

IMPROVING THE R.O.S.E. PROCESS CONTROL AND QUALITY ASSURANCE METHOD

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ABSTRACT

Quality engineers have long known that cleanliness of printed wiring boards is crucial to the assembly's performance and reliability. The common method for process control at the assembly level is IPC 2.3.25 – Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract (R.O.S.E.). R.O.S.E. testing was highly beneficial since the test provided a quick and easy method for manufacturing technicians to statistically control and monitor the production assembly and cleaning processes. The R.O.S.E. method limitations arise from the methods inability to dissolve many of today's flux residues and to remove and detect ionic contamination trapped under low clearance components. These limitations question the validity of the R.O.S.E. test method as a quality assurance and process control indicator on many of today's leading edge circuit assemblies. To improve the R.O.S.E. method, three areas of research are in work: 1. Extract Solvents, 2. Ion Exchange Resins, and 3. Cleanliness Tester Equipment. The purpose of this research paper is to test the effectiveness of new Ion Exchange Resins for removing both ionic and non-ionic contaminants from extract solvents other than IPA and water mixtures.

KEYWORDS

R.O.S.E., Ionic Cleanliness Testers, Cleaning, Reliability

INTRODUCTION

R.O.S.E. testing was designed to provide a process control method for measuring the level of ionic contaminants remaining on the circuit assembly. Ionic residues derived from plating salts, flux activators, metallic salts, and human perspiration have the ability to conduct electrical current.

The R.O.S.E. method tests the level of ionic residues present by correlating the extraction solvents conductive properties. The ability to conduct electricity can be measured in either conductivity (Siemens/cm or mhos/cm) or by its reciprocal, resistivity (ohms-cm). IPA/H₂O is a poor conductor of electricity, which provides high resistance and low conductance.¹ These desirable extraction solvent

properties allow for ionic contamination to be correlated using a linear proportional relationship of strongly ionizable materials.

This research study focuses on improving the R.O.S.E. method, which is a process control method designed to detect cleaning inadequacies, equipment failures, materials handling, and processing errors.

BACKGROUND

Miniaturization and higher functionality in electronic packaging increase the difficulty for measuring surface residues. When these devices malfunction, it can be attributed to manufacturing residues that were not completely removed.¹ An improved method for monitoring the cleaning process is needed to ensure proper removal of contaminants that may, when exposed to time, temperature, and humidity lead to electrochemical and electromigration.

The R.O.S.E. method was developed in the early 1970s, with the initial procedure flowing a solution of isopropyl-alcohol (IPA) and deionized water over the circuit assembly (Figure 1). Contaminants, dissolved into the IPA/H₂O extraction solution, are measured using a resistivity meter with values assigned based on a sodium chloride (NaCl) standard.

Today, a number of companies offer cleanliness testers. With the pass / fail criteria established, equipment manufacturers developed equivalency factors. The theory behind equivalency factors was to develop commonality between the beaker, static, and dynamic testing methods. Over time, equipment advances built in a number of features for addressing the need to measure increasingly complex circuit assemblies.

