

The Effect of a Nitrogen Reflow Environment on the Electrical Reliability of Rosin-Based No-Clean Solder Paste Flux Residues

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Abstract

Rosin-based no-clean solder pastes are the most widely used solder pastes in the world and dominate consumer electronics assembly. The most common rosin-based no-clean solder paste reflow environment is air, which has some obvious advantages such as no-cost and tombstoning mitigation. However, there are a number of manufacturers that use a nitrogen reflow environment for reasons ranging from “shinier” solder joints to head-in-pillow (a.k.a. head-on-pillow) defect mitigation. Most solder paste manufacturers perform J-STD-004 qualification tests in air unless the solder paste specifically requires a nitrogen reflow environment. Of the J-STD-004 tests, the Surface Insulation Resistance (SIR) test is designed to predict the electrical reliability of a no-clean flux residue. In this test, solder paste is printed onto a test coupon and reflowed. This reflow is typically conducted in air, especially for a rosin-based no-clean solder paste. Knowing that the SIR values are typically the result of a reflow heating cycle conducted in air, some have asked if and to what degree would the SIR values be different if the reflow heating cycle had been conducted in nitrogen. This paper will attempt to address this concern.

For this work, three commercially available SAC305 Type 4 rosin-based no-clean solder pastes were used: a halogen-free (ROLO) paste with a traditional residue; a halogen-free (ROLO) paste with a residue optimized for pin-probing; and a halogen-containing (ROL1) paste with a traditional residue. These three different solder pastes were used to see if different chemistries respond differently to a nitrogen reflow environment. These solder pastes were subjected to a total of four different reflow profiles: a nitrogen ramp-to-peak (RTP) profile, a nitrogen soak profile, an air ramp-to-peak (RTP) profile, and an air soak profile. Per J-STD-004B, IPC-TM-650 2.6.3.3 and 2.6.3.7 are the instructions used for SIR testing. In these procedures, solder paste is stencil printed on to IPC-B-24 SIR boards using a .006”/150 μ thick stencil. Each B-24 SIR board has four SIR patterns; A through D. Three SIR boards are prepared per paste per scenario. Also, two controls are prepared. The SIR data from these boards will be shared and compared.

Introduction

There are two aspects to rendering a rosin-based flux residue as “no-clean,” or electrically reliable. One is simply the excellent encapsulating, immobilizing, and insulating properties that the rosin provides. Generally, in order to create conductive paths in the flux residue, such as dendrites, materials in the flux residue, such as ionic species, need to be able to move around under the influence of the electrical bias. Rosin is very effective at mitigating this mobility. Also, for this sort of mobility to occur there is a need for a medium through which the materials can travel. This medium is generally moisture, as rosin acts well as a moisture barrier. The second aspect is the activators. Activators are the ingredients in the flux responsible for oxide removal. Of all the ingredients in the flux, activators have the highest propensity to cause corrosion, which contributes to the formation of conductive paths. For no-clean fluxes, activators are specifically chosen to decompose at elevated temperatures in order to leave a benign flux residue. Much of this decomposition is thermally induced, however, the activators are also “consumed” as a result of their interaction with metal oxides.

It is this second aspect that some have thought to contribute to differences in the electrical reliability of a flux residue. When reflow of a PCB assembly is conducted in air, a certain amount of oxidation occurs to the solder powder, PCB pads, and component leads during the reflow process. Conversely, there would be little additional oxidation that occurs in a nitrogen reflow environment. Therefore, theoretically, more activators will be consumed during air reflow than compared to nitrogen reflow, all else being equal. With that in mind, the question arises as to whether or not that difference in activator consumption will contribute to any noticeable difference in the electrical reliability of the flux residue. Or, does the thermally-induced decomposition of the activators and encapsulating properties of the rosin in the flux residue override any differences that may be seen due to activator consumption?

