

# PH NEUTRAL CLEANING AGENTS: TECHNOLOGY AND PERFORMANCE

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## ABSTRACT

Effective electronic assembly cleaning is greatly challenged due to component miniaturization, larger component packages, higher lead counts, reduced conductor spacing, tighter pitch and lower standoffs. Added to these geometry challenges, greater use of lead-free solder paste and the increasing trend of multiple thermal cycles result in difficult to remove burnt-in flux residues. However, these challenges are overcome with the use of engineered aqueous-based cleaning agents.

Modern cleaning agents can be alkaline or pH neutral depending on the degree of solvency required. Each are environmentally friendly as they are biodegradable and HAP free. However, in addition to excellent material compatibility and ease of handling, pH neutral formulations have the added advantage of potentially eliminating the need for waste water neutralization.

Effective cleaning processes require an optimized balance of mechanical, thermal, and chemical energy. Cleaning agents provide the chemical energy. As electronic assemblies are completely washed with the cleaning agent, the cleaning agents are formulated with inhibition packages in order to ensure effective material compatibility with the substrate and component materials. To maintain a stable pH value throughout the life of the wash solution, pH neutral cleaning agent formulations contain buffering packages preserving the pH level as flux residues accumulate with the wash solution.

## INTRODUCTION

Achieving the cleanliness requirements for long term reliability is the primary concern when cleaning electronic assemblies. However, it is equally important to clean with a process that is compatible with all electronic substrate materials including components and substrate finishes and labels.

There are numerous examples of successful aqueous engineered cleaning processes that very effectively meet Class III long term reliability requirements. This paper

presents key advantages of a pH neutral cleaning process as an alternative to alkaline processes as well as the challenges of pH neutral formulation for long term stability within the cleaning process.

Finally, a case study is presented featuring Lockheed Martin, Oldsmar, FL whereby a pH neutral cleaning process was evaluated, qualified and implemented. Process and performance data are presented detailing cleaning effectiveness and long-term stability of pH neutral cleaning processes.

Key words: pH Neutral defluxing, low standoff components, environmentally friendly cleaning agents, material compatibility, cleaning agent performance

## WHY PH NEUTRAL (EXCERPT FROM BENCHMARK STUDY: PH-NEUTRAL VS. ALKALINE CLEANING AGENTS [1])

Material compatibility issues between sensitive metals and cleaning solution arise when corrosion – the electrochemical deterioration of a metal due to a reaction with its environment – takes place. To prevent corrosion caused by the very cleaning solution that is meant to safeguard the assembly from corroding in-field and potentially fail, inhibitors come into play. In general, corrosion inhibitors are chemicals that form coordinative chemical bonds with metallic surfaces (adsorption), thereby developing a thin protective layer. They are normally distributed through a solution or by dispersion. Inhibitors slow corrosion processes by either increasing the anodic or cathodic polarization behavior, by reducing the movement or diffusion of ions to the metallic surface or by increasing the electrical resistance of the metal’s surface. Corrosion inhibitors can be classified as either inorganic or organic, with the latter being more prevalent due to solubility advantages, performance, and fewer environmental concerns. Examples of typical corrosion inhibitors are silicates, borates, alkanolamines, naphthalene sulfonic acid, triazoles, carboxylic acids, molybdates, polyols, and phosphate [1].

If the respective cleaning media do not work as intended, several types of corrosion can commonly occur on electronic assemblies, such as gas phase, uniform, pitting, electrolytic metal migration, and galvanic [2]. Fortunately, this has been an area of much research. Electronics manufacturers today have a variety of cleaning choices to prevent such issues with newer, more effective aqueous alkaline chemistries being strongly preferred over solvents or traditional surfactants. Overall choice of aqueous defluxing products available has expanded significantly with the introduction of pH neutral formulations.

### **Selecting Inhibitors**

For all aqueous solutions to do a superior job without affecting sensitive metal substrates (i.e. corrosion control) formulators have to add inhibitors. Studies have shown that choosing the correct type and amount of inhibition chemistry is critically important. Otherwise, the inhibitors themselves can present several problems in the SMT production process.

First, the solubility of certain inhibitors in concentrate chemistry is sometimes low and only a small percentage of the inhibitor can be added to the cleaning product formulation. Therefore, to achieve proper protection of sensitive metals using such problematic inhibitors, a higher recommended operating concentration is often required in the wash tank, which leads to unnecessary chemistry consumption. Lowering the concentration leads to a lower amount of inhibitor available to protect sensitive metals. Second, these organic additives can have detrimental effects on the cleaning process as they also interact with any residue as well as the environment and inhibit the dissolution of such residue into the cleaning fluid. Finally, and most importantly, certain inappropriate inhibitors are tightly bound to the metal surface and are more difficult or impossible to rinse from the substrate surface and under components, where they linger insidiously, causing a host of problems over time. This contamination can adversely increase the electrical resistance of the contaminated areas, lead to conformal coating issues and cause unpredictable failures, thereby threatening the long-term reliability of the assembly.

The type and amount of inhibitors selected is also a function of the pH conditions in the process. Some inhibitors that work well at a certain pH will not function as well or at all if the pH is outside of this range. Therefore, pH-neutral cleaning agents, offer distinct advantages. They require very small amounts of inhibitors because at this pH range (7 +/- 0.5), a unique and customized set of corrosion inhibitors is very effective, thereby solving the problems mentioned above. Due to their lower surface tension (less than 30 mN/m vs. 72 mN/m for DI-water), pH-neutral solutions can penetrate the tiny spaces in and around components, remove contamination even at low concentrations and can be easily rinsed and dried [3]. Furthermore, pH-neutral cleaners are more environmentally friendly and potentially eliminate waste water neutralization processes.

### **BACKGROUND**

Looking broadly at the electronic cleaning industry, the market is dominated by alkaline cleaning products and only recently has the cleaning industry begun to see advanced pH neutral products. We should immediately point out before beginning any discussions of pH neutral vs alkaline cleaning agents that neither product type is completely superior to the other, since different processes have different requirements. To this point, cleaning ability may be the most important factor in selecting a cleaning agent, but we cannot discount safety, local laws, material compatibility, a customer's feelings towards environmental stewardship, etc. Alkaline products dominate the electronic cleaning market for two key reasons: first, alkalinity can aid in cleaning solder pastes and fluxes and second, alkaline products are overall easier to formulate.

To the first point, most solder pastes and fluxes contain a reasonable amount of acids and so a simple acid / base reaction can usually net some cleaning benefits. Additionally, in the case of rosin and some resins, esters (or other functional groups capable of reacting with an alkaline moiety) can be present and alkaline components can aid cleaning through hydrolysis or other similar reaction mechanisms. In this sense, we can then think about the alkaline component as slowly being consumed as it works to clean the electronic part. One may then ask: why then will a cleaning solution remain alkaline throughout the life of the bath if it is slowly being consumed by one of the cleaning mechanisms?

