Validity of the IPC R.O.S.E. Method 2.3.25 Researched

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Abstract
Miniaturization and higher functionality in electronics packaging require the use of advanced packages and small components. This trend has translated into the use of new package types such as Quad Flat Pack – No Lead (QFP) (also referred to as Leadless Plastic Packages), increased use of chip scale packages as well as increased component density and tighter PCB layouts. Advanced package innovations and new flux types may compromise the validity of the R.O.S.E. cleanliness testing method. This paper researches the effectiveness of the R.O.S.E. cleanliness testing process for dissolving and measuring ionic contaminants from boards soldered with no-clean and lead-free flux technologies. The paper researches quality assurance and process control improvements needed to clean, extract, and measure the resistivity of solvent extract on today's circuit assemblies.

Introduction
Printed wiring board cleanliness improves yields, assembly performance and long term reliability. Ionic and non-ionic contamination can impact the operation and reliability of the device on which they are present.[1] In a humid environment, ionic contaminants can cause problems such as shorting between conductors by electrolytic dendrite growth, corrosion that erodes the conductors themselves or loss of insulation resistance.[2] Non-ionic residues are not conductive, but the insulating properties of residue surrounding conductors can lead to unwanted impedance.

In the 1960's, the Department of Defense (DOD) became increasingly concerned about the failure of circuit assembly cards due to ionic contamination. The DOD collaborated with industry experts to develop a quality assurance and process control method for quantifying the level of ionic contaminants remaining on circuit cards following the manufacturing process. The method detected and measured ionizable surface contaminants by resistivity of solvent extract, more commonly referred to as R.O.S.E. testing. The R.O.S.E. process control tool can be used to inspect printed wiring boards or printed wiring assemblies and determine if they conform to the requirements of the user's performance specification.[3]

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 component clearances. 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 address these limitations, extract solvents, contamination detection sensors, and dynamic cleanliness test instruments need to be improved to meet the quality assurance and process control needs of electronic assembly manufacturing operations.

Miniaturization and Flux Changes
Miniaturization increase reliability demands as electronic assemblers move upstream from conventional designs and toward threshold and leading edge technologies.[4] 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.[5] The current or traditional normal view of quality assurance correlated with visual residue and the resistivity of solvent extract measurements. With the reduction in component size and low standoff clearances, the ability to dissolve, extract, and measure ionic residues using the R.O.S.E. method is much more suspect. 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.[6] 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/H_2O 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/H_2O 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.

Problem Statement
The R.O.S.E. test method has been the industry standard production line test for measuring the cleanliness of electronic assemblies for more than forty years. Originally established as a test for military electronic hardware soldered with RMA fluxes, the test is performed by flowing a 75%/25% mixture of isopropyl alcohol (IPA) and de-ionized water across the surface of a previously cleaned electronic assembly and measuring the drop in resistivity of the IPA/H_2O reagent. The R.O.S.E. test was incorporated into military specifications (Mil-P-22809) in the 1970's as a final cleanliness requirement for building circuit assemblies for military hardware.

Cleanliness testing equipment provides an automated method for running the R.O.S.E. test. The automated R.O.S.E. test remains the current standard for conducting daily production tests for electronic assemblies. The current standard allows the use of many new flux types, but many of these new flux types are not soluble in the standard 75%/25% IPA/H_2O extraction solvent. The current cleanliness standard makes no allowance substitution of extraction solvent if the flux is not soluble in the IPA water blend.

Measurement using the R.O.S.E. test relies on dissolving the ionic residues that are bound in an organic flux matrix, to measure the effect on resistivity. Thus, if the ionic residues are not dissolved in the IPA/H_2O mixture, then undissolved ionic residues are not detected by the R.O.S.E. test, which results in an inaccurate determination of electronic circuit board cleanliness. Acerbating the problem, the continual miniaturization of electronic assemblies created further problems dissolving ionic residues under low clearance components. This leaves companies producing high reliability production hardware with significant exposure due lack of acceptable cleaning and testing systems.

Improving the ROSE Cleanliness Testing Process
The process cleaning rate theorem holds that the static rate (solubility of the flux residue in the extract solvent reagent) plus the dynamic rate (thermal and mechanical energy) equals the process cleaning rate. The static cleaning rate represents the thermodynamic properties of the extraction solvent reagent for the flux residue. Flux soils with similar solubility parameters will be miscible and dissolve in extraction solvent reagents whose solubility parameters are not too different from their own. The basic principle is that "like dissolves like." Extraction solvent reagents that do not match up to the flux residue in question will not dissolve or detect the ionic nature of the flux residue. The main problem with the ROSE test today is that it is limited to the IPA/H_2O mixtures, and under current processing conditions, does not adequately dissolve most of today's fluxes trapped under SMT components.

