

ADDRESSING THE PROBLEMS WITH IONIC CLEANLINESS TESTING ON MODERN CIRCUITS

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ABSTRACT

The Ionic cleanliness of a circuit card can be assessed with several methods. Visual cleanliness is an indicator that a board is clean but this can be deceiving. Several methods are available to assess the contamination level quantitatively. The most common ionic circuit test is the ROSE test. This test has remained unchanged for the last 50 years while soldering materials and component design changed radically. Several ways to update and improve the ROSE test are proposed and evaluated.

Key words: ROSE, Ionic, testing, inline, batch, cleaning

ROSE TESTING – A HISTORICAL PERSPECTIVE

Shortly after the electronic circuit card replaced point to point wiring in the 1950's, the reliability of manufactured circuit assemblies was directly linked to the amount of "freely" ionizable material remaining on the board following assembly. These residues in the presence of moisture and a voltage differential will undergo both chemical and dendritic electrochemical reactions resulting in rapid and catastrophic circuit failure.

In response to several high profile space and military systems failures in the 1960's, the US Military developed and incorporated the "Resistance of Solvent Extract" test or as it became commonly known as the ROSE test.

The ROSE test initially was performed manually using a procedure in which a known volume of clean solution of 75% IPA and 25% purified water was sprayed onto the assembly being tested and collected in a clean beaker. The resistance of the soiled solution was measured and compared to standard NaCl solutions. Based upon final resistance, the amount of NaCl equivalent on the test assembly could be determined with some accuracy.

The amount determined to be present was divided by the area of the circuit being tested and an average ionic contamination was determined. A limit of no more than 10 micrograms per square inch, or 1.56 micrograms per square centimeter, was set based upon test data and reliability data from failed units.

This remains the default standard today. The ROSE method quickly gained acceptance and was incorporated in the US military specification, Mil-P-28809 in 1971¹. Automated testing machines became available and replaced the manual method. From that point forward, virtually all US contracts to build electronic hardware required that a sample board be pulled from normal production and a daily ROSE test performed.

Everything got more complicated in the 1980's with the introduction of SMT assembly design because the flux in solder paste got a lot more complicated. Viscosity modifiers and thixotropic agents were added to improve printability. Tack extending agents and anti-slump compounds are present to keep things in place until the reflow soldering process heats and melts the solder. All these additions make cleaning and cleanliness testing more difficult.

In 1990 the Montréal protocol was enacted which stopped the production and use of the primary class of cleaning agents being used. In response the Industry developed a new class of fluxes referred to as "No-clean fluxes". This gave license to the flux and solder paste formulators to put things in that cannot be cleaned or at the very least were difficult to clean. Cleaning and testing got even more difficult with the introduction of higher temperature "lead free" solders which expose flux residue to a longer and hotter assembly profile.

Through all of this, the ROSE test protocol remained virtually unchanged for 50 years. Confidence in the cleanliness test has eroded significantly because of concerns about dissolving and measuring the "right stuff". ROSE testing is still required on most high reliability electronic build contracts because there is no better method which is practical in production. Figure 1 shows the evolution of cleaning and testing.

A lot has been said about the validity of the current IPC ROSE test². Some say it is no longer valid because most new flux residues are not soluble in the IPA/water extraction solvent. Others have concerns about contaminated board escapes allowed by sample testing versus 100% testing of all product. There is no doubt, it is time to bring the test method up to date.