To the second point of alkaline products being easier to produce, with pH neutral cleaners you have lost the previously mentioned cleaning mechanism and secondly one must consider how to keep the cleaner pH neutral. To the first statement, if any given cleaning mechanism is stopped or reduced, another cleaning mechanism must be boosted to maintain performance. So, if the cleaner is no longer alkaline, cleaning has to be boosted from a different set of chemicals in the formula. Regarding keeping the cleaner pH neutral, the system must now be properly buffered and all the stresses (and restocking) the buffer will encounter must be considered. When this is realized, one may ask: why then is the cleaning solution not becoming acidic overtime and instead staying pH neutral?

Here we intend to address both of the previously stated questions. Since the development of our first pH neutral defluxing product, we have had the task of defining some points as well as the charge to answer questions provided by the market on the topic.

### **What is pH neutral?**

To answer the two previous mentioned questions, a few items need to be clearly defined and stated. First, it must be stated what we consider to be pH neutral. A very strict scientific definition for pH neutral would require the concentration of hydroxide and hydronium ions to be present in a solution at exactly the same concentration. Such a strict definition then comes down to a philosophical argument of how many

decimal points one wishes to see after the number 7. Additionally, when considering how the activity coefficient of a salt can affect the pH and how quickly CO<sub>2</sub> adsorbed from the atmosphere can form carboxylic acid in an aqueous solution, in practice it becomes very difficult to realize such a philosophical argument pertaining to pH. However, a looser definition, such as what is used in the consumer industry, would imply anything around pH = 5.5 should be labeled as skin pH neutral. The argument then being a pH of about 5.5 would be very mild and present no harm to your skin, in terms of alkaline or acidic nature of the product. At ZESTRON, for the purpose of product development and since the pH scale is logarithmic, we consider anything up or down by one unit from 7 to be pH neutral.

Secondly it was stated that our pH neutral products are buffered to maintain a stable pH. We must then state that a buffer is a component added to the cleaner which acts to stabilize the pH of the system by resisting pH change to both acidic and alkaline additions to the solution. If a proper level of buffer is added to the cleaner, the cleaner will then maintain a desired pH range. This means that for a buffered pH neutral cleaner, the cleaner actually contains extra components to allow it to resist pH change as it becomes loaded with residues and impurities.

Going back then to the previous two questions of: 1) why will an alkaline cleaning solution remain alkaline throughout the life of the bath if one of the cleaning mechanisms can consume the alkaline components? And 2) why is a pH neutral cleaning solution not becoming acidic overtime instead of staying pH neutral? Both questions can be answered by understanding that the cleaning process is not static but instead a complex process with many moving pieces where the chemistry is constantly being consumed and replenished. During the cleaning process chemistry is constantly being lost through drag out, evaporation, and to a lesser extent, potentially through a cleaning mechanism (if such a mechanism exists for the impurity being cleaned) as well as the concentration of a cleaner is constantly being monitored and refreshed to maintain a desired concentration. To this then, drag out and evaporation both offer an opportunity for fresh chemistry to be added to the bath to maintain a given concentration. Thus, as the cleaning process continues, a type of steady state is achieved where a certain amount of all components of the cleaner will always be present at a given concentration. This means for the alkaline cleaner, if the cleaning mechanism is consuming the alkaline components, the consumed alkaline components are slowly being carried out while fresh components are added. Similarly, for the pH neutral cleaner, fresh buffer is constantly being added as the consumed buffer is drug out.

#### **CASE STUDY – LOCKHEED MARTIN, OLDSMAR, FL**

This site is a military defense, communications, and aerospace contractor. As such, cleaning is essential to their electronics manufacturing process. They employ various cleaning processes that include SIA (Spray-In-Air) systems.

For many years their SIA process successfully utilized an aqueous based inhibited alkaline cleaning agent. However, as electronic assembly designs evolved, this process was challenged with more complex assembly builds increasing the need for material compatibility.

As production capability required a ramp up, an increase in their cleaning capacity was required as well. Thus, new inline cleaning equipment was identified requiring qualification, and this provided the site with an opportunity to evaluate new cleaning agents as well. A key performance indicator for the new cleaning agent was overall effectiveness, that is, it must exceed current cleaning agent performance history. However, given the variety of new materials used for assembly builds, material compatibility with components, labels and substrate surface finish, was key, as assemblies could undergo as many as ten (10) wash cycles.

Finally, in order to minimize cleaning cost as well as the cost of man-hours and line down time required for bath changes, an extended bath life beyond what was achievable with the current cleaning agent was desired.

Concluding an extensive performance evaluation that involved main industry cleaning agent suppliers, the site selected to the pH neutral cleaning agent. Provided that this cleaning agent could meet the performance, material compatibility, and bath life requirements expected, it also offered the added advantage of possibly eliminating the need for process effluent neutralization.

Upon selecting the new pH neutral cleaning agent, a DOE was established and conducted, to complete the necessary product qualification. The initial trials were conducted at the ZESTRON Technical Center. For this analysis, twenty-four (24) CCAs were provided by Lockheed Martin. The boards were divided into five (5) groups and labeled as Group 1 through Group 5.

All substrates were populated, and most were doubled sided. Several assemblies had a metal baseplate. Component types included capacitors, inductors, hybrid wafer filters, vented BGAs, and various size resistors and connectors. The assemblies were aged for up to eight (8) weeks prior to wash trials, increasing the challenge for the cleaning agent. For these substrates, and in accordance with IPC Test Methods, cleanliness assessment techniques utilized included visual inspection, ROSE test, full board and localized ion chromatography.

For visual inspection, as part of the process performance evaluation, high magnification pictures were taken on specifically investigated components before and after the wash process. For post cleaning visual inspection, the specific components were mechanically sheared from the substrate to validate under-component cleaning effectiveness.

Localized extraction conducted using a C3 (Critical Cleanliness Control) system test technique, was utilized in order to create the eluent for ion chromatography analysis. It is also noted that C3 technology incorporates an electrical test developed by the manufacturer that correlates leakage current over time with corrosive residues. Based on standards developed by the manufacturer, test results can classify the test area as ‘Clean’ or ‘Dirty’ [5]. Although the electrical test is not an IPC standard, this result was also noted and reported.

Finally, thirteen (13) SIR test coupons were prepared, cleaned and sent to an independent test lab for analysis in accordance with IPC-TM-650 Method 2.6.3.7.

All substrates utilized for this DOE were assembled utilizing a no-clean solder paste.

### Qualification Cleaning Trial

Lockheed Martin site prepared the assembled substrates and sent them to the ZESTRON Technical Center for the defined cleaning trials. For all trials, an inline cleaner was used that was identical to that selected by Lockheed Martin. The inline cleaner process parameters used for the qualification process are detailed in Table 1.