Experimental

The instructions for SIR testing can be found in IPC J-STD-004B, IPC-TM-650 2.6.3.3 and 2.6.3.7. In these procedures, solder paste is stencil printed onto IPC-B-24 SIR boards using a .006”/150 μ m thick stencil. Each B-24 SIR board has four SIR patterns, A through D (Figure 1). Three SIR boards are prepared per paste per scenario. For example, a total of twelve boards were prepared for each paste. Of the twelve boards for each paste, three boards were reflowed for each of the four

reflow profiles. Two controls are also prepared. The control boards are clean and bare B-24 SIR boards. See Table 1. Figures 2 and 3 show the reflow profiles—Ramp-to-Peak and Soak—that were used with both air and nitrogen. Table 2 shows a summary of the reflow profiles.

After printing and reflow, the boards are put in a chamber that is 40°C and 90%RH. A DC bias is applied to the patterns to create a field strength of 25V/mm. A resistance reading is taken at least once every 20 minutes over the course of 168 hours (1 week). All readings after 24 hours must be $\geq 100M\Omega$ ($1 \times 10^8\Omega$) in order to constitute a pass. There is also a limit as to what degree dendrites can grow. However, this is not critical to this work because known good solder pastes are being used, so no dendritic growth is expected.

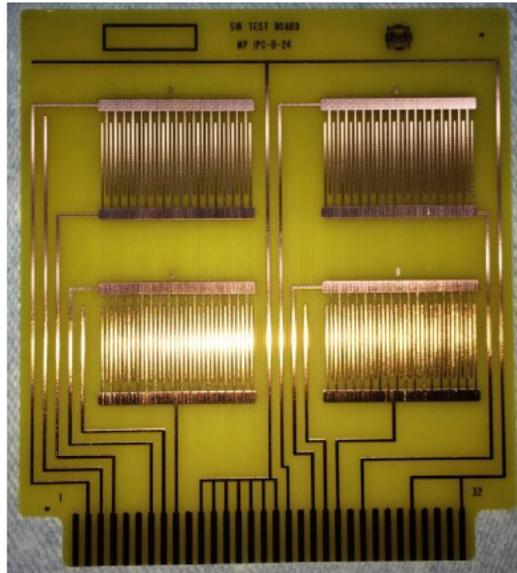
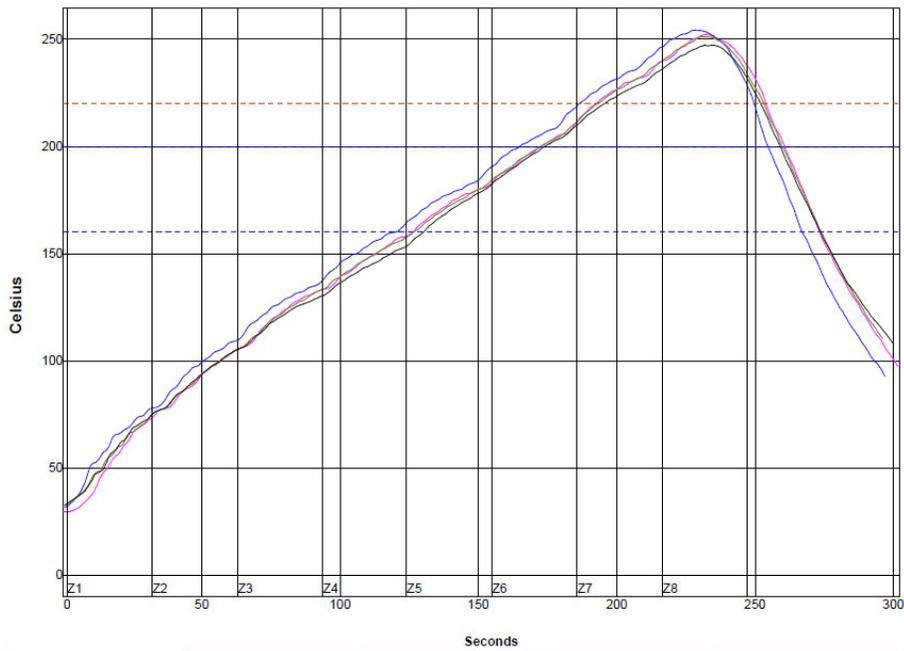


