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# **Cleanliness Analysis**

BEST Project #1818-36 PO #Trade

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Cleanliness Analysis Project #1818-36 Bob Wettermann

# Project Goal

The goal of this project is to determine the level of cleanliness on samples after various cleaning procedures.

# Background / History / Assembly Material

Sixteen test boards were sent in for evaluation of the cleaning procedures.

- IPA control Four test boards
- No cleaning Four test boards
- Water cleaning Four test boards
- IPA cleaning Four test boards

# Analytical Approach

All residues in this evaluation were characterized using Ion Chromatography per IPC-TM-650 method 2.3.28 using the C3 localized extraction and testing system.



U9

BEST-1818-36

10/2/2015 Page **2** of **9** 





# Ion Chromatography Data

	Sample Description	F	$C_2H_3O_2$	$CH_2O_2$	Cl	$NO_2^-$	Br <sup>-</sup>	$NO_3^-$	PO4 3-	SO4 2-	WOA	MSA	Li <sup>+</sup>	Na⁺	$\mathrm{NH_4}^+$	K⁺	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Results	Time(sec)
Foresite recommended limits for Bare Boards		2.5	2.5	2.5	2.0	2.5	2.5	2.5	2.5	3.0	n/a	0.5	2	2	2.5	2	n/a	n/a	Clean	>120
Foresite recommended limits for PCBA (clean)		1	3	3	6.0	3	6.0	3	3	3.0	25	1	3	3	3	3	n/a	n/a	Clean	>120
Foresite recommended limits for PCBA (no clean)		1	3	3	3.0	3	6.0	3	3	3.0	150	1	3	3	3	3	n/a	n/a	Clean	>120
ID																				
1	IPA Control - Board 1 U9	0	0.94	0	0	0	0	0	0	0.05	0	0	0	0	0	0	0	0	Clean	180
2	IPA Control - Board 2 U9	0	0.57	0	0	0	0	0	0	0.04	0	0	0	0	0	0	0	0	Clean	180
3	IPA Control - Board 3 U9	0	0.69	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	Clean	180
4	IPA Control - Board 4 U9	0	0.91	0	0	0	0	0	0	0.01	0	0	0	0	0	0	0	0	Clean	180
5	No cleaning - Board 5 U9	0	20.44	1.34	97.92	1.65	0	18656.12	262.01	279.13	9.91	0	0	4503.39	0	208.99	187.59	0	Dirty	1
6	No cleaning - Board 6 U9	0	19.92	1.77	121.04	1.24	0	19338.31	253.72	199.19	14.14	0	0	4212.21	0	193.72	196.25	0	Dirty	1
7	No cleaning - Board 7 U9	0	24.11	1.52	105.64	1.50	0	19627.14	272.98	293.30	13.25	0	0	4876.62	0	225.97	182.00	0	Dirty	1
8	No cleaning - Board 8 U9	0	20.79	1.91	123.98	1.99	0	20662.79	206.88	395.07	12.62	0	0	5079.09	0	202.81	171.07	0	Dirty	1
9	Water cleaning - Board 9 U9	0	0.54	0	0	0	0	0.28	0	0	0	0	0	1.24	0	0	0	0	Clean	176
10	Water cleaning - Board 10 U9	0	0.69	0	0	0	0	0.78	0	0	0	0	0	1.35	0	0	0	0	Clean	180
11	Water cleaning - Board 11 U9	0	0.58	0	0	0	0	0.65	0	0	0	0	0	1.29	0	0	0	0	Clean	180
12	Water cleaning - Board 12 U9	0	0.57	0	1.66	0	0	0.59	0	0	0	0	0	1.59	0	0	0	0	Clean	180
13	IPA cleaning - Board 13 U9	0	4.98	0	1.98	0.06	0	20.82	1.07	0.54	2.43	0	0	19.51	0	1.25	1.53	0	Dirty	73
14	IPA cleaning - Board 14 U9	0	4.93	0	1.18	0.04	0	29.49	1.21	0.57	3.31	0	0	21.28	0	1.51	1.34	0	Dirty	51
15	IPA cleaning - Board 15 U9	0	4.63	0	1.04	0.08	0	28.44	1.37	0.66	2.05	0	0	18.25	0	1.33	1.71	0	Dirty	53
16	IPA cleaning - Board 16 U9	0	4.27	0	1.52	0.05	0	24.18	1.22	0.27	2.24	0	0	17.71	0	1.08	1.62	0	Dirty	62

# Findings / Conclusions

Samples from the group of no cleaning show very high levels of ionic contamination. The residues found on the IPA cleaning was much higher than the water cleaning supporting the fact that the IPA is not great on polar ionics. The cleaning was done with a solvent or water and a brush indicating that the water is a better cleaning agent if the conditions for brush cleaning are equal.