**Table 1.** Inline Cleaner Process Parameters

Wash Stage	
Equipment	Inline Cleaner
Cleaning Agent (Concentration)	pH Neutral (15%)
Wash Spray Configuration	12 spray bar standard intermix (6 V-Jet & 6 JIC spray manifolds)
Pre-Wash Pressure (Top/Bottom)	40 PSI / 40 PSI
Wash Pressure (Top/Bottom)	80 PSI / 70 PSI
Wash Hurricane Pressure (Top/Bottom)	40 PSI / 40 PSI
Wash Temperature	150°F / 65.55°C
Chemical Isolation Pressure (Top/Bottom)	25 PSI / 25 PSI
Rinsing Stage	
Rinsing Agent	DI-water
Rinse Spray Configuration	4 spray bar enhanced intermix (2 V-Jet & 2 JIC spray manifolds)
Rinse Pressure (Top/Bottom)	80 PSI / 70 PSI
Rinse Hurricane Pressure (Top/Bottom)	40 PSI / 40 PSI
Rinse Temperature	140°F / 60°C
Final Rinse Pressure (Top/Bottom)	25 PSI / 25 PSI
Final Rinse Temperature	Room Temperature
Drying Stage	

Drying Method	Hot Circulated Air
Drying Temperature (D1)	170°F
Drying Temperature (D2)	200°F
Drying Temperature (D3)	200°F

Two conveyor belt speeds were evaluated on the conducted trials. These were 1 fpm and 1.5 fpm.

### Cleanliness Assessment Analytical Test Techniques

One substrate from each board group was selected for cleanliness analysis as detailed in Table 2.

**Table 2.**

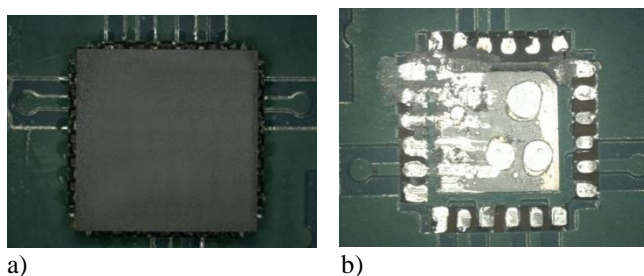
ROSE Testing (4 PCBs)	C3 Electrical Test + IC (5 PCBs)	Full Board Ion Chromatography (5 PCBs)
Group 1	Group 1	Group 1
Group 2	Group 2	Group 2
Group 3	Group 3	Group 3
Group 5	Group 4	Group 4
--	Group 5	Group 5

### Qualification Cleaning Trial Results

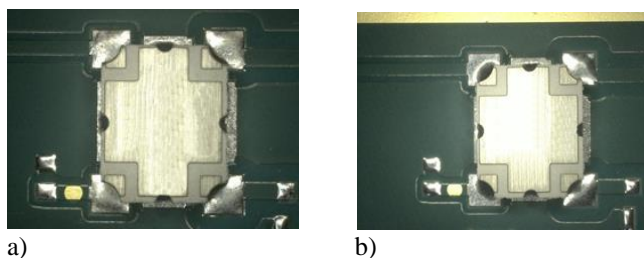
Optimum cleaning results were observed utilizing a conveyor belt speed of 1 fpm. Thus, all results and conclusions that are detailed in this study were derived from the data utilizing the 1 fpm conveyor belt speed.

### Visual Inspection Test Results

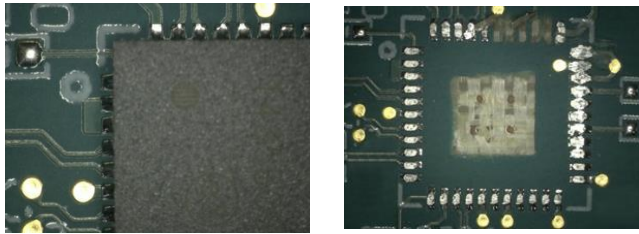
Figures 1 - 5 are representative pictures of specific component areas from each board group before and after cleaning. For under-component pictures, the component was mechanically sheared from the substrate surface. Figure 6 represents the SIR test rack.



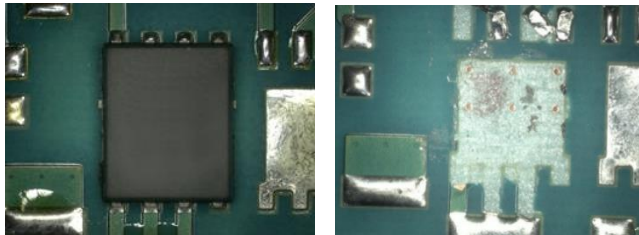
**Figure 1.** Group 1 boards: Qty 5, double sided, aged 5 weeks prior to cleaning. **a)** before cleaning. **b)** after cleaning: under-component



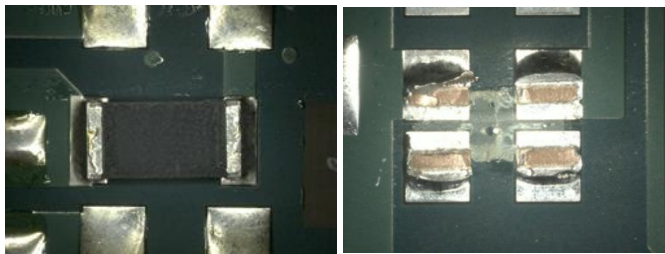
**Figure 2.** Group 2 boards: Qty 5, double sided, aged 3 weeks prior to cleaning. **a)** before cleaning **b)** after cleaning



a) b)  
**Figure 3.** Group 3 boards: Qty 5, double sided, aged 8 weeks prior to cleaning. **a)** before cleaning **b)** after cleaning

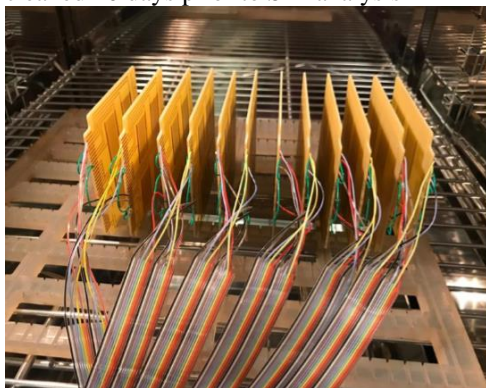


a) b)  
**Figure 4.** Group 4 boards: Qty 4, single side with metal baseplate, aged 4 weeks prior to cleaning. **a)** before cleaning **b)** after cleaning



a) b)  
**Figure 5.** Group 5 boards: Qty 4, double sided, aged 5 weeks prior to cleaning. **a)** before cleaning **b)** after cleaning: under-component

SIR Test Coupons: Qty 13, aged 2 days prior to cleaning & cleaned 10 days prior to SIR analysis



**Figure 6.** SIR Test Rack.

### Ionic Contamination Test Results

All the four (4) CCAs passed the ROSE test since the ionic values were found to be well below the pass/fail limits as per IPC-TM-650 and J-Std-001F standards. Reference Table 3.