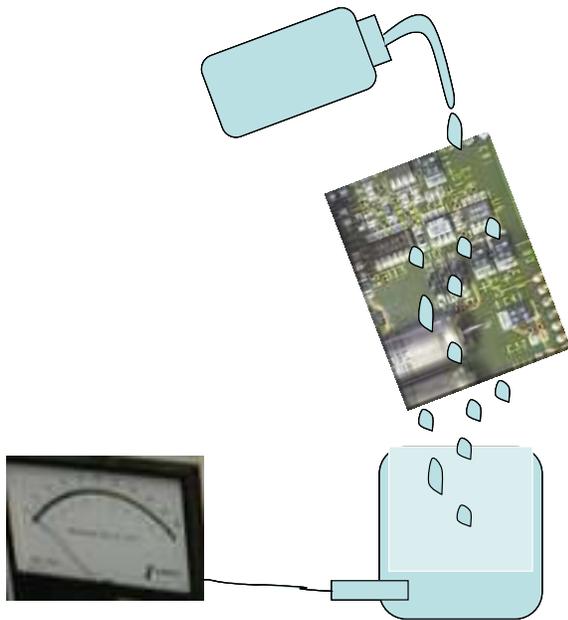
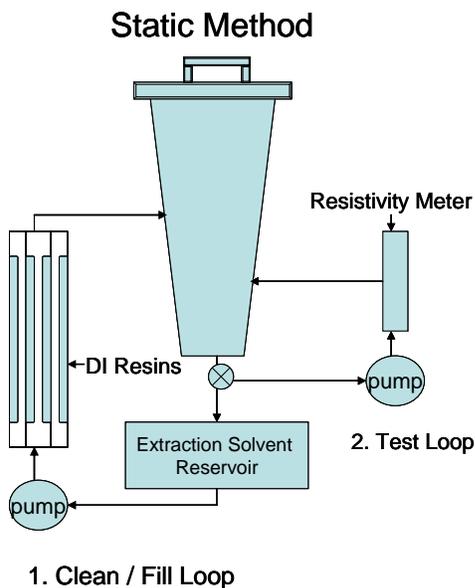


Figure 1: Beaker Test Method

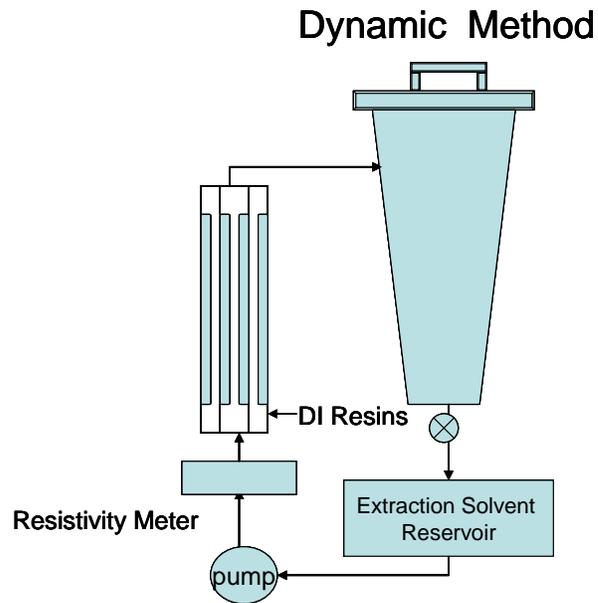
The static method uses a fixed volume when performing the ionic contamination test (Figure 2). In the clean/fill mode, the static system deionizes the extraction solvent from the reservoir and fills the test cell with a known volume. While in the test mode, the static system continuously recirculates the extraction solvent from the test cell, past a resistivity probe, then back into the test cell. Ionic residues are dissolved and distributed throughout a set volume to measure the resistivity drop. The final resistivity measurement is compared to the initial starting resistivity, and the change is correlated to the total ionic contamination. When testing is complete, the clean/fill loop is again used to clean and regenerate the solvent back to a high resistivity before starting the next test.



**Figure 2: Static Test Method
Clean Loop**

The dynamic process takes solvent from the test cell, measures the resistivity, deionizes, and then flows the solvent over the assembly in the test cell (Figure 3). The resistivity reading for the dynamic process starts at a high baseline, drops as the residue is removed, then returns to the high baseline as the solvent is once again deionized. As contaminated solvent is being replaced with clean / deionized solvent, the resistivity will climb back to the initial starting point. The drop in resistivity and the response time to recover back that resistivity value can be used to determine total ionic contamination.

The static and dynamic methods are commonly used in industry. The static method starts at a high point and measures the drop in resistivity. With the dynamic method, the solution is sprayed or immersed and the drop in resistivity correlates the reading to contamination and the time to recovery. Both methods are dependent on the extraction solvents functionality for removing the non-ionic and ionic contaminants left behind following the cleaning process.



