Ion Chromatography Failure Analysis Test Results: EPA's Design for the Environment Printed Wiring Board Program

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1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this report is to present the results and conclusions of CSL's ion chromatography testing on select Design for the Environment (DfE) test vehicles. Using ion chromatography as a tool to analyze boards that failed 85°C/85%RH exposure, we aim to determine if a link exists between board contamination from fabrication and assembly process residues and the reported electrical anomalies.

1.2 SCOPE

This report contains all relevant information regarding the failure analysis, including: identification of test samples, photo documentation of various visual anomalies on the test samples, test methodology, discussion of ion chromatography data, and CSL's conclusions.

2.0 APPLICABLE DOCUMENTS

The Institute for Interconnecting and Packaging Electronic Circuits. "Ionic Analysis of Circuit Boards Ion Chromatography Method." *IPC-TM-650 Test Methods Manual*. Lincolnwood, IL: IPC, 1995.

Iman, Ronald L. Ph.D., and Koon, Jeffry F. *Analysis of Test Results for EPA's Design for the Environment Printed Wiring Board Program.* N.p.: n.p., April 2000.

3.0 TEST SAMPLE IDENTIFICATION

The test matrix consisted of two board groups: DfE boards that fail after exposure to 85°C/85%RH and DfE boards that have not at all been subjected to the 85°C/85%RH environment. The latter group serves as the control for the analysis. Table 2.2 of Iman and Koon's report identifies 10 boards that exhibit various anomalies following 85°C/85%RH testing. We selected these 10 boards for analysis.

For the untested group, we selected 10 boards representing each of the six surface finishes and a variety of assembly processes and sites. Table 3.1 below summarizes the 20 boards selected for ion chromatography analysis.

Table 3.1 Identification of Assemblies Selected for Ion Chromatography Analysis

Board #	Assembly Process	Finish	Site
Untested Boards (Control Group)			
077-4	LR	HASL	1
096-2	WS	HASL	2
061-2	WS	OSP	3
103-4	WS	Immersion Sn	4
068-4	WS	Ni/Au	7
085-4	WS	Immersion Ag	8
074-3	LR	Immersion Ag	9
034-4	LR	Immersion Sn	10
017-4	LR	Ni/Au	12
001-4	LR	Ni/Pd/Au	15
Post 85/85 Exposure (Anomaly Group)			
083-2	WS	HASL	1
056-4	LR	OSP	5
030-4	WS	Immersion Sn	9
032-4	LR	Immersion Sn	8
086-2	WS	Immersion Sn	7
102-4	WS	Immersion Sn	10
082-2	LR	Immersion Ag	11
094-4	WS	Immersion Ag	12
013-1	LR	Ni/Au	13
015-4	LR	Ni/Au	14

4.0 VISUAL OBSERVATIONS

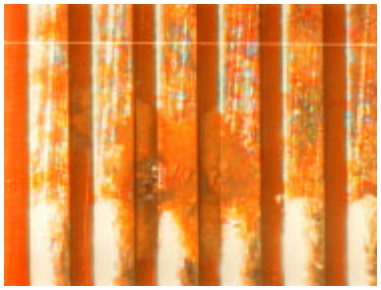
We performed a visual inspection on the 10 tested boards to identify any obvious anomalies or defects. All 10 boards exhibited visual anomalies in varying degrees. The most common anomalies were solder cracking and discoloration of the surface metalization. Less common were pinholes and foreign material (e.g., solder balls). Figure 4.1 shows examples of the more prominent visual defects.

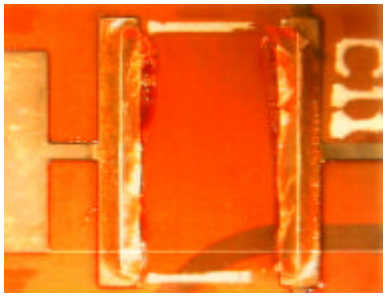
5.0 TEST METHOD

The following describes the fundamental test steps for conducting ion chromatography analysis per IPC-TM-650, method 2.3.28.

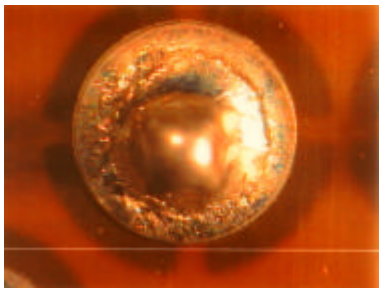
5.1 The lab technician (LT) placed the test board(s) into clean KAPAK™ (heat-sealable polyester film) bag(s).