**Table 3.**

PCB #	Ion Contamination Values ( $\mu\text{g}/\text{inch}^2$ )	Omegameter Equivalence ( $\mu\text{g}/\text{inch}^2$ )	Ionograph Equivalence ( $\mu\text{g}/\text{inch}^2$ )
Group 1	0.01	0.00	0.01
Group 2	0.06	0.02	0.03
Group 3	0.17	0.06	0.09
Group 5	0.02	0.01	0.01

**Pass/Fail Limit: 10.06 ( $\mu\text{g}/\text{inch}^2$ )**

### Ion Chromatography Test Results – Full Board

This Ion Chromatography evaluation followed the IPC standard IPC-TM-650, method 2.3.28 to characterize process residues. For all ion chromatography tests conducted, the maximum contamination levels indicated for the ion species listed were established through collaboration with top analytical test experts in the electronics industry. The IC values are expressed in  $\mu\text{g}/\text{in}^2$  of substrate surface area.

One substrate from each board Group was analyzed. All reported values are below the allowable maximum contamination levels. Reference Tables 4 – 5 in the appendix for test results.

### C3 Electrical Test and Localized Ion Chromatography Test Results

For this test, four (4) locations on each substrate were selected for analysis. Component types selected per board group are detailed in Table 6.

**Table 6.**

	Location 1	Location 2	Location 3	Location 4
Group 1	QFN	LCC	SOIC	LCC
Group 2	Multi-layer wafer filter	Multi-layer wafer filter	Hybrid wafer filter	Hybrid wafer filter
Group 3	Vented BGA	QFN	QFN	QFN
Group 4	1210	Transformer	Capacitor	SOIC

Group 5	1210	1210	QFN	QFN
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### C3 Results (IC and Electrical)

For Ion Chromatography analysis, all reported values are below the allowable maximum contamination levels. For the C3 analysis, all tests yielded a passing result. Reference Tables 7 - 11 in the appendix.

### SIR Results

All SIR patterns met the minimum requirement of 100 megohms for insulation resistance after 24 hours of temperature and humidity exposure in accordance with IPC-TM-650 Method 2.6.3.7. Reference Figure 7 in the appendix.

### Results Review

Excellent Cleaning results were achieved as exemplified through visual inspection (after component shearing and inspecting under component cleanliness), ROSE test, Ion Chromatography, both full board and localized as well as C3 Electrical Test.

- Visual inspection: all the flux residues were completely removed from underneath all investigated components
- ROSE test: All four (4) CCAs passed the ROSE test as the ionic values were found to be well below the pass/fail limits as per IPC-TM-650 and J-Std-001F standards.
- Ion Chromatography test (full board and localized): all results for the ion species were below the allowable maximum contamination levels per IPC-TM-650, method 2.3.28
- C3 Electrical test: all substrates yielded passing results on the critical components as identified by Lockheed Martin.
- SIR Tests: all results were above the IPC standard per IPC-TM-650 Method 2.6.3.7

### Process Implementation

Based on the DOE test results, Lockheed Martin production site qualified the pH neutral cleaning agent, and the new process was implemented in June 2018. As part of the cleaning process, this site added an automated concentration management system in order to monitor and maintain the desired wash bath concentration.

The qualification process confirmed that the pH neutral cleaning agent met the cleaning requirements specified for the long-term electronic assembly reliability. Once the process was implemented, ongoing ionic contamination analysis was used to confirm the cleanliness assessment and used for historical data comparison. As the neutrality of the cleaning agent was critical to the process performance in terms of meeting the both the cleaning and material compatibility requirements of the substrates, the pH value of the wash bath was monitored and recorded daily in order to confirm consistency.

With any aqueous based cleaning process, wash bath integrity is critical to achieving cleanliness requirements. As the number of substrates cleaned increases, the wash bath will

become saturated with impurities and will require replacement. Thus, in addition to the pH level, other critical wash bath process parameters were monitored and recorded daily in order to ensure that process performance and efficiency were maintained, and wash bath life was maximized. Cleaning process parameters monitored included:

- Concentration: Wash bath concentration can vary due to evaporation and drag out (wash solution carried away on the conveyor belt and/or substrate surfaces). Thus, it is critical to monitor and correct concentration throughout the production process. The new SIA process was installed with a concentration management system that measured and continuously adjusted the concentration by adding cleaning agent concentrate as required in order to maintain the desired concentration.
- Temperature: the optimum wash bath concentration and temperature for this cleaning process was determined through the qualification process. The inline cleaning equipment maintains the proper wash bath temperature and the wash solution concentration monitoring system maintains the required concentration. Each are recorded continuously as the wash process is in operation.
- Conductivity: Measured as mS/cm, this is a measure of ionic residues within the wash bath. Ionic residues are generated through the cleaning process and do accumulate in the wash bath over time. NVR (Non-Volatile Residues) is an indication of contamination [3]. For the fresh wash solution, the NVR value will average 1%. This amount is largely due to the high amount of inhibitors included in the cleaning agent formulation. This value increases as flux loading in the wash solution increases. Based on field experience, as the NVR value approaches 8% or so, the wash bath is deemed to be fully loaded therefore requiring replacement [4].
- Wash bath hours (wash bath life): As the wash bath accumulates residues through the defluxing process, it will become saturated over time and require replacement. Typically, this is evidenced through an increase in ionic contamination levels. As the wash bath becomes loaded with ionics and other impurities, the rinse stage becomes compromised yielding the higher IC values. This will also be evidenced by increases in conductivity values.

Following the implementation, wash bath life was closely monitored and resulted in 555 hours prior to change. During this time, the average wash bath use was 34 hours per week cleaning an average of 794 assemblies per week. Cleaning process data was accumulated throughout the 555-hour wash bath life cycle and is included in this study.

### Lockheed Martin Cleaning Process Data (555 hour wash bath timeframe):

- Highest IC reading: 2.031 ug/in<sup>2</sup> (Reference Figure 8 in the appendix)
- Average pH value: 7.29 (Reference Figure 9 in the appendix)

- Average concentration: 15.22% (Reference Figure 10 in the appendix)
- Wash bath conductivity value (Reference Figure 11 in the appendix)

**Post Implementation IC Tests and Results**

Over the course of the following year, additional IC and C3 testing was performed to re-confirm cleanliness assessment. Six (6) cleaned PCBs were sent to the ZESTRON Technical center for Ion Chromatography evaluation, both full board and localized. Individual board tests are detailed in Table 12.

**Table 12.**

PCB #	Test to Be Conducted
PCB 1	Full Board Ion Chromatography
PCB 2	Full Board Ion Chromatography
PCB 3	C3 Electrical Test + Ion Chromatography
PCB 4	Full Board Ion Chromatography
PCB 5	Full Board Ion Chromatography
PCB 6	C3 Electrical Test + Ion Chromatography

For C3 analysis, three (3) components per PCB were selected. Component types are detailed in Table 13.

