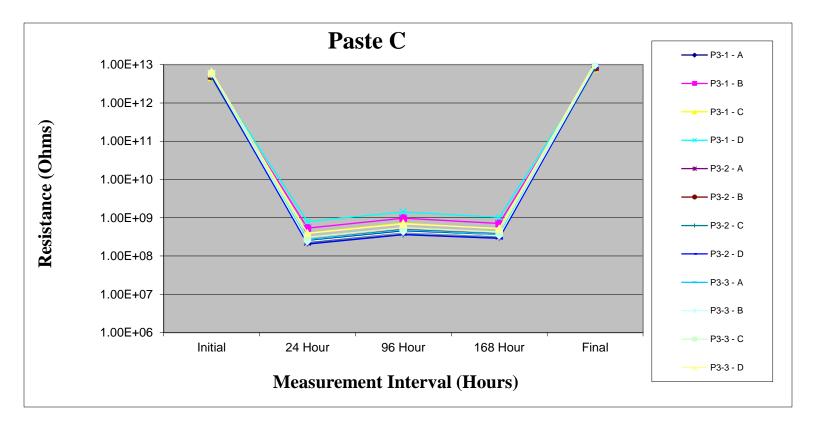


SIR - Solder Pastes

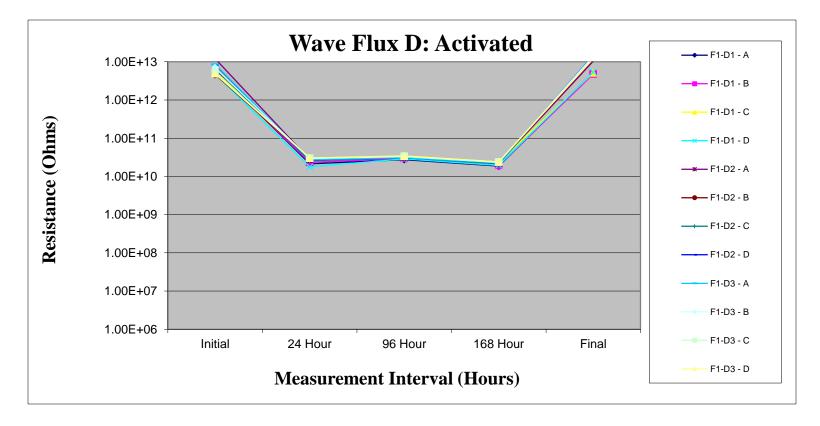




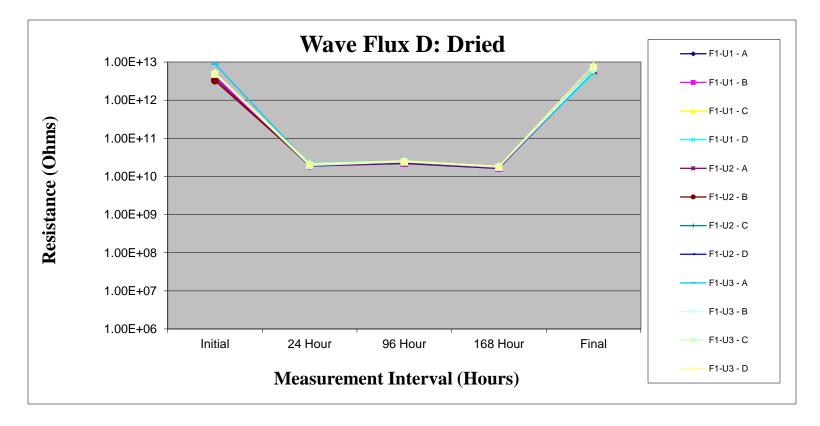
SIR - Solder Pastes



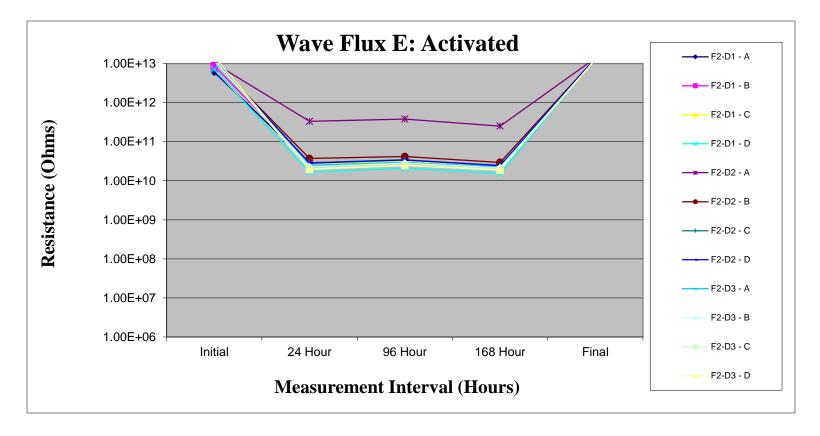




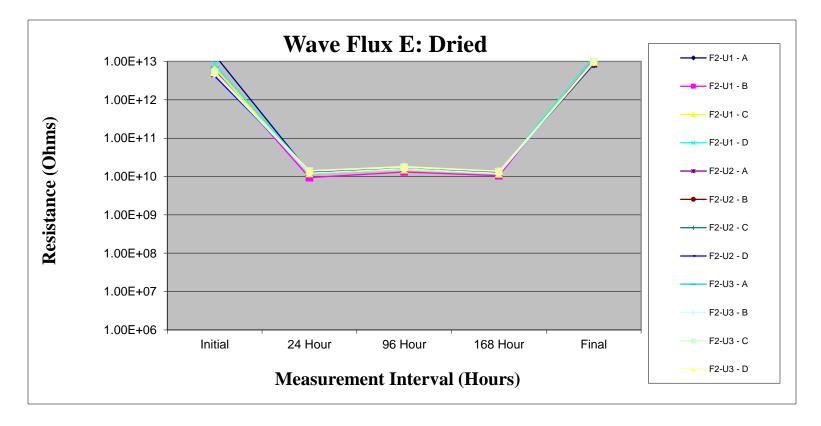