The solution is to rethink the R.O.S.E. testing process. Three driving forces must be considered:
1. Innovative New Extraction Solvent Blends that dissolves today's flux residues
2. Compatible Resins to Absorb ionizable residues from the extract solvent
3. Cleanliness Test Equipment designs that provide improved solvent compatibility, higher extraction energies, and process improvements such as density calibration and board drying.

Innovative Extraction Solvents
Flux residue solubility parameters are directly related to the energy required to dissolve the flux residue into the cleaning agent. By first characterizing the flux soil types, an interaction zone can be developed for cleaning a broad range of soils. Designing an extract reagent requires research into multiple flux types with the design objective to engineer a new reagent that is positioned within the interaction zone in order to rapidly remove the flux residue in question. Matching the solubility parameters of the reagent with the flux residue interaction zone assures excellent cleaning and detection performance.

It is important to know whether the flux residue is a single or multiple phase mixture. Most flux compositions contain multiple phases, which require reagent engineered with building blocks that dissolve the various components present in the

flux residue. Since flux compositions contain multiple components, a composite HSP for each flux residue soil can be established. The insight gained from this level of research provides insight into the extract solvent material properties needed to rapidly dissolve and remove multiple flux soil variations. The data set that follows compares the standard 75%/25% IPA/H_2O extraction solvent to a new solvent blend formulation to match the HSP of some of the most common fluxes used today.

Methodology
The solubility of twelve solder pastes into the IPA/H_2O extract solvent and into an engineered extract solvent designed to improve the static cleaning rate on many of today's flux residues was researched. Three of the solder pastes were lead-free water soluble, three were tin-lead rosin, three were tin-lead no-clean, and three were lead-free no-clean. Circuit cards were printed with each solder paste, reflowed, and exposed to the extract solvents in the test matrix. The extract solvents were tested at 20°C, 40°C, and 60°C for ten minutes using a shaker table to rotate the reagent during the test. The rate of soil removal from the part was examined and graded based on the grading scale in Table 1. Figure 1 illustrates the scoring classifications used to grade the rate of removal.

Table 1: Grading Scale

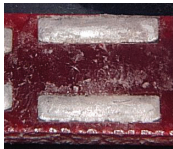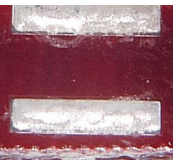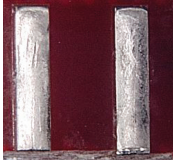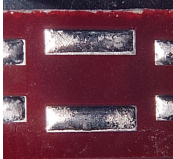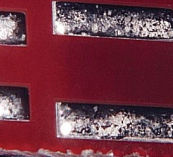| Score	Description
1	Easily dispersed, very stable dispersion
2	Takes a bit of effort to disperse, reasonably stable
3	Disperses but fairly soon starts to settle
4	Pretty reluctant to disperse
5	Some hint of dispersal
6	Just sits and laughs at the solvent
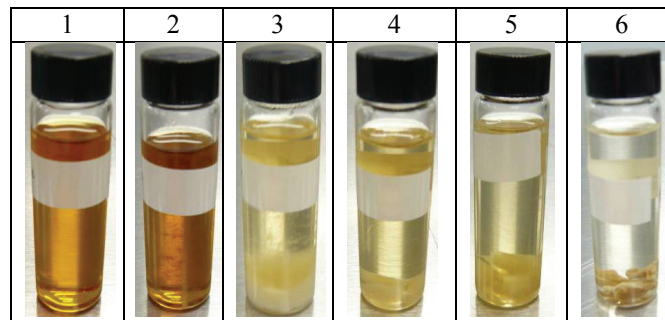
Figure 1: Grading Scale Sample Illustrations
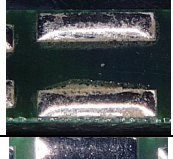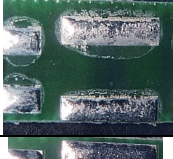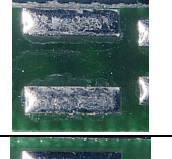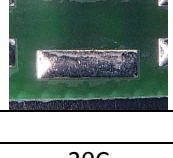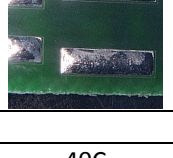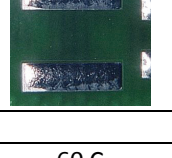
Data Findings
The DOE in Table reports the grade and visual findings.
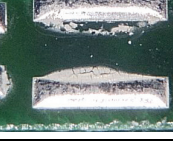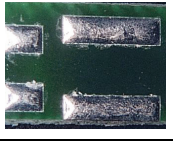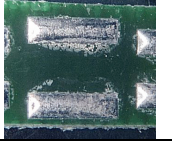
Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Water Soluble 1 Lead-Free	IPA/H_2O	10 min.	100 RPM	1 @ 20C 1 @ 40C 1 @ 60C			
Water Soluble 1 Lead-Free	New Reagent Design	10 min.	100 RPM	1 @ 20C 1 @ 40C 1 @ 60C			