**Table 13.**

	Location 1	Location 2	Location 3
C3 test component type:	CLC	QFN	QFN

**Ion Chromatography Test Results – Full Board:**

All reported values are below the allowable maximum contamination levels. Reference Table 14 in the appendix for test results.

**C3 Results (IC and Electrical)**

For Ion Chromatography analysis, all reported values are below the allowable maximum contamination levels. For the C3 analysis, all tests yielded a passing result. Reference Table 15 in the appendix for test results.

**CONCLUSION**

A cleaning system that is properly understood in terms of strains on the system and options for replenishing that system, appropriate levels of each involved raw material can be added such that a stable cleaning process can be realized. This statement then applies to each constituent of the cleaner (if desired to be present): buffers, alkaline components, solvents, inhibitors, etc.

Aqueous based, pH neutral defluxing cleaning agents offer electronic assembly manufacturers excellent cleaning performance and superior material compatibility. Due to the pH neutral formulation, acid neutralization may be avoided

although a monitoring system may be required. Additionally, properly buffered pH neutral formulations will maintain stable pH value throughout the cleaner wash bath life.

As detailed in the case study, Lockheed Martin realized significant benefits through qualifying and implementing the pH neutral cleaning process. Critical process variables were monitored and documented daily throughout a wash bath cycle that lasted 555 hours.

These included:

- Excellent cleaning results as verified by Visual Inspection, ROSE Test (values as compared with their historical data), IC (full board and localized), C3 analysis and SIR analysis;
- Wash bath concentration and conductivity remained steady averaging 15.22% and 2.47 mS/cm respectively;
- Achieved 555-hour wash bath life thereby optimizing the cleaning process efficiency;
- pH value remained steady throughout the wash bath life cycle averaging 7.29.

Additionally, the pH neutral cleaning process provided excellent material compatibility with components surfaces, substrate finish and labels.

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APPENDIX

Table 4.

	Ionic Species	Max Contamination	Group 1	Group 2	Group 3	Group 4	Group 5
ANIONS	Fluoride (F <sup>-</sup> )	1	0.0707	0.0571	0.0310	0.5168	0.0551
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	0.3128	ND
	Chloride (Cl <sup>-</sup> )	6	ND	ND	ND	0.0384	ND
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND	ND
	Bromide (Br <sup>-</sup> )	6	0.0089	0.0137	0.0775	0.3406	0.1005
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	ND	0.2451	ND	0.1207	ND
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	0.0000	0.0000	0.0000	0.0000	0.0000
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	ND	ND	0.0006	ND	ND
	Sodium (Na <sup>+</sup> )	3	0.1081	0.1133	0.0000	0.2221	0.1415
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0998	0.2699	0.1286	1.2941	0.1213
	Potassium (K <sup>+</sup> )	3	0.0000	0.1177	0.0000	0.0000	0.0708
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0000	0.0000	0.0000
	Calcium (Ca <sup>2+</sup> )	n/a	0.0000	0.0000	0.0223	0.0145	0.0000
IC Test Results			Pass	Pass	Pass	Pass	Pass
ND – Not Detected; 0 – Blank value is higher than sample value All values in µg/in <sup>2</sup>							

Table 5.

	Ionic Species	Maximum Contamination Levels	Group 5	Group 1
ANIONS	Fluoride (F <sup>-</sup> )	1	0.0551	0.0707
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND
	Chloride (Cl <sup>-</sup> )	6	ND	ND
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	ND	ND
	Bromide (Br <sup>-</sup> )	6	0.1005	0.0089
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	ND	ND
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	0.0000	0.0000
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	ND	ND
	Sodium (Na <sup>+</sup> )	3	0.1415	0.1081
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.1213	0.0998
	Potassium (K <sup>+</sup> )	3	0.0708	0.0000
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000
	Calcium (Ca <sup>2+</sup> )	n/a	0.0000	0.0000
Ion Chromatography Test Results			Pass	Pass
ND – Not Detected 0 – Blank value is higher than sample value All values in µg/in <sup>2</sup>				

**Table 7.**

		Acceptance Criteria	Group 1			
			Area #1	Area #2	Area #3	Area #4
ANIONS	Fluoride (F <sup>-</sup> )	1	0.0000	0.0000	0.0000	0.0800
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0000
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Bromide (Br <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0000
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.0000	0.0000	0.1260	0.0440
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	0.0000	0.0000	1.2460	1.9040
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	0.0000	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	0.0000	ND	ND	0.0000
	Sodium (Na <sup>+</sup> )	3	0.0740	0.0000	0.0000	0.0000
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Potassium (K <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0320	0.4720
	Calcium (Ca <sup>2+</sup> )	n/a	0.5760	0.4300	0.5820	0.0660
Localized IC Results			Pass	Pass	Pass	Pass
C3 Electrical Test Results		<250µA for 120s or more	Clean	Clean	Clean	Clean

ND – Not Detected      0 – Blank value is higher than sample value    All values in µg/in<sup>2</sup>

**Table 8.**

		Acceptance Criteria	Group 2			
			Area #1	Area #2	Area #3	Area #4
ANIONS	Fluoride (F <sup>-</sup> )	1	0.0280	0.0180	0.0000	0.4040
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	6	0.0000	0.0000	0.0000	2.5460
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	0.0000	0.0280	0.0440	0.1120
	Bromide (Br <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0120
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.1060	0.4060	0.0560	0.2380
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	1.3200	1.5120	1.0480	1.8280
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	ND	0.0000	ND	0.0000
	Sodium (Na <sup>+</sup> )	3	0.0000	0.0000	0.0000	1.7700
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Potassium (K <sup>+</sup> )	3	0.0000	0.0000	0.0000	1.5580
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.8600	0.0000	0.2080
	Calcium (Ca <sup>2+</sup> )	n/a	0.5020	0.2720	0.0100	0.0880
Localized IC Results			Pass	Pass	Pass	Pass
C3 Electrical Test Results		<250µA for 120s or more	Clean	Clean	Clean	Clean

ND – Not Detected      0 – Blank value is higher than sample value    All values in µg/in<sup>2</sup>

**Table 9.**

		Acceptance Criteria	Group 3			
			Area #1	Area #2	Area #3	Area #4
ANIONS	Fluoride (F <sup>-</sup> )	1	ND	0.0000	0.0600	0.0000
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	1.6740	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	0.6400	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0000
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	ND	0.0000	0.0000	0.0000
	Bromide (Br <sup>-</sup> )	6	1.2220	ND	0.0000	0.0000
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	1.2340	0.0000	0.0000	0.0000
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	ND	0.0000	0.0000	0.0000
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	2.5200	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	0.0000	ND	0.0000	0.0000
	Sodium (Na <sup>+</sup> )	3	1.7360	0.0000	0.0000	0.0000
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	1.1460	0.0000	0.0000	0.0000
	Potassium (K <sup>+</sup> )	3	1.1480	0.0000	0.0000	0.0000
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0000	0.0000
	Calcium (Ca <sup>2+</sup> )	n/a	0.0000	0.5400	1.0100	0.6460
Localized IC Results			Pass	Pass	Pass	Pass
C3 Electrical Test Results		<250µA for 120s or more	Clean	Clean	Clean	Clean
ND – Not Detected      0 – Blank value is higher than sample value    All values in µg/in <sup>2</sup>						

