

# Implementation of Linear Sweep Voltammetry for Assessing the Corrosivity of Flux Residues

## Abstract

Residue on a PCB can originate from contamination, cleaning agents, resins, fluxes, and the reflow process. These residues may cause corrosion if not controlled or removed when necessary. The Resistivity of Solvent Extract (ROSE) test was widely used to determine cleanliness; however, a more current revision of IPC-J-STD-001, Rev G, Amendment 1, establishes new requirements for cleanliness testing. ROSE testing is no longer considered an acceptable method to determine product cleanliness, consequently, a new analytical technique must be used to provide objective evidence. In this study, Linear Sweep Voltammetry (LSV) was used to determine the corrosion currents of flux residues left on PCB boards after the reflow process. The corrosion currents correlate to the SIR pass/fail results. Water-soluble flux residues show high corrosion currents and fail the SIR tests. Conversely, no-clean flux residues show low corrosion currents and pass the SIR tests. The results indicate that LSV can be used to assess the corrosivity of a residue left on a PCB and predict the pass/fail in the SIR tests accurately and efficiently.

---

## 1. Background

Residues are generally found on PCB surfaces and/or around solder joints and can originate from materials used in the PCB assembly process. These materials include fluxes, resins, solvents, cleaning agents, solder mask, and component contaminants. There are a variety of residues that can be left on a circuit board and may prove to be harmful to the final product. For example, water-soluble fluxes are meant to be cleaned after the reflow process; otherwise the ionic composition of these fluxes would compromise the reliability of the PCB from corrosion. However, no-clean fluxes may be left on the boards assuming they are properly activated, leaving minimal and benign residue amounts [1].

Insufficient cleaning of solder flux residue can lead to product failures such as electromigration, which causes the formation of dendrites [2]. Corrosion occurs from the combination of several factors: ionic residues and moisture being present on the board, relative humidity, and an applied voltage. It is important to determine the corrosivity of an unknown residue left on the board in order to prevent failure in the final product.

Residues formed on PCBs are not limited to fluxes and solvent cleaning. These residues include various chemical reactions between the flux components, components materials, PCB contaminants, and cleaning agents. The chemical reactions may occur from an incorrect soldering temperature in the reflow oven, or from an improper cleaning agent [3].

The Resistivity of the Solvent Extract (ROSE) test is the oldest and most widely used cleaning test of PCBs in the electronics industry. It involves a simple conductivity measurement to quickly assess the overall cleanliness and determine the quality of the final product [4]. However, the test does not offer localized extraction of the residue. The IPC-J-STD-001, Rev G, Amendment 1 establishes new requirements for cleanliness testing. ROSE testing is no longer

considered an acceptable method on its own in determining the acceptability of product cleanliness. This method requires objective evidence from additional testing, such as Ion Chromatography (IC) or Surface Insulation Resistance (SIR), to produce reliable results [5].

IC and Fourier Transform Infrared Spectroscopy (FTIR) are analytical methods that allow quantification and qualification of the ionic materials that are present in a PCB [4]. The methods can be applied to either the full board, or to localized areas as needed. These techniques are used at the laboratory level and are not used in the factory due to their complexity and higher costs. SIR testing is the most common direct method for measuring the corrosive nature of a residue through the formation of dendrites and sub surface metal formation [6], [7].

Electrochemical corrosion measurements are used extensively in laboratory environments to measure the underlying electrochemical phenomenon of corrosion. These measurements can provide insight into the residue’s potential to corrode. Linear Sweep Voltammetry (LSV) is a potentiodynamic technique that uses a potential sweep applied to the working electrode, while the resulting current is measured. Tafel Analysis depicts the relationship between the current and applied potential. The extrapolation of the linear regions on the plot allow for the determination of the corrosion rate through the intercept of the anodic and cathodic branches, which correspond to the current at equilibrium [8]. LSV is a non-destructive technique that may be applied to study corrosivity of residues on PCBs.

In this study, LSV was used to examine the corrosivity of various flux residues extracted from SIR test boards. Simultaneously, SIR testing was performed using the same materials studied with LSV. The objective of this study was to investigate the relationship between the corrosion currents measured with the electrochemical measurements and the SIR results, which allows for the prediction of SIR failure.

## 2. Materials and Methods

### 2.1. Solder Paste for IPC-B-24 Tests Vehicles

Solder pastes of varying compositions and flux types were used to prepare the IPC-B-24 test vehicles (Table 1). A variety of lead-based and lead-free solder pastes with water-soluble and no-clean fluxes were studied to determine the difference between flux types and their influence in the corrosion process. Standard IPC-B-24 test vehicles were used to study the SIR responses of formed flux residues.