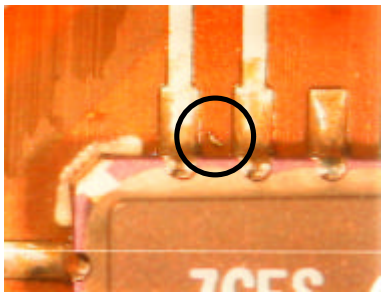
Figure 4.1 Visual Observations of Anomalies on Select Test Boards

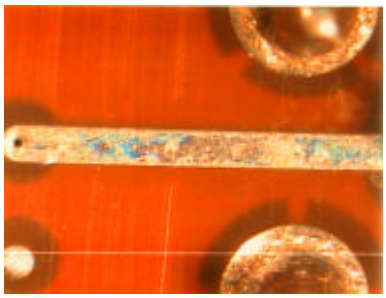
A. 

B. 

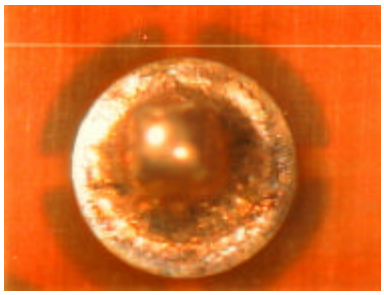
C. 

D. 

E. 

F. 

G. 

H. 

A. Burnt etches near U102 on Board #082-2
B. Excessive WS paste residues beneath SMT component on Board #030-4
C. Pin hole in feed through on Board #056-4
D. Solder crack around perimeter of J5 connector feed through on Board #056-4
E. Solder ball between pins of U3 on Board #013-1
F. Discoloration on trace connected to J6 on Board #015-4
G. Solder crack around perimeter of filled via near C16 on Board #102-4
H. Solder crack around perimeter of J9 connector feed through on Board #086-2

5.2 The LT introduced a mixture of isopropanol (75% volume) and deionized water (25% volume) into the bag(s), immersing the test board(s). NOTE: The heat-sealed bag(s) included an opening for ventilation.

5.3 The LT inserted the bag(s) into an 80^{o}C water bath for one hour.

5.4 The LT removed the bag(s) from the water bath.

5.5 The LT separated the test board(s) from the bags.

5.6 The LT placed the test board(s) on a clean holding rack for air drying at room temperature.

5.7 The LT performed controls and blanks on the Dionex ion chromatography system before the test began. NOTE: CSL uses NIST-traceable standards for system calibration.

5.8 The LT injected a 1.5 ml sample of each test sample's extract solution using a 5 mM sodium bicarbonate eluent.

6.0 TEST RESULTS AND DISCUSSION

The following tables show the ion chromatography data for this evaluation, reported as micrograms of the residue species per square inch of extracted surface (µg/in^2). NOTE: One should not confuse this measure with micrograms of sodium chloride equivalent per square inch, which is the common measure for most ionic cleanliness test instruments.

Table 6.1 Ion Chromatography Anion$^{(-)}$ Data (HASL)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #077-4	LR	1	5.87	3.82	154.33
Board #096-2	WS	2	14.53	10.01	3.01
Tested Boards (Anomaly Group)					
Board #083-2	WS	1	5.36	2.73	7.15

Table 6.2 Ion Chromatography Anion$^{(-)}$ Data (Immersion Sn)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #034-4	LR	10	0.87	5.26	140.45
Board #103-4	WS	4	5.10	2.98	3.30
Tested Boards (Anomaly Group)					
Board #032-4	LR	8	1.75	4.12	15.78
Board #030-4	WS	9	1.70	5.68	15.46
Board #086-2	WS	7	2.99	3.30	9.23
Board #102-4	WS	10	2.33	3.16	4.63

Table 6.3 Ion Chromatography Anion$^{(-)}$ Data (Immersion Ag)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #074-3	LR	9	0.60	6.53	159.48
Board #085-4	WS	8	4.77	2.64	5.22
Tested Boards (Anomaly Group)					
Board #082-2	LR	11	2.59	3.25	4.28
Board #094-4	WS	12	2.53	4.65	5.78

Table 6.4 Ion Chromatography Anion$^{(-)}$ Data (Ni/Au)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #017-4	LR	12	1.01	5.34	150.81
Board #068-4	WS	7	4.57	1.78	3.08
Tested Boards (Anomaly Group)					
Board #013-1	LR	13	2.44	3.56	15.13
Board #015-4	LR	14	1.63	2.80	14.04

Table 6.5 Ion Chromatography Anion$^{(-)}$ Data (OSP)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #061-2	WS	3	3.75	3.45	2.57
Tested Boards (Anomaly Group)					
Board #056-4	LR	5	2.40	4.28	26.41

Table 6.6 Ion Chromatography Anion$^{(-)}$ Data (Ni//Pd/Au)

Sample Description	Assembly Process	Site	Ion Chromatography Data		
Untested Boards (Control Group)			**Cl^-**	**Br^-**	**WOA**
Board #001-4	LR	15	0.84	5.15	151.18

6.1 BROMIDE. Bromide is generally attributable to the bromide fire retardant added to epoxy-glass laminates to give fire resistance and which is subsequently extracted in the ion chromatography analytical procedure. Bromide can also sometimes come from solder masks, marking inks, or fluxes that have a bromide activator material. Bromide, when from the fire retardant, is not a material that typically degrades the long-term reliability of electronic assemblies. If bromide comes from a flux residue, it can be corrosive as other halides can be. 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.