BEST -- 1818-36

10/2/2015 Page **3** of **9** 



# APPENDIX – FORESITE RECOMMENDED CLEANLINESS LIMITS

		Fluoride	Acetate	Formate	Methane Sulforic Acid	Chloride	Nitrate	Bromide	Nitrite	Phosphate	Sulfate	Weak Organic Acid	Weak Organic Acid	Lithium	Sodium	Potassium	Ammonium	Calcium	Magnesium	C3 - Class 2- 3	C3 - Class 1
All values in µg/in <sup>2</sup>			C <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	CH₂O	MSA	Cl	NO <sub>3</sub> -	Br⁻	NO2 <sup>-</sup>	PO43-	SO42-	SMT hand & selective	Wave direct contact	Li+	Na⁺	K⁺	NH4+	Ca <sup>2+</sup>	Mg <sup>2+</sup>	time / μΑ	time / μA
s	PCB Pre-mask																				۲.
arc	Via or PTH	1																		⊳s∕	l o
B	Soldermask Surface	3	2.5	2.5	0.5	2	2.5	2.5	2.5	2.5	3	n/a	n/a	2	2	2	2.5	n/a	n/a	>1209 250μ	/ 2(
Bare	SMT Pad Area																				SO SO
	Innerlayer																				~
Component	BGA	1	3	1	1	1							n/a	1	2	2	2.5				Ψ'n
	Reballed BGA																	n/a	n/a	ry s∕	00
	Tinned						2	6	2	2	1	25								>120 250	/5
	IC Flip Chip																				sos
	Trayed Component																				×
0	NC Via Top	1		3	1	3														>120s / 250μA	Αų
Ĕ	Solder Area						3						150	3	3	3	3		n/a		8
BA lea	NC SMT		3					6	3	3	3	25						n/a			/ 2
L C C	NC Wave																				S0s
	Rew orked																				×
PCBA (clean)	NC / WSF Via Top	1	3	3	1	6	3	6	3		3		25	3	3	3	3			20s / 50µA	٩
	Selective																				00
	NC / WSF SMT									3		25						n/a	n/a		/5
	NC / WSF Wave																			$\sim \sim$	SOS
	Rew ork / Misprint																				×
Support Hardware	Heat Sink	1	3	3	1	2	3	6			3		n/a	1	1	3	2			Ā	⊲
	Housing / ESD Foam											n/a								20 <sup>μ</sup>	<sup>л1</sup> 0(
	Thermal Material								3	3								n/a	n/a	s / 2	/ 50
	Thermal Pad																			120	.00s
	Battery Housing																			^	

10/2/2015 Page **4** of **9** 



# APPENDIX – DATA DISCUSSION

#### Purpose

Halides present on electronic hardware have varying impacts on product performance and can be introduced throughout the manufacturing process.

# Chloride (Cl<sup>-</sup>)

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 metal migration and electrolytic corrosion, when combined with water vapor and an electrical potential.

#### Chloride on Bare Boards

The amount of allowable chloride on a bare board is difficult to assess. If the board enters an assembly process that incorporates cleaning, then a higher level of chloride can be tolerated. If the bare board enters an assembly process void of cleaning (no-clean), then a more stringent level of acceptable chloride is necessary.

Foresite recommends a maximum chloride level of less than 2.0  $\mu$ g/in<sup>2</sup> for bare boards used in a no clean or water-soluble flux soldering process. With this low level of contamination on the incoming bare boards, the process can be optimized to deal with assembly flux residues.

#### Chloride on Components

Foresite recommends a maximum chloride level of less than 1.0  $\mu$ g/in<sup>2</sup> for plastic plated components. The recommended maximum level does not presently appear in any nationally accepted specification or standard. However, years of failure analysis experience with numerous customers suggest this to be a basis or starting point.

#### Chloride on Assemblies

The tolerance for chloride on an assembly depends on the flux chemistry that is used during manufacture. An assembly processed with high-solids rosin fluxes (RA or RMA) can tolerate higher levels of chloride due to the encapsulating nature of the rosin. Water-soluble fluxes and no-clean fluxes, which flux manufacturers typically formulate using resins or very low levels of rosin, do not have this encapsulating protection. Therefore, they require lower levels of flux on final assemblies.