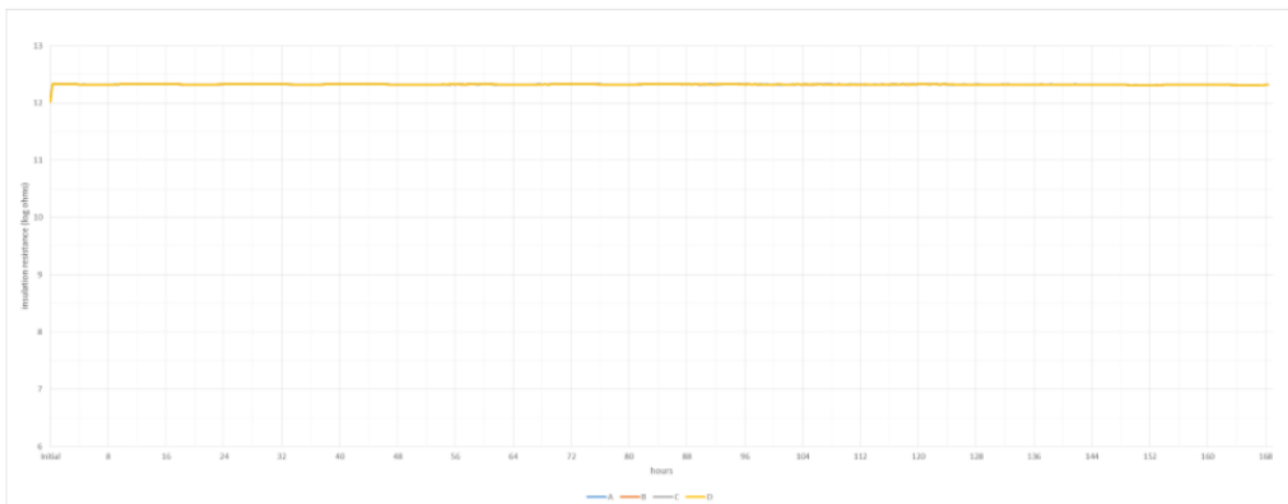
**Table 10.**

		Acceptance Criteria	Group 4			
			Area #1	Area #2	Area #3	Area #4
ANIONS	Fluoride (F <sup>-</sup> )	1	0.0000	0.0000	0.0000	0.0720
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0000
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	0.0000	0.1460	0.0300	0.0000
	Bromide (Br <sup>-</sup> )	6	0.0000	0.0340	0.0000	0.0000
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.0340	0.3620	0.9840	0.0500
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	2.1000	2.4760	0.0000	2.4320
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	1.9000	ND	ND
	WOA (Weak Organic Acid)	25	ND	9.8480	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	ND	0.0040	ND	ND
	Sodium (Na <sup>+</sup> )	3	0.0000	2.3820	0.0000	0.0000
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Potassium (K <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0000	0.0000
	Calcium (Ca <sup>2+</sup> )	n/a	0.7700	0.1060	0.8780	0.1120

Localized IC Results		Pass	Pass	Pass	Pass
C3 Electrical Test Results	<250µA for 120s or more	Clean	Clean	Clean	Clean
ND – Not Detected      0 – Blank value is higher than sample value    All values in µg/in <sup>2</sup>					

**Table 11.**

	Acceptance Criteria	Group 5				
		Area #1	Area #2	Area #3	Area #4	
<b>ANIONS</b>	Fluoride (F <sup>-</sup> )	1	0.0000	0.0000	0.0000	0.0440
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	6	0.0000	0.0000	0.0000	0.0000
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	0.0120	0.0940	0.0300	0.0740
	Bromide (Br <sup>-</sup> )	6	0.0000	0.0120	0.0000	0.0000
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.1820	0.9880	0.0000	0.0180
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	0.7640	1.8280	2.8300	0.5440
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND
<b>CATIONS</b>	Lithium (Li <sup>+</sup> )	3	ND	ND	0.0000	ND
	Sodium (Na <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Potassium (K <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0400	0.0000
Calcium (Ca <sup>2+</sup> )	n/a	0.1040	0.7400	0.9240	0.4100	
Localized IC Results		Pass	Pass	Pass	Pass	
C3 Electrical Test Results	<250µA for 120s or more	Clean	Clean	Clean	Clean	
ND – Not Detected      0 – Blank value is higher than sample value    All values in µg/in <sup>2</sup>						