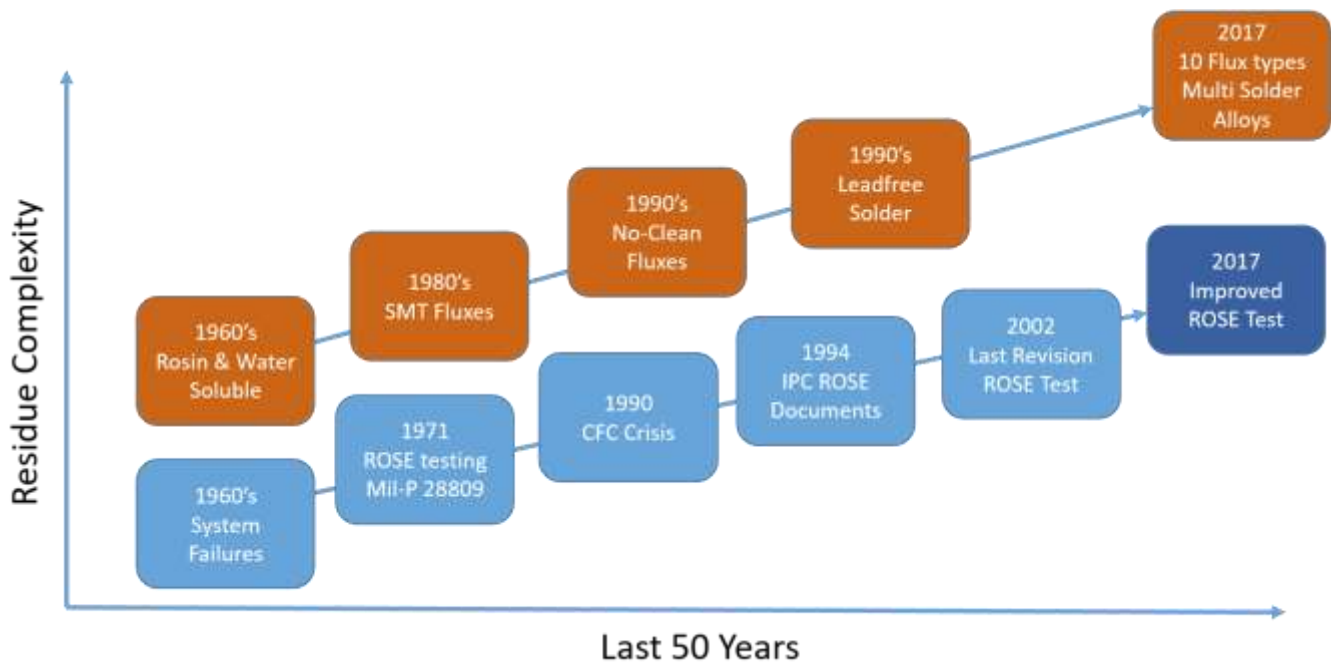
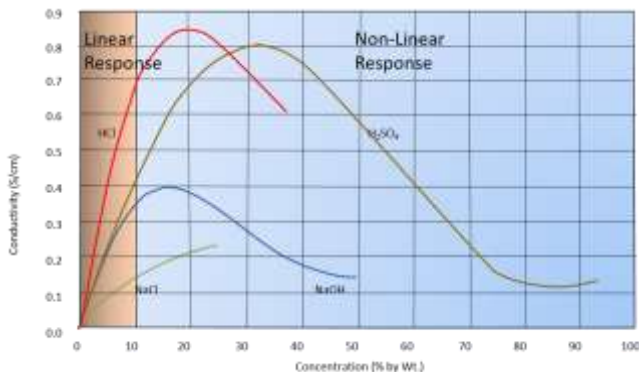


Figure 1 The History of flux cleaning and ROSE Testing for the last 50 years

UNDERSTANDING THE ROSE TEST METHOD

The mathematics of the ROSE Test was based on the relationship between concentration of a given ion in a polar solvent and the effect on the solutions ability to resist or conduct electron flow in that solvent. This relationship is linear for conductance for most ions and extraction solvents with concentrations below 10% as shown in graph 1 below³.



Graph 1 Conductance response vs concentration for common ions in water

In general, salts with a high ionization potential like those shown in Graph 1 give a rapid rise in conductance as concentration increases. Others with weaker ionization potential like organic acids (flux activators) give weaker responses. Because the ROSE test does not discriminate the type of ion present, all results are expressed as sodium chloride equivalent (NaCleq./cm²). This treats all ionic effects on conductance as if all ions were NaCl.

Calculations for the ionic concentration set forth in this paper are based upon conductance changes versus resistance changes because the math is simpler. Resistance is inversely related to conductance as shown in equation 1.