Water Soluble 2 Lead-Free	IPA/H$_2$O	10 min.	100 RPM	4 @ 20C 4 @ 40C 4 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Water Soluble 2 Lead-Free	New Reagent Design	10 min.	100 RPM	3 @ 20C 2 @ 40C 2 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Water Soluble 3 Lead-Free	IPA/H$_2$O	10 min.	100 RPM	1 @ 20C 1 @ 40C 1 @ 60C			
Water Soluble 3 Lead-Free	New Reagent Design	10 min.	100 RPM	1 @ 20C 1 @ 40C 1 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Rosin 1 Tin-Lead	IPA/H$_2$O	10 min.	100 RPM	4 @ 20C 4 @ 40C 4 @ 60C			
Rosin 1 Tin-Lead	New Reagent Design	10 min.	100 RPM	2 @ 20C 1 @ 40C 1 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Rosin 2 Tin-Lead	IPA/H$_2$O	10 min.	100 RPM	5 @ 20C 3 @ 40C 4 @ 60C			

Rosin 2 Tin-Lead	New Reagent Design	10 min.	100 RPM	1 @ 20C 1 @ 40C 1 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
Rosin 3 Tin-Lead	IPA/H$_2$O	10 min.	100 RPM	4 @ 20C 5 @ 40C 2@ 60C			
Rosin 3 Tin-Lead	New Reagent Design	10 min.	100 RPM	2 @ 20C 1 @ 40C 1 @ 60C			

Flux Classification	Reagent	Time	Shaker Table	Grade Table 1	20C Image	40C Image	60 C Image
No-Clean 1 Tin-Lead	IPA/H$_2$O	10 min.	100 RPM	4 @ 20C 4 @ 40C 5 @ 60C			
No-Clean 1 Tin-Lead	New Reagent Design	10 min.	100 RPM	2 @ 20C 3 @ 40C 2 @ 60C			

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No-Clean 2 Lead-Free	New Reagent Design	10 min.	100 RPM	2 @ 20C 3 @ 40C 1 @ 60C			
No-Clean 3 Lead-Free	IPA/H$_2$O	10 min.	100 RPM	5 @ 20C 5 @ 40C 4 @ 60C			
No-Clean 3 Lead-Free	New Reagent Design	10 min.	100 RPM	3 @ 20C 2 @ 40C 1 @ 60C			
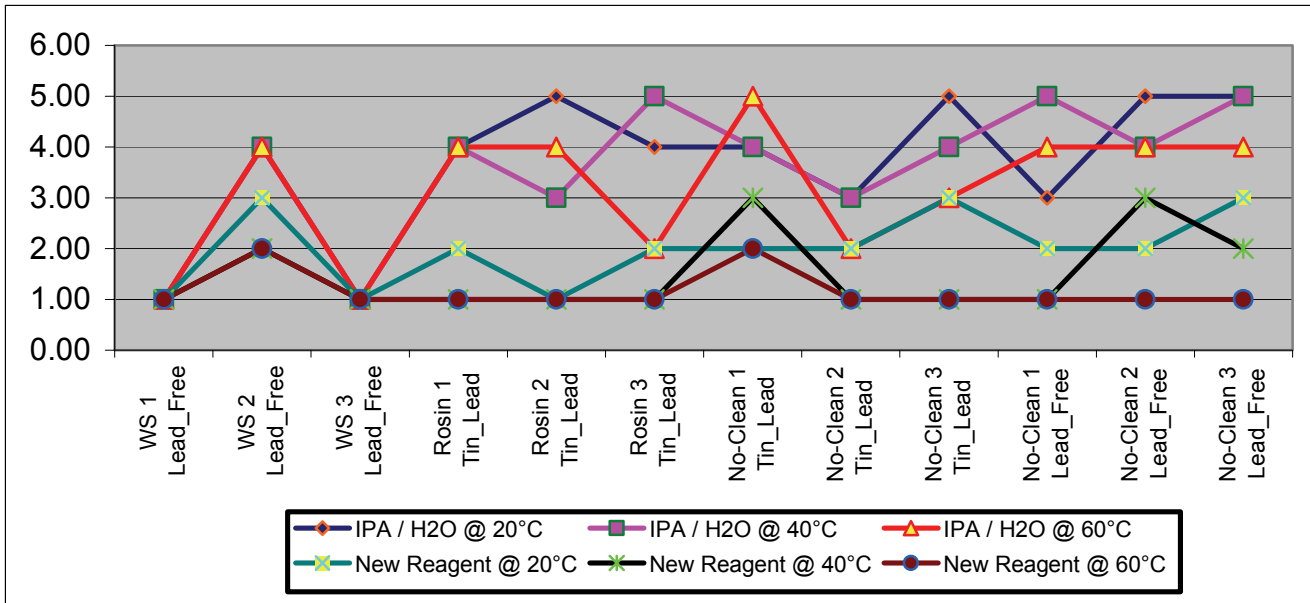
Figure 2: Summary of Data Findings
See Table 1 for Grading Scale