## Bromide (Br)

Bromide ionic species are generally attributable to the bromide fire retardant additives that are included in the manufacture of epoxy-glass laminate circuit boards and subsequently extracted in the ion chromatography analytical procedure. Bromide can also come from solder masks, marking inks, or fluxes (that have a bromide activator). When from the fire retardant, bromide is not a material that typically degrades the long-term reliability of electronic assemblies. If bromide comes from a flux residue, it can be as corrosive as other halides. The level of bromide varies depending on the porosity of the laminate and/or mask, the degree of over/under cure of the laminate or mask, or the number of exposures to reflow temperatures.

BEST-1818-36

10/2/2015 Page **5** of **9** 



# Bromide on Epoxy-Glass Laminate Boards

For epoxy-glass laminate, bromide levels typically fall with the range of  $0 - 7 \mu g/in^2$ , depending upon the amount of fire retardant added by the laminate manufacturer. Exposure to reflow conditions tends to increase the porosity of the laminate and mask. With

several exposures to reflow conditions, bromide can reach levels as high as  $10 - 12 \mu g/in^2$ .

Foresite does not presently consider bromide levels less than 12  $\mu$ g/in<sup>2</sup> to be detrimental on organic printed wiring boards.

However, we consider levels between 12 – 20  $\mu$ g/in<sup>2</sup> to be a borderline risk for failures if attributable to corrosive flux residues. Furthermore, we consider levels above 20  $\mu$ g/in<sup>2</sup> to be a significant threat for failures if attributable to corrosive flux residues.

# Bromide on Cyanate-Ester Modified (CEM) Laminate Boards

For cyanate-ester modified (CEM) laminate, bromide levels can range anywhere from  $0 - 3 \mu g/in^2$  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  $5 - 7 \mu g/in^2$  with several exposures to reflow conditions.

# Bromide on Polyimide Laminate Boards

For polyimide resin materials, either as rigid laminate or as a flex circuit, bromide levels can range from 0 – 3  $\mu$ g/in<sup>2</sup> depending on the amount of bromide fire retardant added by the laminate manufacturer.

The higher glass transition temperature of polyimide material usually suggests that the resin manufacturer can add less bromide and still maintain the same flame retardant characteristics as other circuit board materials. If bromide levels rise appreciably above 8 –  $10 \mu g/in^2$ , then we suspect the use of a brominated flux or similar fluid.

# Sulfate (SO4<sup>-</sup>)

Sulfate, when present in sufficient quantity, can be harmful to electronic assemblies. Sulfates can come from a variety of sources, such as contact with sulfur-bearing papers or plastics, acid processes in fabrication. However, most often these residues come from tap water rinsing / cleaning processes.

When sulfate levels start rising appreciably above  $3.0 \mu g/in^2$ , we look for a sulfate-bearing chemical in the process, such as sodium/ammonium per sulfate or sulfuric acid. Foresite considers sulfate levels above  $3.0 \mu g/in^2$  to be corrosive and detrimental to circuit reliability. As nitrate has approximately the same electro negativity as sulfate, the sulfate recommendations also apply to nitrate residues.

Another possible source of high sulfate may be solder mask. Some solder mask formulations use sulfur-bearing compounds as fillers, dyes, and matting agents. When the bare board is subjected to the ion chromatography extraction procedure, the sulfates are removed from the mask. As with bromide, if the sulfate residues are extracted from the mask during the IC process, they are not detrimental.

BEST-1818-36

10/2/2015 Page **6** of **9** 



## Weak Organic Acids (WOA)

Weak organic acids, such as adipic, malic, glutaric or succinic acid, serve as activator compounds in many flux systems, especially no-clean and water soluble 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.

#### Weak Organic Acids on Assemblies

WOA levels vary greatly, depending on the delivery method (e.g. foam vs. spray) and the preheat dynamics. In general, water-soluble fluxes have a much lower WOA content than do low-solids (no-clean) fluxes, and the amount of residual WOA is proportional to the amount of residual flux. Bare boards typically do not contain WOA residues. When WOA levels are less than 25  $\mu$ g/in<sup>2</sup> for SMT / hand soldering and below 150  $\mu$ g/in<sup>2</sup> for wave soldering, the residues are generally not detrimental.

Excessive WOA amounts (appreciably greater than 25 or 150  $\mu$ g/in<sup>2</sup>) present a significant reliability threat for finished assemblies. An excessive amount of flux can produce the situation in which the thermal energy of preheat is spent driving off the solvent thereby not allowing the flux to reach its full activation temperature. Un-reacted flux residues readily absorb moisture that promotes the formation of corrosion and the potential for current leakage failures.

## Sodium (Na<sup>+</sup>)

In electronics manufacturing, sodium is found in some fluxes, as the counter ion to the acid activator such as sodium succinate. It is also found in solder mask as absorbed residues and can be conductive through or on top of the mask. Levels less than 3.0  $\mu$ g/in<sup>2</sup> have shown good field performance and good SIR test results.