### 2.2. Electrochemical Measurements

Electrochemical measurements were performed using the Vision MARK-1 E-Chem insight and a three-electrode probe configuration (Figure 1). Solder paste ( $\text{Sn}_{63}\text{Pb}_{37}$ ), silver/silver chloride ( $\text{Ag}/\text{AgCl}$ ) and gold ( $\text{Au}$ ) were used as the working, reference, and counter electrode, respectively.

Table 1. Solder paste used in this study

Solder Paste	Alloy	Flux Type
A	$\text{Sn}_{63}\text{Pb}_{37}$	Water-soluble
B	$\text{Sn}_{63}\text{Pb}_{37}$	Water-soluble
C	$\text{Sn}_{63}\text{Pb}_{37}$	Water-soluble
D	$\text{Sn}_{63}\text{Pb}_{37}$	Water-soluble
E	$\text{Sn}_{63}\text{Pb}_{37}$	Water-soluble
F	$\text{Sn}_{63}\text{Pb}_{37}$	No-clean
G	$\text{Sn}_{63}\text{Pb}_{37}$	No-clean
H	$\text{Sn}_{63}\text{Pb}_{37}$	No-clean
I	$\text{Sn}_{63}\text{Pb}_{37}$	No-clean
J	$\text{Sn}_{63}\text{Pb}_{37}$	No-clean
K	SAC 305	Water-soluble
L	SAC 305	Water-soluble
M	SAC 305	Water-soluble
N	SAC 305	Water-soluble
O	SAC 305	No-clean
P	SAC 305	No-clean
Q	SAC 305	No-clean
R	SAC 305	No-clean



Figure 1. Three-electrode probe configuration used for LSV measurements

Localized extraction of the flux residues on the board were performed with 2 of the 4 combs in the IPC-B-24 test vehicle. The residue was extracted using a swab saturated in 75% isopropyl alcohol (IPA). The swab was swirled in 1 ml 75% IPA solution to remove the residue. The swab was then used to scrub the comb a second time before being stirred in the same solution.

LSV was performed using the three-electrode configuration and the residue solution as the electrolyte. The potential was swept between -1.0 and 0.1 V (vs. Ag/AgCl) at  $0.2 \text{ V s}^{-1}$ . Tafel Analysis was performed to determine the corrosion currents associated with the process.  $\log |i|$  vs.  $E$  was plotted; and the linear regions of the cathodic and anodic branches were determined 250 mV away from the corrosion potential. The corrosion currents were determined from the intercept between the cathodic and anodic extrapolated lines.

### 2.3. Surface Insulation Resistance (SIR)

SIR tests were performed using a climatic chamber to control temperature and humidity. The temperature was set to  $40 \pm 0.3 \text{ }^\circ\text{C}$ ; relative humidity (RH) was set to  $90 \pm 5\%$ . The chamber parameters were set for a minimum of 1 hour before beginning the measurements to ensure the desired conditions. Each time, IPC-B-24 boards were tested using the 2 combs not cleaned for the electrochemical measurements. The resistance of each comb was recorded every 20 minutes. The tests were run for 7 days or stopped if the resistance was lower than 100 M $\Omega$  and there was discoloration or corrosion visible on the combs.

## 3. Results

### 3.1. Electrochemical Measurements

Tafel Analysis is widely used to determine corrosion currents. For example, Figure 2 shows the Tafel plot for one of the residues tested in this study. The linear section was determined 250 mV from the corrosion potential in order to measure at least one decade of current. The current value at the corrosion potential intersection is the corrosion current,  $i_{corr}$ .

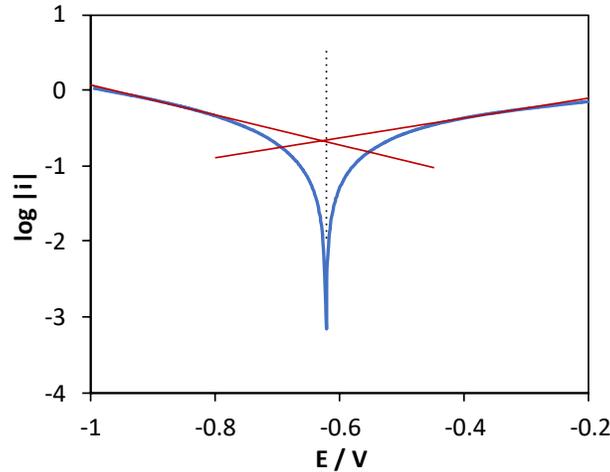


Figure 2. Tafel plot of a residue from Paste Q

The corrosion currents calculated from Tafel Analysis for water-soluble and no-clean fluxes were presented in Figure 3. The calculated currents vary according to the type of flux in each solder paste. The corrosion currents of water-soluble fluxes were larger than those in no-clean fluxes. These results were expected due to the higher conductivity of the water-soluble fluxes and their polar nature, which makes ion transportation easier. On the other hand, no-clean residues, which are not meant to be cleaned, showed lower corrosion currents. The current values are closer to those with 75% IPA. This indicates that the amount of residue removed from the boards is small, and/or that the residues have low ionic content.