Inferences from the Data

The resistivity of solvent extract seeks to answer the question "How Clean is Clean?" IPC-TM-650 2.3.25 defines an acceptable board as having less than $1.5\mu g/cm^2$ NaCl equivalence. To obtain an accurate measure of ionic contamination, the IPA/H_2O extraction solvent must fully dissolve remaining flux residues after the cleaning process. To assure complete flux removal, it is increasingly important to match the solubility parameters of IPA/H_2O with the flux residue. In the 1970s when the R.O.S.E. method was developed, rosin flux residue was the standard and a good solubility match with IPA/H_2O. Today's electronic assemblies and solder flux compositions are increasingly complex and changing. The data indicates that the existing IPA/H_2O extract solvent dissolves very few of these fluxes.

If IPA/H_2O is not a good solubility match for many flux residues, why not increased the extract solvent temperature or design a different extract solvent that is a good match? One process factor studied was the effect of increasing the temperature of IPA/H_2O. Increased temperature improves the static cleaning rate but stills falls short for most flux types. Increased temperature also exposes flammability concerns. The data indicates that new extraction solvent designs improve the solubility rate but also require increase temperatures. The research suggests that a simple change to a new extraction solvent fails to solve the problem. The inferences from the data suggest that the solution requires a holistic approach of designing not only new extraction solvents, but also the design of ion exchange resins that support higher temperatures as well as cleanliness tester equipment that provides increased temperature, sensing accuracy, and mechanical energy.

Conclusions

It is well known that electronic assembly cleanliness is crucial to the assembly's performance and reliability. Monitoring and quantifying the degree of cleanliness is necessary in order to ensure product conformance. The RO.S.E. Cleanliness testing equipment provided a quick and reliable method for monitoring the cleaning process, contamination detection, and process control. Forty years latter the same technology has not changed and is no longer viable as a process control and quality assurance control tool.

To address this problem, the R.O.S.E. test method needs to be improved. It is estimated that there are in excess of 300 flux formulations in use across industry today. The data suggests that the solution to the problem requires new extraction solvents designed with solubility properties closely match to the flux technology. Since these extraction solvents are very different from IPA/H_2O, cleanliness testing equipment and the ion exchange resins must be considered as well.

Follow on Research

Improving the R.O.S.E. method is not a simple replacement of the extraction solvent. The update procedure requires the process to be integrated with the ion exchange resins and cleanliness tester equipment. Follow on research is needed in both these areas.

Compatible Resins to Absorb Ionic and Non-Ionic Residues from the Extract Solvent
The current ion exchange resins are designed for use with IPA/H_2O 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 the new extract reagent and higher temperature processing. To address this limitation, new ion exchange resins, housed and plumbed appropriately, are needed to support the new extraction solvents.

Ion exchange resins are needed to remove ionizable contaminates dissolved in the extraction solvent during the testing process. These new resins are needed to both remove contaminants and to purify the extraction solvent. The test equipment will require 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 metering 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.

Cleanliness Testing Equipment
Based on the test results provided in this paper, new extraction solvents will certainly require a new generation of cleanliness testers. Cleanliness testing equipment uses a purified solvent reagent designed to remove soluble flux residues. The reagent is circulated into and out of the test tank chamber containing the circuit assembly under test. The solvent reagent exiting the tank is passed through a conductivity cell which measures the conductivity continuously. These conductivity values are integrated over the time of the extraction. The mixture is then pumped through the resin columns to purify the reagent solvent before the next test. As ionic materials are extracted from the samples and then pumped out of the test chamber, the conductivity of the reagent will be reduced until all of the extractable ionic material has been removed.

The cleanliness tester design should include a chamber for cleaning and testing, a reagent solvent input into the test chamber, a resistivity sensor positioned for sensing resistivity of the reagent within the test chamber, and a reagent output for removing the solvent from the test chamber. The cleanliness tester should also include a means for physically agitating and/or heating the reagent to improve dissolution. The ion exchange resins must be compatible with both reagent and processing temperatures. Resistivity of the reagent after contact with the electronic assemblies may be measured and compared with the starting reagent resistivity. The level of ionic residues needs to be calculated based on the drop in resistivity.

Basic cleanliness tester design needs to change to facilitate the use of new HSP extraction solvents. Most IPA/H_2O 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.

Authors

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