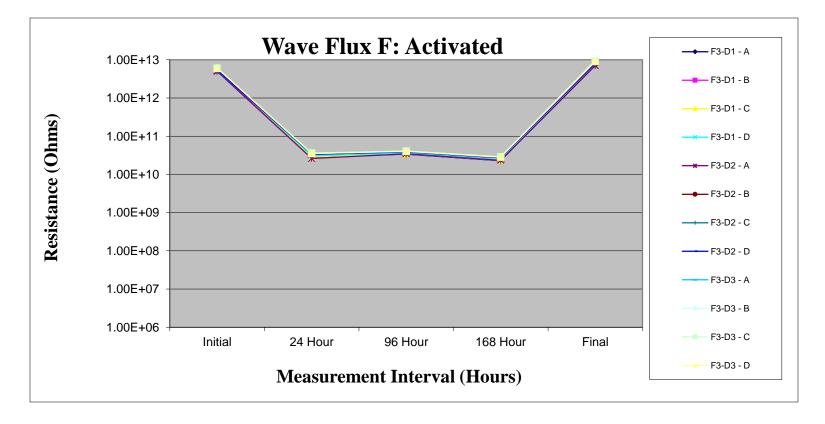




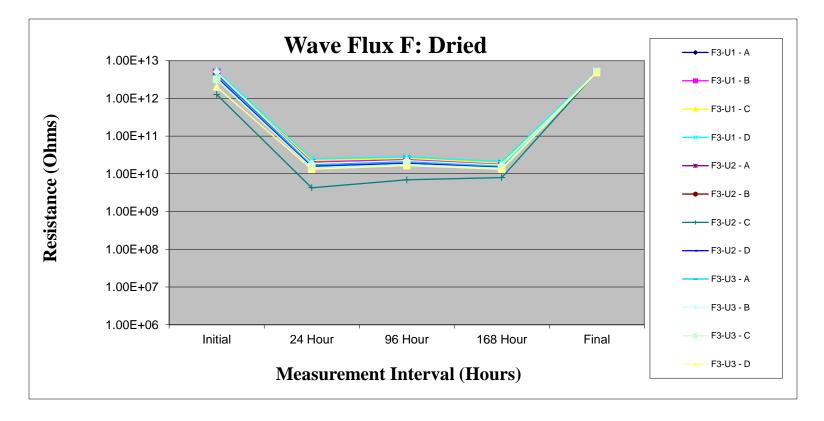








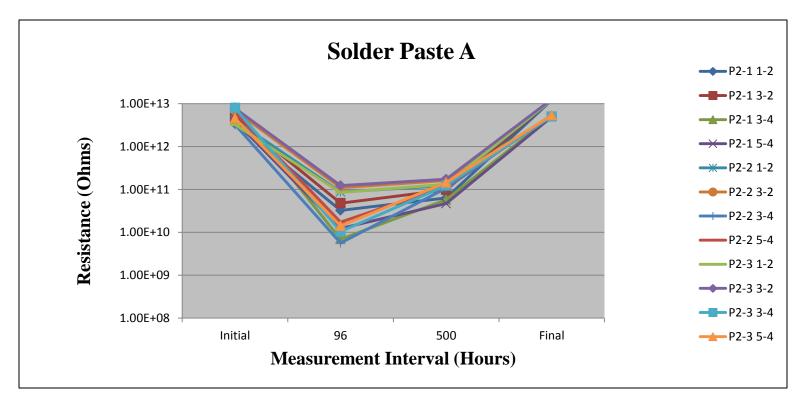




Results Review – Phase 2

2013

ECM - Solder Pastes

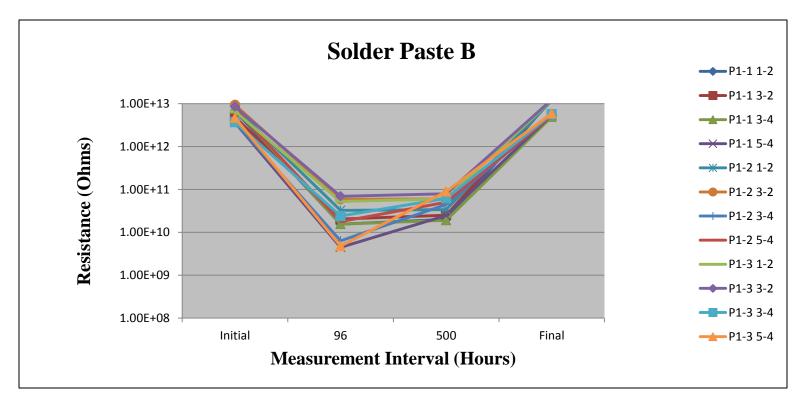


Minimum Requirement: 2.75E+09 (Pass)