The corrosion currents calculated for the same type of flux show differences between the pastes. The water-soluble pastes were more variable than the no-clean pastes. This could be related to the differing chemical nature, or properties, of the pastes. It could also be attributed to a variation in the reflow process. After reflow, flux components, such as activators and solvents, could react and leave undesired sub products.

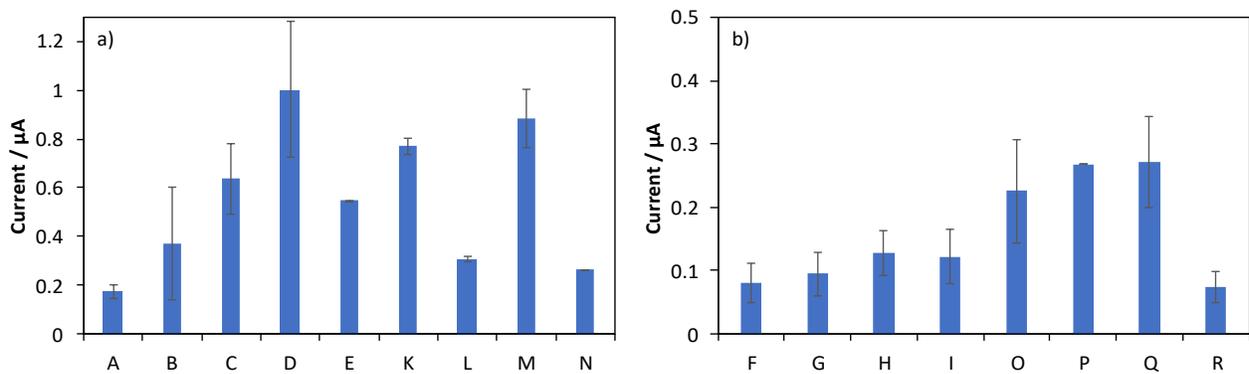


Figure 3. Corrosion currents for flux residues: a) Water-soluble b) No-clean

### 3.2. SIR Tests

The SIR test is designed to test the resistance drop due to contamination, voltage bias, humidity and temperature effects, flux residue activity, and conductor pitch [9]. Figure 4 presents a subset of data in this study which shows results for the no-clean solder pastes presented in

Table 1. Solder paste used in this study. Based on the results, the no-clean residues passed the SIR test because the resistivities were greater than 100 MΩ. On the other hand, the water-soluble residues tested in the SIR failed for all samples tested; the measured resistivities were less than 100 MΩ. In addition, all water-soluble boards showed discoloration and corrosion after being removed from the humidity chamber. The data displays differences based on the type of flux in the paste, the fact that all no-clean residues passed the SIR tests indicates that they were properly activated during reflow.

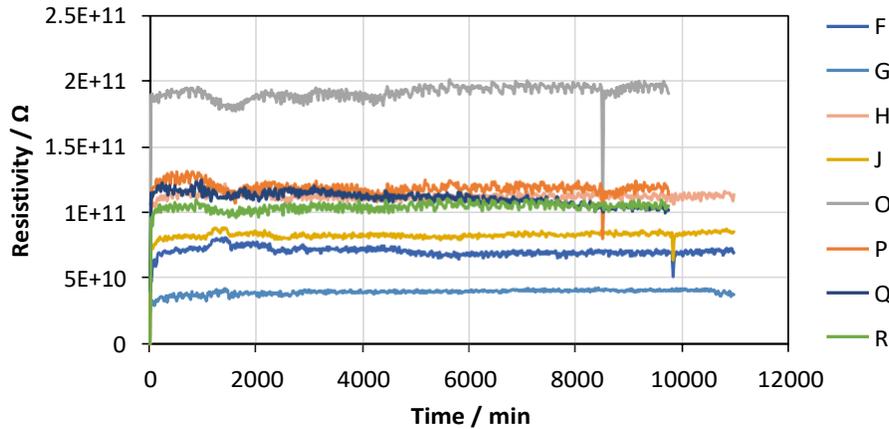


Figure 4. SIR data for no-clean flux residues

### 3.3. Relationship Between Corrosion Current and SIR Results

The objective of this study was to determine the correlation between the corrosion current from electrochemical measurements and SIR test results. Figure 5 displays the corrosion currents as a function of the average resistivity of residues measured in the SIR tests. The results show two distinct regions: the values with large corrosion currents show lower resistivities in the SIR tests; likewise, low values of corrosion currents show large resistivities in the SIR tests. This correlates with the type of flux in each residue. In Figure 5, red dots represent water-soluble residues and green dots represent no-clean residues.