## Ammonium (NH4+)

Amines used in electronic assembly are from the board fabrication process, such as, etchants, HASL flux residues, some water soluble and solder paste materials.

## Potassium (K<sup>+</sup>)

Potassium in electronics is found in dry film solder mask materials. The amount of potassium found in electronics is typically low, but we have seen levels greater than 3.0  $\mu$ g/in<sup>2</sup> that cause leakage problems.

## Calcium (Ca<sup>+</sup>) and Magnesium (Mg<sup>+</sup>)

These ions are typically found in the solder mask as fillers and rarely come into solution or cause electrical leakage and corrosion problems.

BEST-1818-36

10/2/2015 Page **7** of **9** 



## APPENDIX - C3 PRINCIPLES OF OPERATION

# C3 Testing Extraction

Any production floor or analytical test for cleanliness is only as good as the technique to remove the residue from the surface of the assembly. Process cleanliness relates to the type and level of residues that are able to be brought into solution in critical areas, such as pad to pad or hole to hole on a functioning assembly. It is these residues that the C3 has been designed to assess. The extraction solution has been designed to achieve effective ionic residue removal using a heated delivery system consisting of 3 stages.

- 1. Solution heating and delivery to the extraction site
- 2. Soak and ionization time
- 3. Aspiration of solution to collection cell

This cycle is repeated 9 times to effectively remove the surface residues from a  $0.1 \text{ in}^2$  area, generating a 2.5 ml extraction solution to be used during the testing and afterwards for additional testing.

## C3 Electrical Testing

Using a sacrificial Y-pattern electrode immersed in the collected extraction solution, a 10 volt bias (+/-0.1V) is then applied to the electrode and an internal timer is started to measure the time it takes to achieve a leakage event. The system is measuring the leakage current across the electrode generated by the extraction solution plus the residues extracted from the board surface. A threshold of 250  $\mu$ A has been set to identify when a current leakage event has occurred.

This threshold has been set using a combination of SIR and Ion



Chromatography data. The electrical measurement is determined by assessing the time it takes for the extraction solution and the 10 volt biased electrode to reach a 250  $\mu$ A event.

The system works under the theory that the more corrosive / conductive the residue the faster it will take to achieve this event. The less corrosive or conductive the residue the longer it will take to achieve. We have found that C3 timing results to achieve the 250  $\mu$ A event in less than 120 seconds correlates to corrosive residues as set by Foresite's limits and are identified as Dirty. Timing events that take longer than 120 seconds have correlated to cleaner less corrosive residues and are identified as Clean.



# APPENDIX - ION CHROMATOGRAPHY

# Ion Chromatography

- All testing is performed using a Dionex ICS 3000 ion chromatography system with Chromeleon software.
- Reference control and blank tests are performed before the start of testing and controls are run after every 10 test samples.
- *NOTE:* The system is calibrated using NISTtraceable standards (a six-point calibration is recommended.
- A 1.5mL sample of each extracted solution is analyzed using a sodium bicarbonate and sodium carbonate eluent.

# Extraction Techniques

#### Bag Extraction

- 1. The test samples are placed into clean KAPAK<sup>™</sup> brand (or equivalent heat-sealable polyester film) bags.
- 2. A mixture of isopropanol (75% by volume) and de-ionized water (25% by volume) is introduced into the bags, immersing the test samples (with just enough liquid to cover the sample when the bag is immersed in the water bath).

NOTE: The heat-sealed bags include an opening for ventilation.

- 3. The bags are inserted into an 80°C water bath for one hour. After one hour, the bags are removed from the water bath and test samples are removed from the bags. The test samples are placed on a clean holding rack for air-drying at room temperature.
- 4. A 0.1mL sample of each test sample's extract solution is added to a vial and 0.9mL of DI water is added to the vial. The contents of the vial are analyzed using a 1.4mM sodium bicarbonate/4.3mM sodium carbonate eluent.

## C3 Localized Extraction

- 1. A fresh C3 test cell is used for each extraction site to test and analyze the residue extracted from the identified site.
- 2. Each extraction runs for 3 minutes, collecting 2.5ml of extract solution. The C3 test time is an additional 3 minutes.
- 3. The solution is transferred to a test vial and placed into an auto-sampler which injects the solution into the ion chromatograph for ionic analysis.
- 4. A 1.5mL sample of each test sample's extract is analyzed using a 1.7mM sodium bicarbonate and 1.8mM sodium carbonate eluent.

BEST-1818-36

10/2/2015 Page **9** of **9**