**Figure 7.**

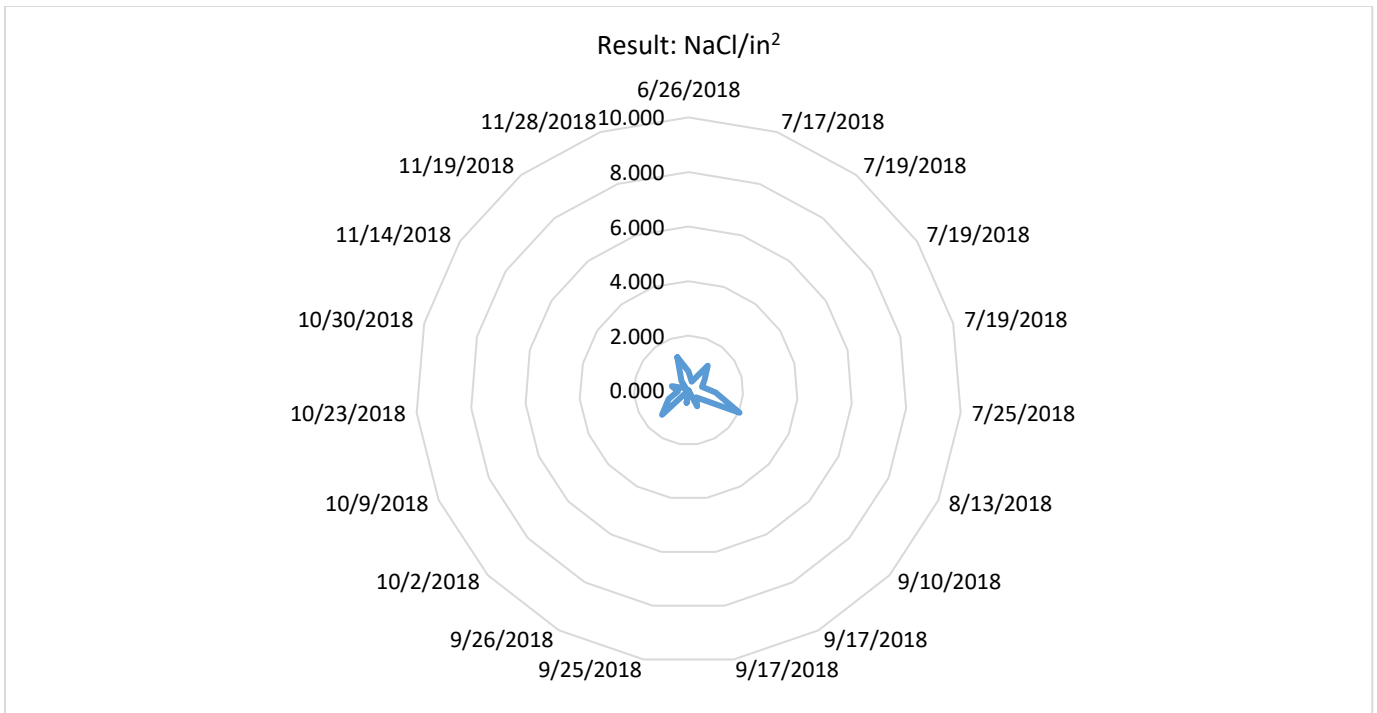


Figure 8. Wash Bath IC Value.

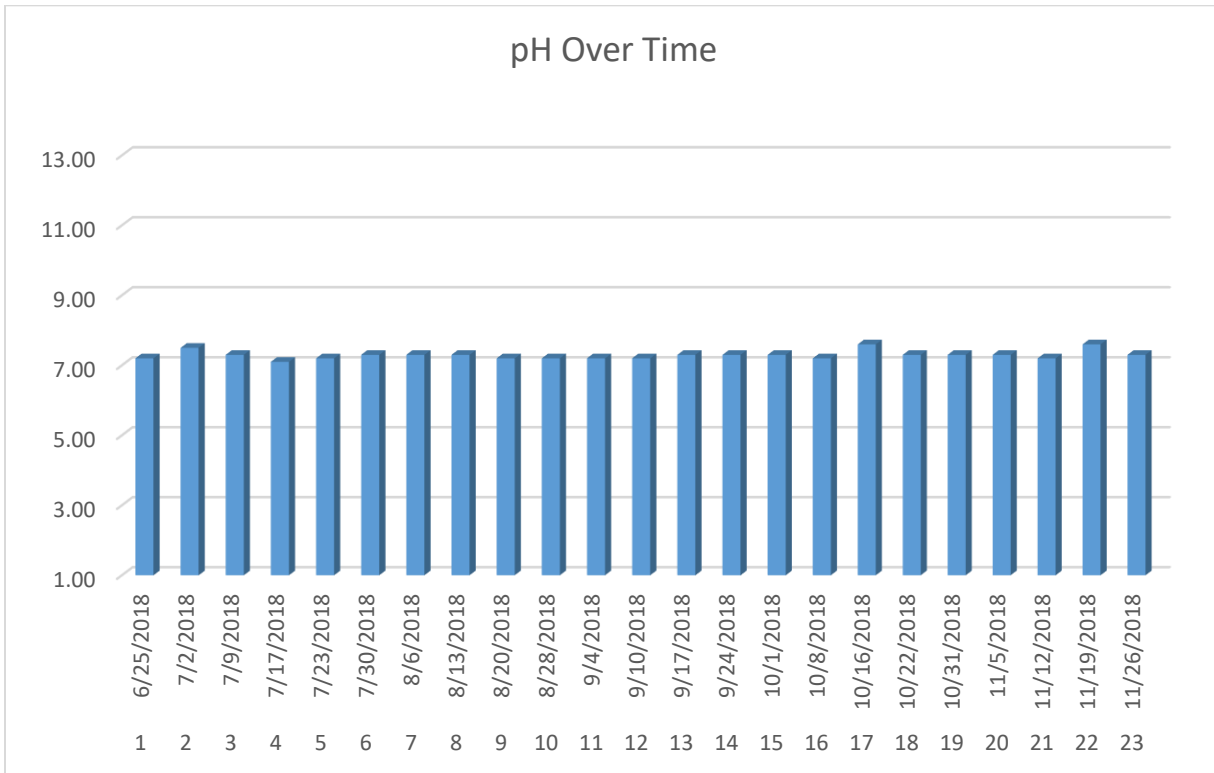
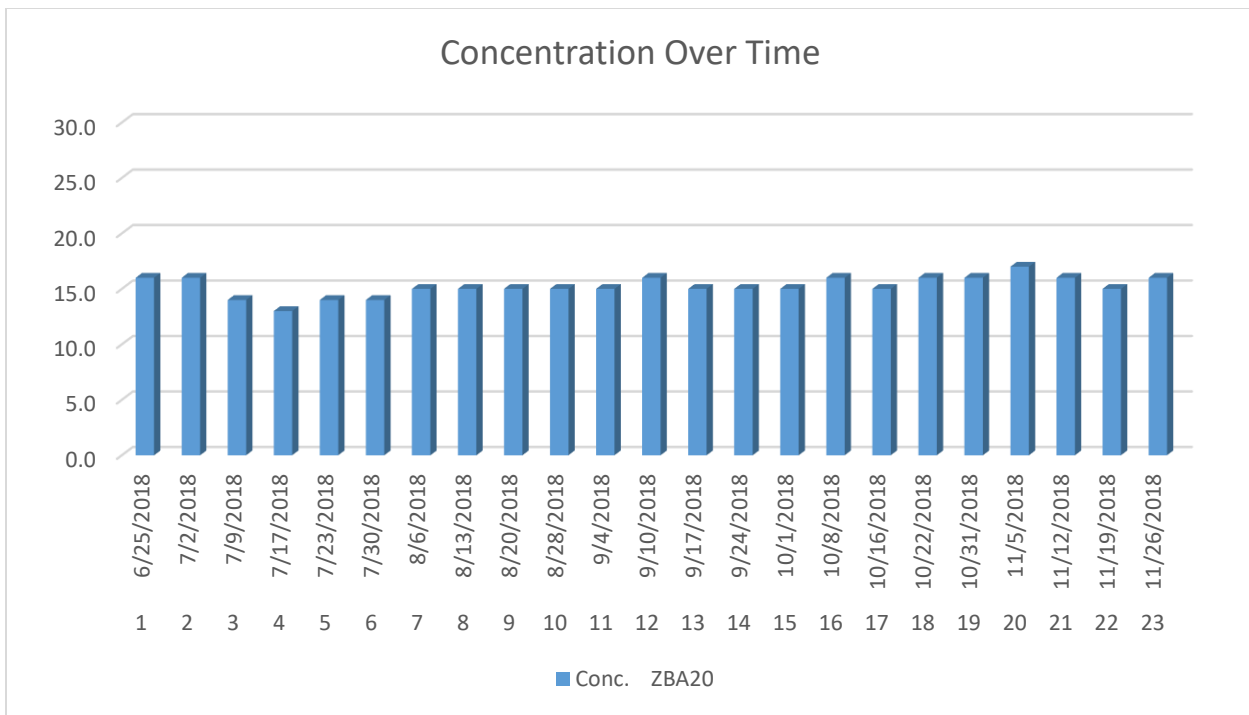
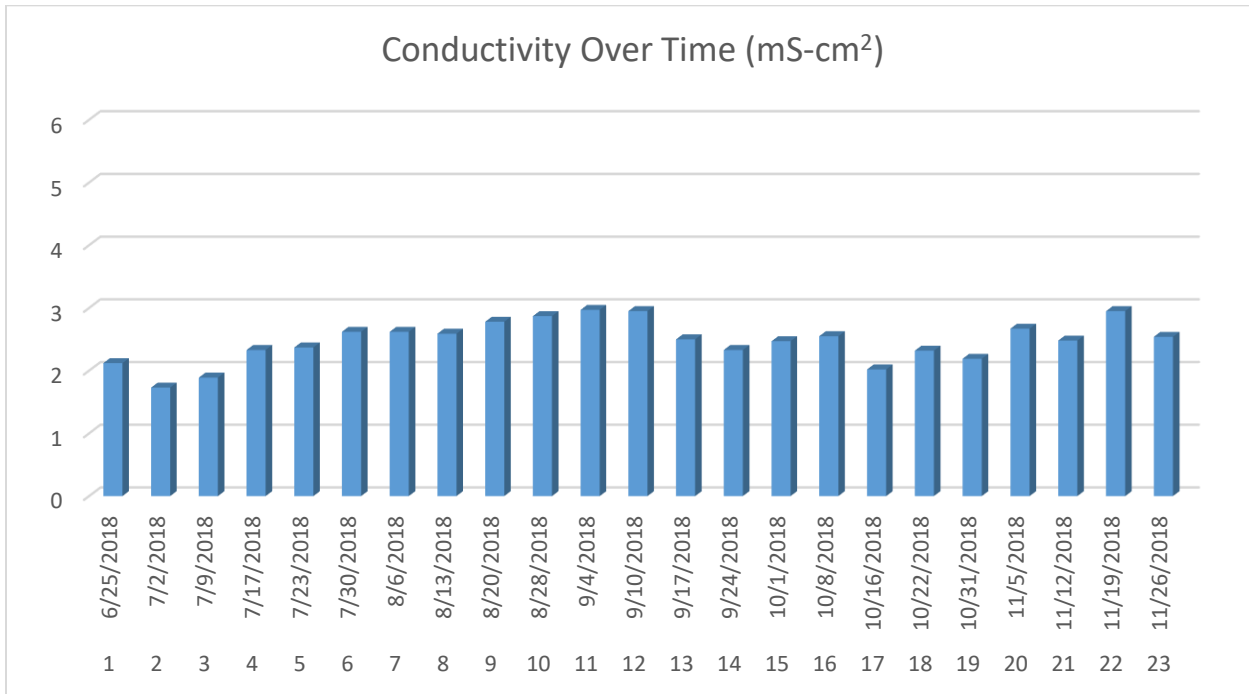