For epoxy-glass laminate, bromide levels typically fall within the range of 0 - 7 $\mu g/in^2$, depending upon the amount of fire retardant the laminate manufacturer has added. 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 $\mu g/in^2$. CSL does not presently consider bromide levels under 12 $\mu g/in^2$ to be detrimental on organic printed wiring boards. However, we consider levels between 12 $\mu g/in^2$ – 20 $\mu g/in^2$ to be a borderline risk for failures if attributable to corrosive flux residues. Furthermore, we consider levels above 20 $\mu g/in^2$ to be a significant threat for failures if attributable to corrosive flux residues.

Based on CSL's guidelines, the bromide levels on the assemblies are acceptably low and as such do not pose a threat for electrochemical failures. We attribute these bromide levels to the fire retardant material in the FR-4 laminate.

6.2 WEAK ORGANIC ACIDS. Weak organic acids (WOAs), 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.

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.

Process	Level
Spray-applied, low solids solder paste deposition	0 – 20 μg/in^2
Foam-applied flux process w/air knife	20 – 120 μg/in^2
Spray-applied, low-solids flux	250 – 400 μg/in^2

When WOA levels are under 400 μg/in^2, the residues are generally not detrimental.

Excessive WOA amounts (appreciably greater than 400 μg/in^2) present a significant reliability threat for finished assemblies. Low levels of WOA can also create electrical performance problems in certain applications.

- 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. Unreacted flux residues readily absorb moisture that promotes the formation of corrosion and the potential for current leakage failures.

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

The observed levels of WOAs on all 20 boards are typical and therefore are not detrimental from an electrochemical standpoint. As expected, more WOA is evident on the boards processed with low residue (LR) fluxes than on those processed with water-soluble (WS) fluxes.

6.3 CHLORIDE. Chloride is one of the more detrimental materials found on printed circuit assemblies. Chloride, which can come from a variety of sources, is most often attributable to flux residues. Chloride will generally initiate and propagate electrochemical failure mechanisms, such as metal migration and electrolytic corrosion, when combined with water vapor and an electrical potential.
The tolerance for chloride on an assembly depends on the flux chemistry that an assembler uses. 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.

CSL recommends a maximum chloride level of no more than 4.5 - 5.0 μg/in^2 for finished assemblies processed with water-soluble fluxes and no more than 2.5 μg/in^2 for finished assemblies processed with low solids (no-clean) fluxes. Although these recommended maximums do not presently appear in any nationally-accepted specifications or standards, years of failure analysis experience dealing with CSL's numerous customers serves as a basis or starting point.

With the exception of the HASL boards, all untested and tested assemblies exhibit levels at or below CSL's recommended guidelines. Therefore, we do not consider the observed chloride levels to be detrimental from an electrochemical standpoint.

The two untested (control) boards with the HASL finish exhibit levels significantly above CSL's recommended limits and are therefore at risk for electrochemical failures. For the board processed with low residue (no clean) flux, we suspect that the high chloride is due mainly to the board fabricator's use of a chloride-activated HASL flux coupled with an ineffective post-HASL cleaning process. For the board processed with water-soluble flux, high chloride may be the result of both HASL residues and water-soluble flux residues. In both cases, ineffective cleaning is the likely culprit.

The one tested HASL board with the reported anomaly exhibits a level only slightly above CSL's recommended limit. Although the chloride in the observed amount places the assembly at slight risk for electrochemical failures, we do not believe in this case that chloride contamination is the root cause for reported open PTH failures on Board #083-2.

Based on the fact the tested boards with known anomalies exhibit levels near or below CSL's recommended guidelines, we can say with reasonable confidence that the anomalies identified in Table 2.2 of Iman and Koon's report are not the result of chloride residues. The majority of the anomalies are either mechanical in nature (e.g., poor solder joint integrity) or component non-conformities (e.g., wrong value and device failures).

7.0 CONCLUSIONS

The observed levels of bromide and WOA on all 20 assemblies are typical and therefore not detrimental from an electrochemical standpoint.

Based on the fact the tested boards with known anomalies exhibit levels near or below CSL's recommended guidelines, we can say with reasonable confidence that the anomalies are not the result of chloride, bromide, or WOA contamination.

From an overall contamination standpoint, the five non-HASL surface finishes tested in this analysis performed as well if not better against the HASL finish.

The few solder joint cracking failures were greater with the HASL finish, than with the alternative finishes. The opens occurred along the interface of the component leads on these older PTH technology boards.