Equation 1

$$R=1/C \text{ or } C=1/R$$

Where: Electrical Resistance = R, Electrical Conductance = C

The current IPC ROSE test method requires a mixture of isopropyl alcohol (IPA) and de-ionized (DI) water. This mixture was selected as the extraction solvent because liquid rosin fluxes were not soluble in water alone. To dissolve the rosin based flux, a mixture of isopropyl alcohol and de-ionized water was selected as the extraction solvent. This solvent and water mixture also gives a predictable linear response, but the rate of change is different depending upon the mix ratio as shown in graph #2 below.

Calculating the NaCl Equivalent

This relationship is mathematically shown in Equation 2 below.

Equation 2

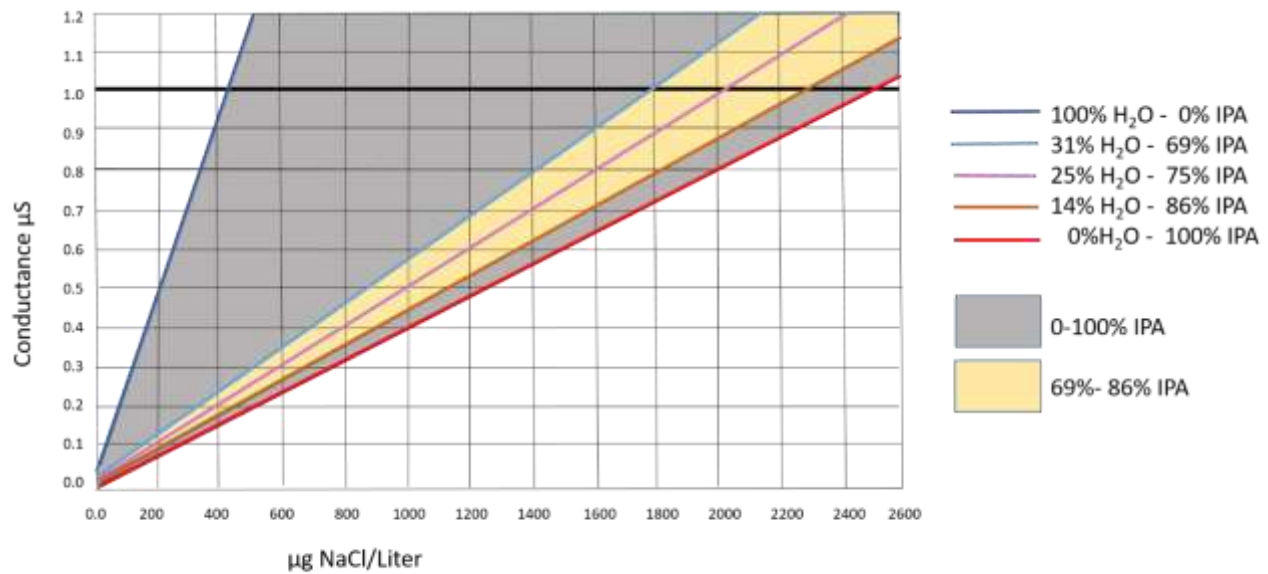
$$\text{Total Ionic Contamination} = \text{Test volume} \times \Delta \text{Conductance} \times \text{SSV}$$

Where

Test volume = volume of the extraction solvent used in liters

Δ Conductance = the change in conductance in the extraction solvent start to finish

SSV = Solvent sensitivity value $\mu\text{gNaCl} / 1\mu\text{S}\Delta$ Conductance



Graph 2 Electrical conductance vs salt concentration for IPA/ water differing based upon mix ratios

SUGGESTED CHANGES TO THE CURRENT ROSE TEST

There are two changes needed to advance the ROSE test to meet the current challenges.

First any extraction solvent should be allowed which meets the following criteria;

- It should dissolve the residues of concern in 5 minutes or less
- It should not affect other residues intended to remain on the circuit.
- It should give a predictable change in conductance as ionic concentration increases.
- It should be compatible with DI exchange for solvent recycling

Second, the ROSE test should be integrated and automated into the cleaning process 100% whenever possible.