Figure 9. Wash Bath pH Value



**Figure 10. Wash Bath Concentration**



**Figure 11. Wash Bath Conductivity**

**Table 14. Ion Chromatography Test Results**

	Ionic Species	Maximum Contamination Levels	PCB 1	PCB 2	PCB 4	PCB 5
ANIONS	Fluoride (F <sup>-</sup> )	3	0.0445	0.1193	0.2129	0.0487
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	1.8282	0.7712	1.8633	1.2408
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	3	ND	ND	ND	ND
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND
	Bromide (Br <sup>-</sup> )	6	0.3390	0.3655	0.0953	0.1504
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.0337	0.0575	0.0803	0.0568
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	0.0054	0.0000	0.0214	0.0000
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	ND	ND	ND	ND
	Sodium (Na <sup>+</sup> )	3	0.0223	0.0408	0.0137	0.0197
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0067	0.0201	0.0143	0.0000
	Potassium (K <sup>+</sup> )	3	0.0665	0.1052	0.0376	0.0413
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0000	0.0000	0.0000	0.0000
	Calcium (Ca <sup>2+</sup> )	n/a	0.0000	0.0000	0.0000	0.0000
Ion Chromatography Results			Pass	Pass	Pass	Pass
ND – Not Detected      0 – Blank value is higher than sample value All values in µg/in <sup>2</sup>						

**Table 15. C3 Results**

	Acceptance Criteria	PCB 3			PCB 6			
		#1	#2	#3	#1	#2	#3	
ANIONS	Fluoride (F <sup>-</sup> )	3	0.0000	0.0700	0.0000	0.0400	0.0000	0.1120
	Acetate (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> )	3	ND	ND	0.0380	ND	0.0260	ND
	Formate (CHO <sub>2</sub> <sup>-</sup> )	3	ND	ND	ND	ND	ND	ND
	Chloride (Cl <sup>-</sup> )	3	0.0000	0.0120	0.0940	0.0000	0.0000	0.0420
	Nitrite (NO <sub>2</sub> <sup>-</sup> )	3	0.0260	0.0160	0.0740	0.0000	0.0440	0.0100
	Bromide (Br <sup>-</sup> )	6	0.0000	0.0360	0.1260	0.0000	0.1060	0.1440
	Nitrate (NO <sub>3</sub> <sup>-</sup> )	3	0.0000	0.0000	0.0680	0.0000	0.0720	0.0000
	Phosphate (PO <sub>4</sub> <sup>2-</sup> )	3	ND	ND	ND	ND	ND	ND
	Sulfate (SO <sub>4</sub> <sup>2-</sup> )	3	0.5620	0.3840	0.8300	0.4060	0.3200	0.5500
	WOA (Weak Organic Acid)	25	ND	ND	ND	ND	ND	ND
CATIONS	Lithium (Li <sup>+</sup> )	3	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
	Sodium (Na <sup>+</sup> )	3	0.0000	0.3180	0.2460	0.1040	0.0000	0.2740
	Ammonium (NH <sub>4</sub> <sup>+</sup> )	3	0.0000	0.0000	0.3180	0.0000	0.4640	0.0000
	Potassium (K <sup>+</sup> )	3	0.0000	0.1680	0.0142	0.0580	0.0000	0.0160
	Magnesium (Mg <sup>2+</sup> )	n/a	0.0900	0.0100	0.0000	0.0000	0.0000	0.0600
	Calcium (Ca <sup>2+</sup> )	n/a	0.8900	0.5800	0.6020	0.4267	0.5480	0.2760
Localized Ion Chromatography Results			Pass	Pass	Pass	Pass	Pass	Pass
C3 Electrical Test Results		< 250 µA for 120s or more	Clean	Clean	Clean	Clean	Clean	Clean
ND – Not Detected      0 – Blank value is higher than sample value All values in µg/in <sup>2</sup>								