**Figure 3: Dynamic Test Method
Clean /Test Loop**

PROBLEM STATEMENT

Miniaturization increases reliability demands as electronic assemblers move upstream from conventional designs and toward threshold and leading edge technologies.² Over the past two decades, conventional surface mount technologies successfully adopted low residue no-clean soldering practices. Today's challenge for printed circuit board manufacturers hinges on high functionality housed in smaller platforms. High performance electronic assembly designs will be driven by multi-chip density, increasing number of I/O's, decreased area array pitches, and tighter component standoff heights.

Higher density, smaller components, and lower standoffs are changing the definition of circuit board cleanliness.³ The current or traditional normal view of quality assurance correlated with visual residue and the resistivity of solvent extract measurements. The problem is that the reduction in component size and low standoff clearances reduces the ability to dissolve, extract, and measure potentially reactive ionic residues. The production process cleanliness test takes on a whole new cleaning definition of removing residue that can be seen visually and residue entrapped under components that is commonly out of sight.

Chip caps, flush mounted to the board, create a flux dam under the component during reflow.⁴ The flux dam seals the underside of the component with flux residues that are difficult to completely remove and detect. Devices placed in tightly packed arrays further increase cleanliness detection difficulty, as there is very limited access for the extraction reagent to reach the contaminant. To dissolve the residue left under chip caps requires both improved extraction reagents and mechanical equipment designs.

The static extraction rate will vary based on the flux make-up, time after reflow, and reflow temperature. Water-soluble flux residues typically dissolve in the IPA/H₂O extraction reagent at a faster rate than do rosin flux residues, which typically dissolve at a faster rate than no-clean flux residues. For many of the new engineered flux compositions, IPA/H₂O extraction solvents are non-effective. Additionally, detection of the flux residue becomes more difficult with the passage of time after reflow. Higher reflow temperatures allow the lower molecular weight solvent molecules to evaporate at a faster rate, leaving higher molecular weight resin molecules, which increases the difficulty of dissolving and detecting the residue.

RESEARCH PURPOSE

Improving the R.O.S.E. method is not a simple replacement of the extraction solvent. The test method requires the process to be integrated with the ion exchange resins and updated cleanliness tester equipment.

The current ion exchange resins are designed for use with IPA/H₂O and may not be compatible with new extraction solvents. Additionally, the current test equipment and ion exchange resins, housings, and plumbing connections are usually constructed on a base of plastics like acrylics and polyvinylchloride ("PVC"), which may not be compatible with new extract reagents and higher temperature processing. To address these limitations, new ion exchange resins and equipment, housed and plumbed appropriately, are needed to support the new extraction solvents.

Ion exchange resins are needed to remove ionizable contaminants dissolved in the extraction solvent during the testing process. Ion exchange resins are needed to remove contaminants and purify the extraction solvent. New test

equipment requires similar methodology to the existing testers. The dynamic conductivity measurement systems requires a test tank, a temperature compensated conductivity cell, ion exchange columns and a pump connected together in a recirculating loop. The conductivity readings must be integrated over time using electronic integration. The ion exchange resins must have the compatibility with heated reagent to accelerate extraction of ionic soils from low clearance components found on surface mount assemblies. The purpose of this research paper is to test the effectiveness of new Ion Exchange Resins for removing both ionic and non-ionic contaminants from improved extract solvents.

EXTRACTION SOLVENTS

Flux residue solubility parameters are directly related to the cohesive energy parameters, which represents the chemical energy required to dissolve the flux residue into the extraction solvent. Solubility theory takes known solvents with established solubility parameters and exposes those solvents to the soils in question. The tests are conducted at static conditions (no heat or energy applied). Applying Hildebrand and Scott's theorem that "like dissolves like," the residue set of soils are immersed into solvents with known solubility parameters. The effectiveness of these solvents for dissolving the soil can be used to calculate a composite solubility parameter for a specific soil.

Each solvent tested has known dispersive, permanent dipole – permanent dipole, and hydrogen bonding properties. Dispersive cohesive interactions occur from the solvent's free energy to dissolve non-polar structures. The permanent dipole – permanent dipole interactions occur from solvents attraction to positive or negative charges in the soils valence electron shell. Hydrogen bonding cohesive energy occurs from the solvent's propensity to share electronics with electronegative atoms such a fluorine, oxygen, or nitrogen. Calculating these properties for a given soil provides a roadmap of the extraction solvent properties needed to dissolve and measure the conductive properties on the board's surface and under components.