EXTRACTION SOLVENT CHANGES

Allowing any solvent or solvent blends that are designed to dissolve the residues of concern improves the detection of those ions. Residues of concern on circuits are generally those substances that become more conductive because of “free ions” created when exposed to humidity and temperature over time. Free ions are salts that become hydrated and ionize. These residues are electrically conductive and mobile and can participate in un-biased chemical corrosion, galvanic corrosion or electro-chemical migration when they bridge conductors with a voltage bias. Contrast this with ions locked in an insoluble matrix like some no-clean flux residues. These salts are not ionized if the matrix is impervious to the ionizing solvent over the lifetime of the product. Extraction solvents should be selected that do not disturb the matrix but do dissolve free ions. Following this logic, Table 1 shows the recommended new extraction solvent for current electronic grade flux types.

Table 1 Proposed Changes to ROSE Extraction Solvents *likely to cause bulk residue to turn white.

Flux Type	New ROSE Solvent (Recommended)	Old ROSE Solvent (Mil-P-28809)
Rosin (no-clean) low activity (ROL0)	DI Water	75% IPA/25% H ₂ O± 5%*
Rosin (no-clean) medium activity (ROL1)	DI Water	75% IPA/25% H ₂ O± 5%*
Rosin (to be cleaned) low activity (ROLO)	100% IPA	75% IPA/25% H ₂ O± 5%*
Rosin (to be cleaned) medium activity (ROL1)	100% IPA	75% IPA/25% H ₂ O± 5%*
Resin (no-clean) low activity (RELO)	DI Water	75% IPA/25% H ₂ O± 5%*
Resin (no-clean) medium activity (REL1)	DI Water	75% IPA/25% H ₂ O± 5%*
Resin (to be cleaned) low activity (RELO)	Hansen Solvent	75% IPA/25% H ₂ O± 5%*
Resin (to be cleaned) medium activity (REL1)	Hansen Solvent	75% IPA/25% H ₂ O± 5%*
Water Soluble (to be cleaned) low activity (ORLO)	DI Water	75% IPA/25% H ₂ O± 5%
Water Soluble (to be cleaned) med activity (ORL1)	DI Water	75% IPA/25% H ₂ O± 5%

Solvents recommended in Table 1 are pure solvents. 100% water and 100% IPA are single solvent systems and are easier to maintain than solvent blends like the 75%/25% IPA/water approved solvent. Water is the best and safest for free ion extraction. Pure IPA is a better solvent for organic residues like rosin. Difficult to clean, no-clean, resin based fluxes often require a special solvent selected by similar solubility parameters. These parameters called “Hanson Solubility Parameters” are unique to each soil and solvent. Selecting a solvent that is well matched with the soils Hanson parameters is the best way to find a solvent for difficult to remove residues⁴.

PROPOSED ROSE PROTOCOL CHANGES

The key to improving the control of the cleaning process is to eliminate control by a sampling plan and go to a 100% ROSE test incorporated automatically into the cleaning process. Doing so eliminates the labor and delay associated with sampling. More importantly, it provides real-time control of the cleaning process and lessens the chance of improperly cleaned escapes and unnecessary rework.

New cleaning equipment is now available in the market that allows automatic 100% ROSE testing of products in the cleaning cycle with new ion extraction solvents like DI water or other organic solvents. Figure 2 shows the protocol changes made to allow automatic ROSE testing.

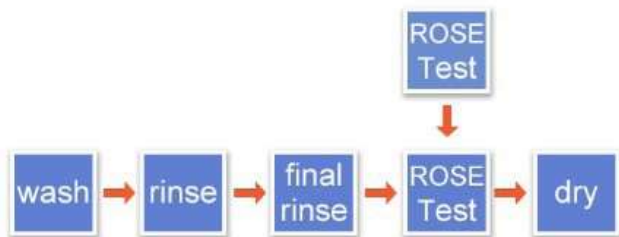


Figure 2 Cleaning process diagram showing insertion of automatic ROSE test

Testing each board or batch cleaned requires software and a few hardware changes to the cleaning equipment. The ROSE test is added to the cleaning cycle following the final rinse in inline cleaners. The cleaning cycle time including ROSE test is lengthened by a few minutes in batch cleaning and adds no time to the inline cycle.