Results Review – Phase 2

2013

ECM - Solder Pastes

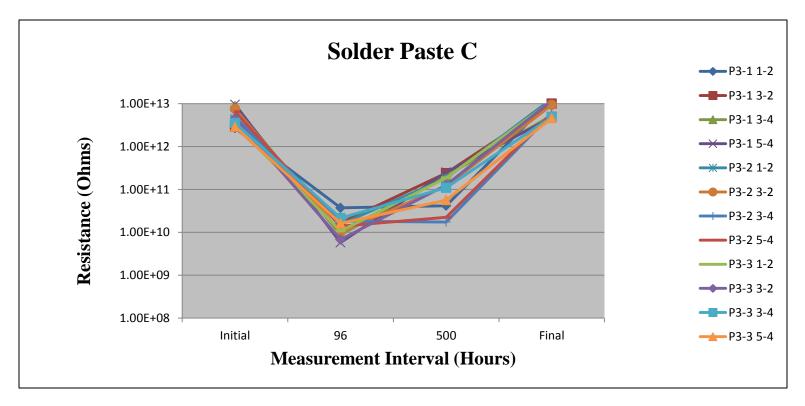


Minimum Requirement: 1.88E+09 (Pass)



2013

ECM - Solder Pastes

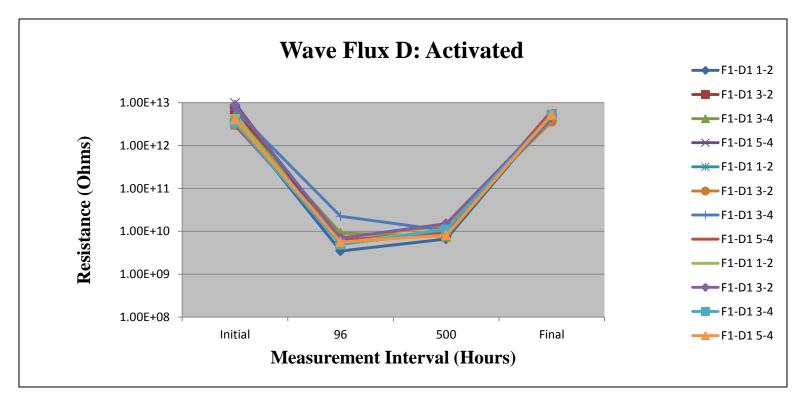


Minimum Requirement: 1.33E+09 (Pass)