One of the complexities associated with this level of testing is that fluxes are typically multiple phase compositions. This typically requires extraction solvents engineered with material properties that dissolve various components present in the flux residue. Since flux compositions contain multiple components, a composite solubility parameter for the residue set can be established. The level of testing provides insight into the material properties of extraction solvents needed to dissolve and measure the soil set.

As circuit assembly innovations occur, flux demands increase. The electronic assembly flux sets used today are very different from the rosin soils used to develop the R.O.S.E. test method. To illustrate this point, Table 1 Shows the grading scale used for the solubility of various solvents tested on flux soils. Table 2 illustrates the visual appearance of the graded test coupons. A grade of 1 equates to excellent

solvent dissolution properties whereas a grade of 6 equates to very poor solvent dissolution properties.

Score	Description
1	Easily dispersed and cleaned
2	Signs of cleaning efficacy with minor residue present
3	Signs of cleaning efficacy with significant residue present
4	Some signs of interaction, with heavy residue present
5	Minor interaction, with heavy residue present
6	No solvent interaction

Table 1: Grading Scale

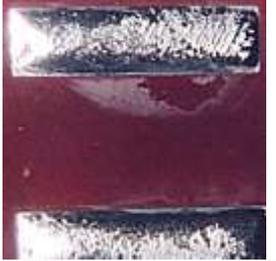
Grade 1	Grade 2
	
Grade 3	Grade 4
	
Grade 5	Grade 6
	Picture to be inserted when I arrive back to the office

Table 2: Grade Illustrations

Figures 4 & 5 illustrate the solubility properties of IPA and H₂O on a large composite of solder materials in which a composite solubility parameter was developed. Both IPA and H₂O were exposed to the soil for one hour at room temperature. The data findings show poor solubility on most of the flux sets commonly used within the assembly process. IPA is very poor at dissolving no-clean flux residues. This finding is worst on the lead-free no-clean flux residues. Water is ineffective on both rosin and no-clean flux residues. Water is valuable for removing ionic residues, but is predicated on IPA effectiveness at dissolving the flux residue present on the assembly.