BATCH CLEANING WITH AUTOMATIC ROSE TESTING

Adding ROSE testing to a batch cleaning process improves the control of the process. Most batch cleaners rely on the purity of the final rinse water, as determined by the conductance or resistance, to assure a proper cleaning cycle. This is commonly referred to as “rinse to resistivity”.

The problem with this approach is that a full load of boards will rinse longer than a partial load to achieve a fixed programmed conductance setting. This is because more total contamination must be removed. This either requires more time to rinse or more rinse cycles to get to the programmed conductance. Small loads are rinsed less than a fully loaded batch and this leads to cleaning variation, batch to batch.

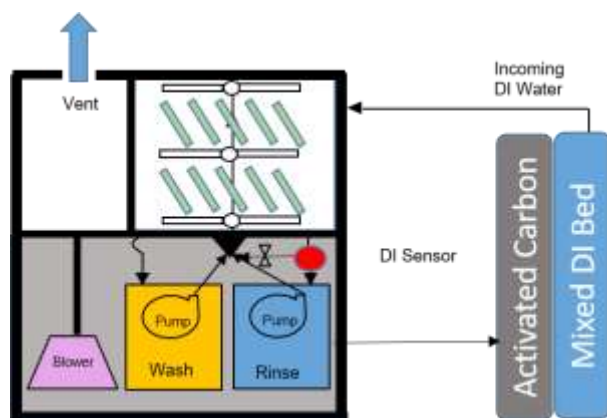


Figure 3 Closed Loop Batch Cleaner with full load. Requires more rinsing to achieve set point.

The amount of variation depends on the dissolution rate of the ions remaining on the circuit and resulting ionic concentration in the rinse water.

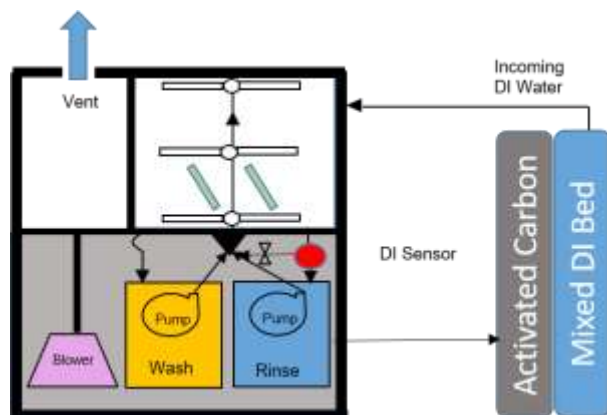


Figure 4 Closed Loop Batch Cleaner with partial load. Rinses less to achieve set point.

A single assembly will have a given rate and quantity based upon parameters such as the board size, ion type and location of the ions being rinsed.

Adding a second assembly as shown in figure 4 will double the magnitude of the ions being dissolved at any given time. A third would triple the rate and so on. The total ion dissolved at any time in the rinsing operation is therefore a multiple of the total circuit area of all circuits.

As previously mentioned, rinse water conductance is directly related to ionic concentration. A typically programmed rinse to conductance of $1\mu\text{S}/\text{cm}$ or $1\text{Mohm}\cdot\text{cm}$ resistance would correspond to a NaCl concentration of $400\mu\text{g}/\text{liter}$ ⁵ in the final rinse water.

Figure 5 shows the change in solution conductance typical for a batch cleaning cycle with ROSE testing. Note that the single board curve results in fewer rinse cycles because the clean to value is met sooner for a single assembly. In this example the single board met the conductance requirement in two cycles because it had less over-all ions to remove, not because it was cleaner. The fully loaded batch required two additional cycles to meet the conductance requirement.