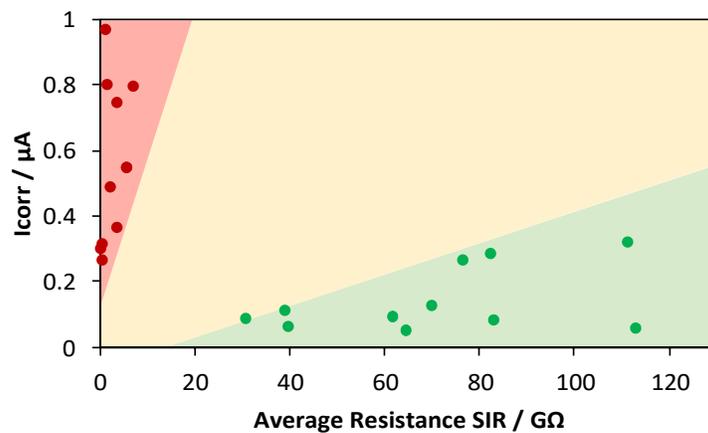


Figure 5. Corrosion currents against average resistivity of residues on PCB

While no technique can entirely replace SIR testing, corrosion currents measured using electrochemical techniques can be related probabilistically to the outcome of an SIR tests (Figure 6). This correlation could help to rapidly assess residues on PCBs in the factory environment.

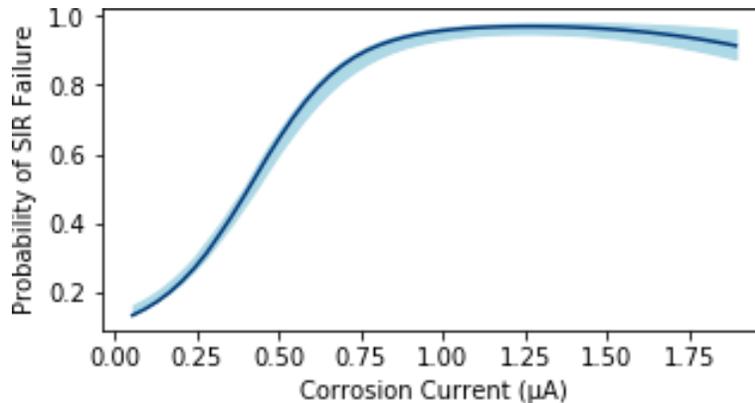


Figure 6. Probability of SIR test failure vs. output corrosion current

#### 4. Conclusions

The purpose of this study was to identify the correlation between electrochemical measurements (via LSV) and the SIR tests. Electrochemical measurements showed high corrosion currents for water-soluble flux residues, while lower corrosion currents values were obtained for no-clean flux residues. Differences between residues from the same type of flux were related to variation in the soldering process for each solder paste used in the study. SIR tests were performed using the same test vehicles used in the residue extraction. All no-clean combs passed the SIR tests, while all water-soluble combs failed. The results obtained in this study indicate that LSV is a fast and reliable technique that can be used to identify the potential corrosivity of a residue left on a PCB, and therefore predict SIR test results.

#### References

- [1] H. Conseil, M. S. Jellesen, and R. Ambat, "Contamination profile of Printed Circuit Board Assemblies in relation to soldering types and conformal coating," p. 10.
- [2] P. Jain, "Root Cause Analysis of Solder Flux Residue Incidence in the Manufacture of Electronic Power Modules," p. 76.
- [3] D. F. Bernier, "The Nature of White Residue on Printed Circuit Assemblies," p. 7.
- [4] "Cleanliness Testing for Printed Circuit Boards | NTS," *National Technical Systems*. [Online]. Available: <https://www.nts.com/services/testing/chemical/chemical-cleanliness/>. [Accessed: 15-Sep-2019].
- [5] "IPC-J-STD-001G-AM1: Requirements for Soldered Electrical and Electronic Assemblies," *IPC*. [Online]. Available: <https://shop.ipc.org/IPC-J-STD-001G-AM1-English-D>. [Accessed: 15-Sep-2019].
- [6] C. Nash and E. Bastow, "Understanding SIR," p. 9.
- [7] R. Michalkiewicz, J. Green, S. Opperhauser, and H. Valley, "Surface Insulation Resistance Testing of Soldering Pastes and Fluxes," p. 10.
- [8] A. J. Bard and L. R. Faulkner, *Electrochemical Methods: Fundamentals and Applications*, 2nd ed. Wiley, 2000.
- [9] M. Bixenman, M. McMeen, and B. Tolla, "Why Clean a No-Clean Flux," p. 6, 2016.