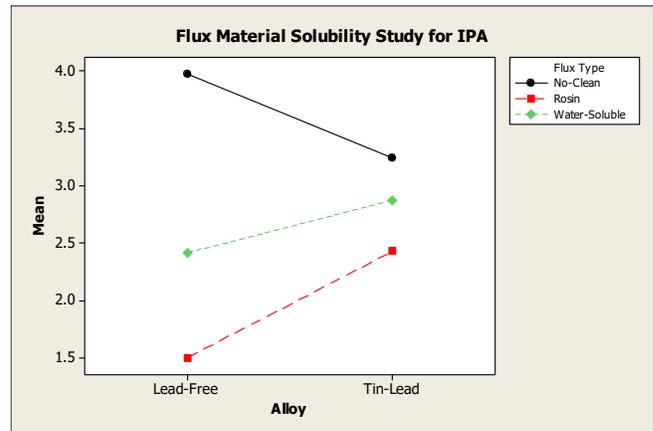


Figure 4: IPA Solubility with Current Flux Residues

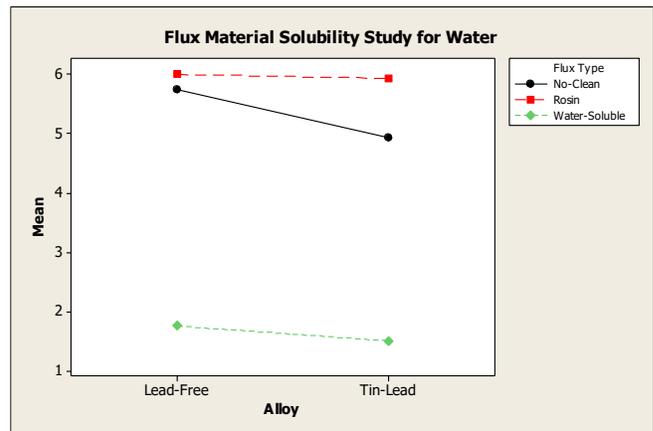


Figure 5: H₂O Solubility with Current Flux Residues

METHODOLOGY

Replacing the R.O.S.E. method is not as simple as finding a better extraction solvent. The water resins used for the current process are not compatible with extraction solvents that show potential for dissolving the no-clean and rosin flux residues. Some of the extraction solvents require higher processing temperatures, which increase compatibility issues.

Ion exchange resins are needed to cleanse the solvent of polar ions dissolved during the testing process. A stoichiometric mixture of ion exchange resins that provide high loading and compatibility with new extraction solvents were tested. These patented resins were tested on 14 solvents that show potential for dissolving no-clean and rosin flux residues.

The resistivity ranges and ionic efficiency were measured using the following protocol (Figure 5):

1. Measure and record the resistivity of the solution (neat) as received
2. Pour ~500ml in a 1000 ml graduated cylinder
3. Add 1 ml of Alpha 615 RMA liquid flux to the solvent and re-measure the resistance
4. Add additional 1ml aliquots of flux stopping to measure and record solvent resistance after each addition

5. After the final addition of flux, add ~100grams of mixed Mega Resins to the container and re-measure and record the resistance
6. Record the time to recover the starting resistance of 80 meg-ohms
7. Note any resin compatibility issues

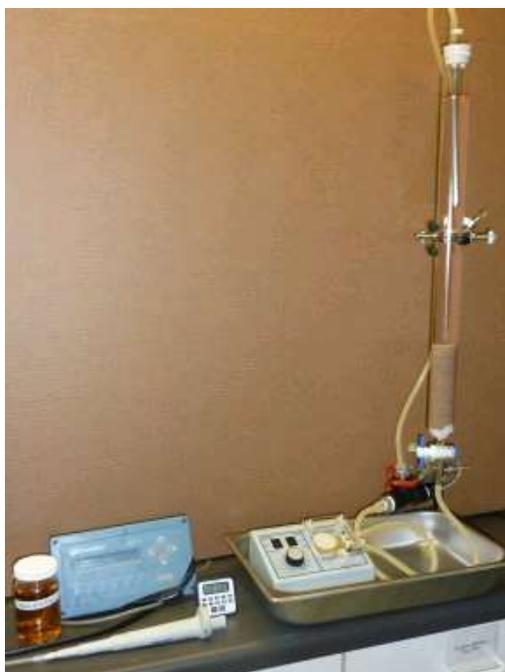


Figure 5: Ion Exchange Resin Test Apparatus

Test 1: The test control evaluated the current water resins response with IPA. To reach the starting resistivity purity (80 meg-ohm) for the static cleaning test, the contaminated IPA solution with 5 ml of Alpha 615 added to the 500 mls of IPA required 8 hours to reach a purity level of 80 meg-ohm

IPA (Meg-ohms) with Current Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
5.17	2.28	1.50	1.15	0.91	0.78	8 hrs

Table 3: IPA Control with Current Water Resins

Test 2: The second test evaluated the new stoichiometric ion exchange resins response with IPA. The new ion exchange resins provide a very fast response. After 5 minutes, IPA reached a purity level of 80 meg-ohms.

IPA (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
2.63	1.8	1.32	1.11	0.97	0.85	5 min

Table 4: IPA Control with New Resins

The test procedure used for the new extraction solvents was modified to use the dynamic test method. The objective was to recirculate the solvent through the ion exchange resins while adding the Alpha 615 flux. After the 5 ml was added, the time to max out was recorded.