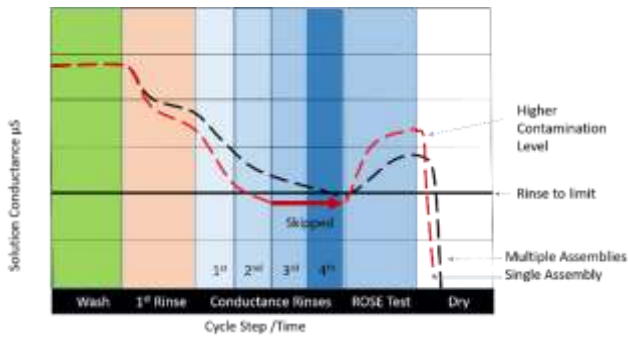


Figure 5 Graph of solution conductance at each batch process step for a single assembly vs full batch

The conductance rises in the ROSE portion of the cycle, as the final rinse water is sprayed onto the assemblies in a closed loop circulation for a programmed time without adding fresh DI water. This rise in conductance over time allows a quantitative calculation of total NaCl equivalent concentration to be calculated using equation 2 given previously. Dividing this quantity by the total surface area of the assemblies give the average cleanliness value for the batch cleaned.

Incorporating the ROSE test following the “rinse to resistivity” assures that the variation in cleanliness does not exceed the allowable cleanliness limit of 1.56 micrograms per square centimeter for small or large batches. This is not possible in a standard batch cleaning protocol and requires a separate ROSE test to do so. Tracking the ROSE cleanliness results batch to batch allows trend analysis with action limits to prevent problems before they become serious. The “rinse to resistivity” control limit does determine the amount of rinsing that occurs, but this does not assure all assemblies cleaned will pass the ROSE test limit. Currently a ROSE test is required to assure the proper level of cleanliness is achieved.

INLINE CLEANING WITH AUTOMATIC ROSE TESTING

Adding Rose testing to an inline cleaning process requires a slightly different approach versus batch cleaning. Here we are dealing with an individual assembly, one at a time as shown in Figure 6.

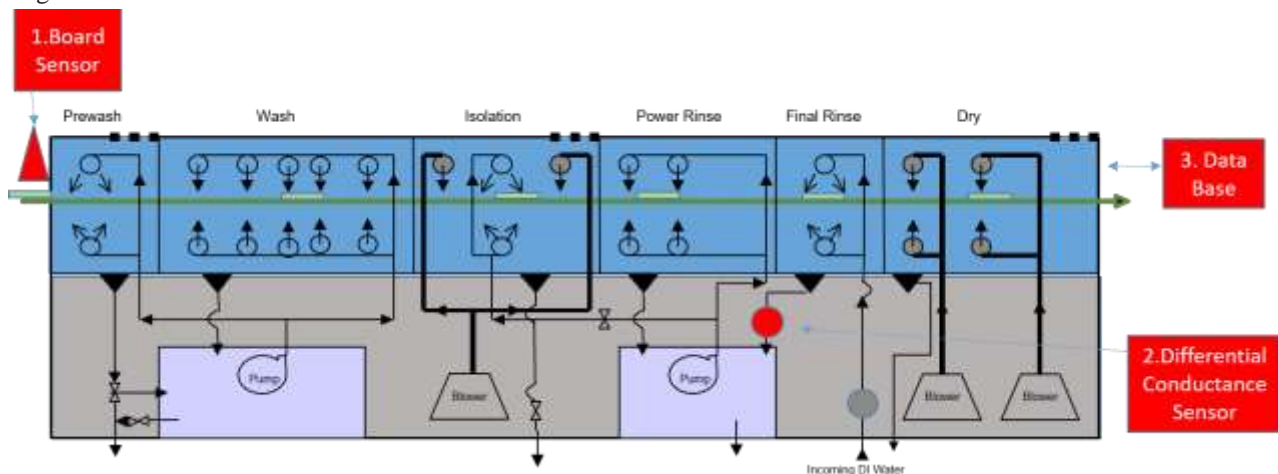


Figure 6 Inline cleaner diagram showing design changes needed to incorporate ROSE testing.