Results Review – Phase 2

2013

ECM - Wave Fluxes

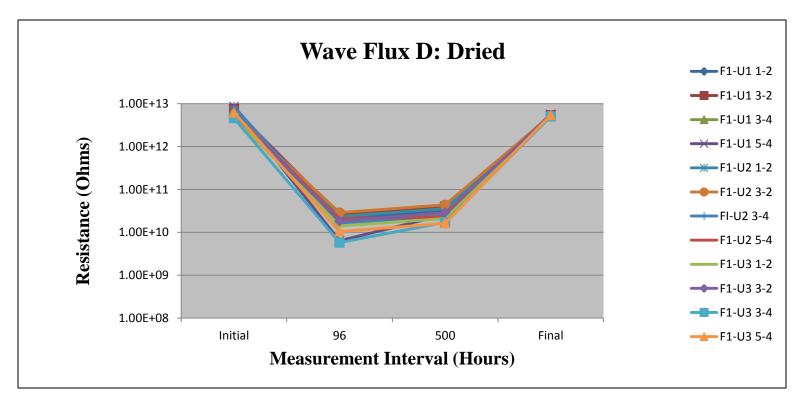


Minimum Requirement: 6.41E+08 (Pass)

Results Review – Phase 2

2013

ECM - Wave Fluxes

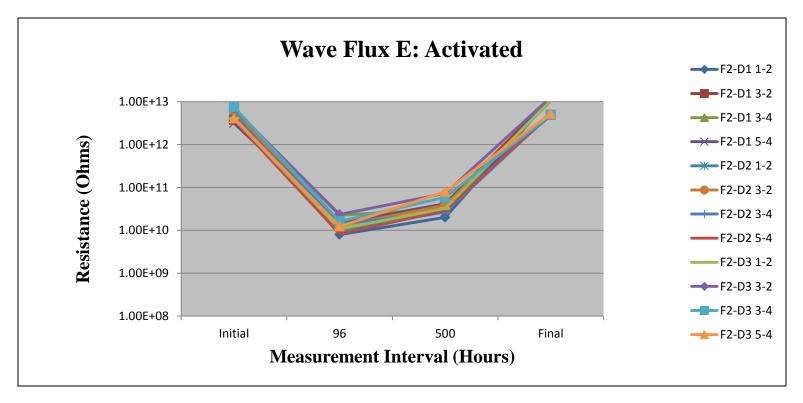


Minimum Requirement: 1.57E+09 (Pass)

Results Review – Phase 2

2013

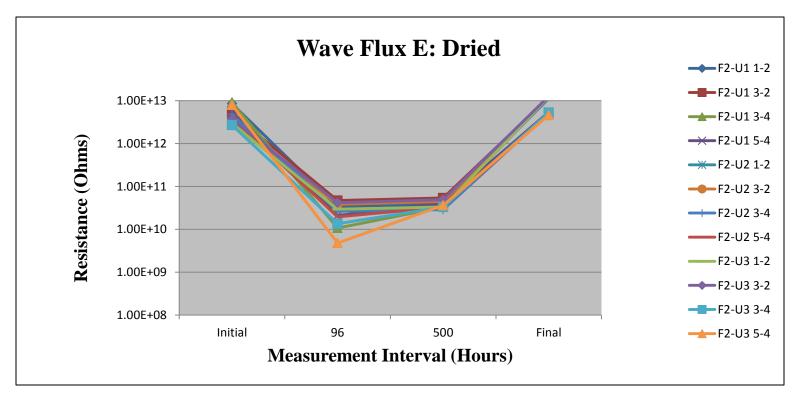
ECM - Wave Fluxes



Minimum Requirement: 1.32E+09 (Pass)