Test 3: Solvent 1 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	43.8	42.9	42.1	41.2	40.5	5 min

Table 5: Solvent 1 with New Resins

Test 4: Solvent 2 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	75.1	66.41	57.75	51.2	46.3	5 min

Table 6: Solvent 2 with New Resins

Test 5: Solvent 3 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	7.20	6.00	4.74	3.83	3.34	20 min

Table 7: Solvent 3 with New Resins

Test 6: Solvent 4 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	80	80	80	63.99	57.94	5 min

Table 8: Solvent 4 with New Resins

Test 7: Solvent 5 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	0.24	0.23	0.22	0.21	0.20	52 min

Table 9: Solvent 5 with New Resins

Test 8: Solvent 6 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml	2ml	3ml	4ml	5ml	Max

	Alpha 615	of Alpha 615	of Alpha 615	of Alpha 615	of Alpha 615	to 80 meg-ohms
80	80	80	80	80	80	0 min

Table 11: Solvent 6 with New Resins

Test 9: Solvent 7 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	11.01	8.43	7.05	6.18	5.54	10 min

Table 12: Solvent 7 with New Resins

Test 10: Solvent 8 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	5.78	4.56	3.97	3.62	3.18	15 min

Table 13: Solvent 8 with New Resins

Test 11: Solvent 9 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	57.9	48.76	48.56	39.81	37.72	5 min

Table 14: Solvent 9 with New Resins

Test 12: Solvent 10 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	38.14	29.57	25.03	22.31	19.92	5 min

Table 15: Solvent 10 with New Resins

Test 13: Solvent 11 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
NR*	NR	NR	NR	NR	NR	NR

*Conductivity meter did not recognize the solvent

Table 16: Solvent 11 with New Resins

Test 14: Solvent 12 (Meg-ohms) with New Ion Exchange Resins						
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Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	2.21	1.68	1.37	1.17	1.03	20 min

Table 14: Solvent 12 with New Resins

Test 15: Solvent 13 (Meg-ohms) with New Ion Exchange Resins						
Initial	1ml Alpha 615	2ml of Alpha 615	3ml of Alpha 615	4ml of Alpha 615	5ml of Alpha 615	Max to 80 meg-ohms
80	8.76	6.06	4.97	4.23	4.07	15 min

Table 15: Solvent 13 with New Resins

INFERENCES FROM THE DATA FINDINGS

The data findings indicate that the new stoichiometric mixtures of ion exchange resins are effective at absorbing polar residue sets in non-aqueous solutions. The recovery response time was much improved over water resins.

The range of solvents tested provides insight into the molecular structures that provide the best response. This information is critical in engineering the extraction solvent(s) that provide the best properties for dissolving a wide range of flux residue types. The data also provides insight into the response time for detecting and measuring the contamination level.

This testing and previous testing continue to indicate that water is not helpful in removing polar soils bound in complex hydrophobic organic matrices such as those used today on modern electronic packages. The flux matrix itself must be dissolved to release the ionizable compound contained within. Waterless extraction solvents with sufficient molecular di-pole moment can provide the solvency and resistance response required to make a complete assessment of ionic residues.

CONCLUSION

The R.O.S.E. method has a number of limitations due to the advances in flux technology and miniaturization. Many modern flux technologies do not clean well with IPA/H₂O extraction solvent. The second challenge comes from highly dense circuits and how to measure contamination under low gap components. This requires a holistic though process that takes into consideration new extraction solvents, ion exchange resins and test instrumentation.

This paper is the third publication that researches the process conditions needed to develop an improved R.O.S.E. method. The first two papers focused on solubility theory and improved extraction solvents. The objective of this paper was to research the response time of patented stoichiometric mixture of ion exchange resins. The research findings provide insight into the molecular structures and

solvent family types that respond well to the ion exchange resins. The next step is to engineer extraction solvent blends and develop the response slope.

FOLLOW ON RESEARCH

Basic cleanliness tester design needs to change to facilitate the use of new HSP extraction solvents. Most IPA/H₂O testers are constructed of plastics and plastic parts. These would most likely be incompatible with HSP engineered extraction solvents such as the one tested herein. These material changes would facilitate the ability to heat the solvent, improving the solubility rate. Fluid delivery improvements can increase the physical energy delivered, allowing better solvent flow in tight spaces.

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