Individual boards need to be identified by barcode or RFID so individual data can be transferred and tracked. Once an assembly enters the cleaner it is identified and its location is known. When cleaning and rinsing is complete and it enters the final rinse, the increase in conductance of the water can be measured after contact with the assembly and the amount of contamination remaining can be calculated based upon the area under the curve as shown in Figure 7 below.

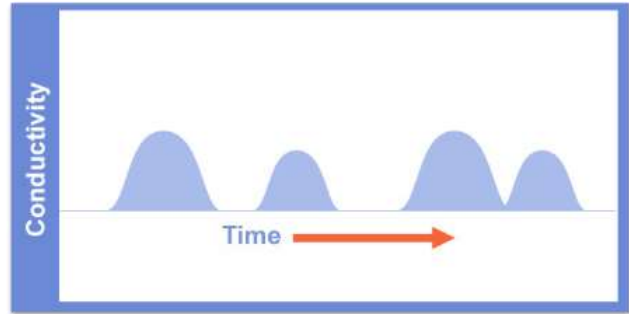


Figure 7 Inline cleaner ROSE testing data stream

CALIBRATION OF ROSE TEST IN BATCH AND INLINE CLEANERS

Calibration of the ROSE test is accomplished using the same method used in other ROSE testers. Calibration is achieved by injecting a known quantity of NaCl into the process chamber in the beginning of the ROSE portion of the cycle and confirming the calculated output matches within the tolerance of the equipment.

One additional step is required when calibrating ROSE testing integrated into the cleaning process. A background level should be measured and subtracted since the test is sensitive to all ions regardless of their origin. Ions originating from things other than the assemblies, chamber walls, racks, and belts that have retained contaminated rinse water should be characterized and subtracted from the absolute reading. This is true for both batch and inline cleaners with ROSE testing capabilities.

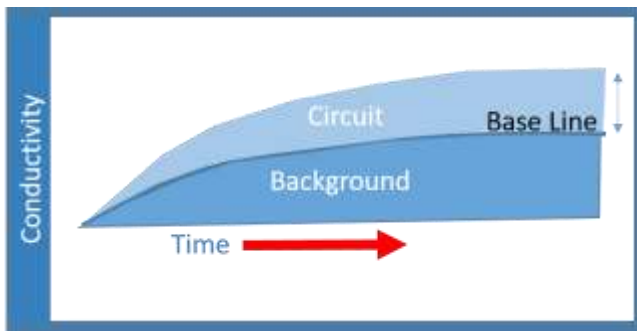


Figure 8 ROSE calibration graph for batch cleaner

The background is measured by running a ROSE Test with an empty chamber with a standard cleaning cycle. Once the value is determined for any given process, it is entered as a test variable. Recalibration of the background is required when changing the wash chemistry or access parameters.

COSTS AND BENEFITS

The new system saves time, increases the production rate and improves yields and product quality. Time is saved because the need to pull a sample and perform a separate test is eliminated. Production rates are increased because there is no product waiting on test results. Electrical test yields can be improved because there is better cleanliness. Product quality and reliability are improved when assemblies are cleaned properly before they ship to the customer. All of this can add up to a lot of savings that improves the price/profit plans of any company manufacturing electronic modules.

CONCLUSIONS

It is time to take a new look at our 50 year old ROSE test. Changes over this time to soldering processes and component and circuit design have introduced a completely new set of cleanliness challenges for companies to deal with. New and improved cleaning equipment with self-testing, feedback represent a significant development and step forward in the development of future electronic assembly. These new capabilities will allow real time control of product cleanliness as the items are being cleaned so that we can better control factory output.

Providing the use of new extraction solvents allows the use of safer selections that are more compatible. DI water is a great ion extraction solvent that can be easily used to provide ROSE results in both batch and inline cleaning. New organic extraction solvent should be considered for difficult to clean residues. It is time for the Industry to evaluate changes to the process that controls circuit board cleanliness in our factories. These changes are potentially very significant both in terms of manufacturing efficiency and over-all product reliability.

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