ECM - Wave Fluxes

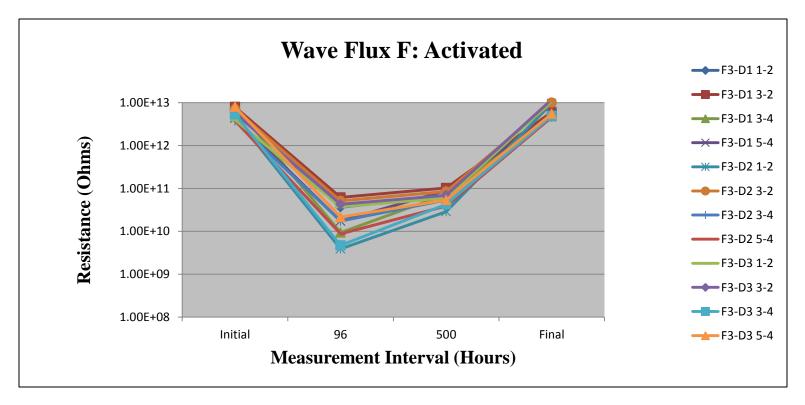


Minimum Requirement: 2.24E+09 (Pass)

Results Review – Phase 2

2013

ECM - Wave Fluxes

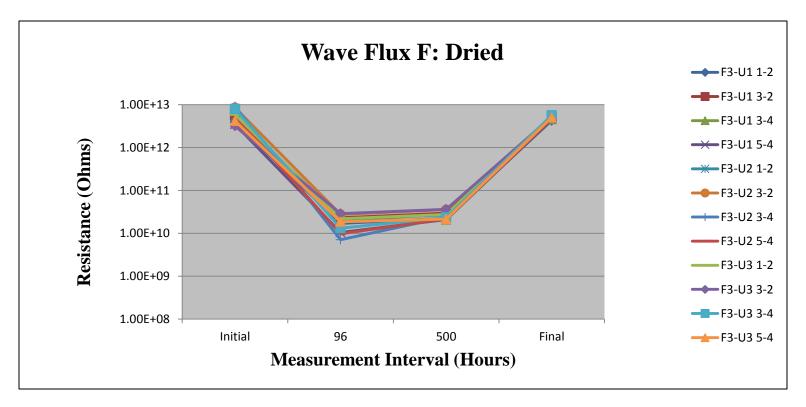


Minimum Requirement: 1.86E+09 (Pass)

Results Review – Phase 2

2013

ECM - Wave Fluxes



Minimum Requirement: 1.68E+09 (Pass)

Label Compatibility

- Lead-free label:
 - Completely compatible under all conditions
- Leaded label:
 - > Top coat varnish base peeled during the wash process
 - Recommended using lead-free top coat
 - > Change was made and met EMS specifications



Label Compatibility - Lead-free label



Before Cleaning



After Cleaning

Label Compatibility - Leaded label



Before Cleaning



After Cleaning

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Component Compatibility: Short term results - weight difference

ID	Exposure Time	Chemistry	Conc. (%)	Pre-Test Measurement	Post-Test Measurement	Change Observed (%)	Remarks
A1	15 minutes	Cleaning Agent A	30 %	67.95 grams	68 grams	+ 0.07%	No changes observed
В	15 minutes	Cleaning Agent A	30%	2.61 grams	2.63 grams	+ 0.76%	Purple color faded out. No other changes observed.
E1	15 minutes	Cleaning Agent A	30%	1.44 grams	1.44 grams	0.00%	No changes observed

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Component Compatibility: Long term results - weight difference

ID	Exposure Time	Chemistry	Conc. (%)	Pre-Test Measurement	Post-Test Measurement	Change Observed (%)	Remarks
A1	24 hours	Cleaning Agent A	30%	67.95 grams	68.12 grams	+ 0.25%	No changes observed
В	24 hours	Cleaning Agent A	30%	2.61 grams	2.60 grams	- 0.38%	Changed from purple to grey. No other changes observed.
E2	24 hours	Cleaning Agent A	30%	1.48 grams	1.48 grams	0.00%	No changes observed

Introduction

- Methodology Phase 1
- Results Review Phase 1
- Methodology Phase 2
- Results Review Phase 2
- Conclusion
- Questions & Answers

Conclusion

- Building Class III products require a high cleanliness level and a thorough understanding of the cleaning process
 - Performance verified by IPC Test Methods and Standards:
 - Visual inspection
 - □ IC, SIR and ECM analyses
 - Optimization based on:
 - Board design
 - Paste and flux types
 - Cleaning agent compatibility

Conclusion

- The cleaning process was implemented at the EMS site
- Cleaning results were validated based on the OEM and EMS specifications



Questions?

2013

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