Figure 1 – IPC-B-24 SIR Board

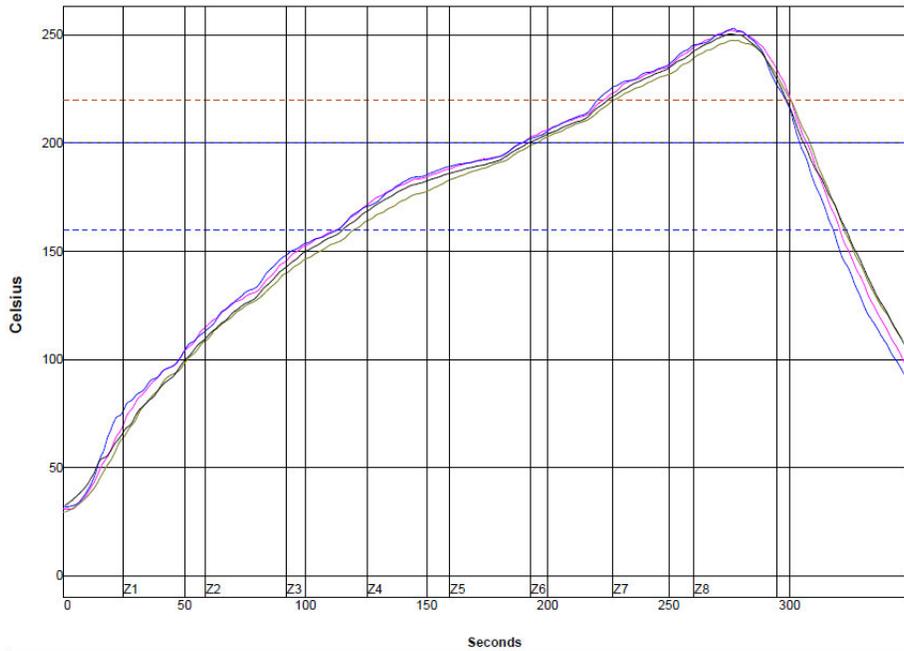
Table 1 – Test Matrix and Board Count

	Reflow Profile/Environment				
	N ₂		Air		
Solder Paste	RTP	Soak	RTP	Soak	
ROL0 Trad. Res.	3	3	3	3	
ROL1 Prob Res.	3	3	3	3	
ROL1 Trad. Res	3	3	3	3	
Controls					2
Total					38



PWI= 160%	Soak Time 160-200C		Reflow Time /220C		Peak Temp		Tot Time /220C	
<TC2>	46.08	-120%	61.41	5%	252.51	133%	61.41	-91%
<TC3>	45.27	-124%	63.27	11%	254.50	160%	63.27	-78%
<TC4>	45.98	-120%	60.36	1%	251.39	119%	60.36	-98%
<TC5>	43.76	-131%	56.55	-12%	247.34	65%	56.55	-123%
Delta	2.32		6.72		7.16		6.72	

Figure 2 – Ramp-to-Peak (RTP) Reflow Profile (N₂ and Air)



PWI= 141%	Soak Time 160-200C		Reflow Time /220C		Peak Temp		Tot Time /220C	
<TC2>	76.06	30%	77.39	58%	252.17	129%	77.39	16%
<TC3>	75.83	29%	77.72	59%	253.06	141%	77.72	18%
<TC4>	75.59	28%	73.01	43%	247.50	67%	73.01	-13%
<TC5>	76.65	33%	73.70	46%	250.40	105%	73.70	-9%
Delta	1.06		4.71		5.56		4.71	

Figure 3 – Soak Reflow Profile (N₂ and Air)

Table 2 – Reflow Profile Averages and Summary

Profile Name	Avg. Soak Time (160°C-200°C)	Avg. Reflow Time (Over 220°C)	Avg. Peak Temp.
Ramp to Peak	45.27 sec	60.4 sec	251.44°C
Soak	76.03 sec	75.46 sec	250.78°C

Results and Discussion

After the boards were printed and reflowed they were submitted for SIR testing per IPC-TM-650 2.6.3.7 for 168 hours. Figures 4 through 17 show the graphed SIR values for each individual scenario. It should be noted that Figure 5 shows the results of the halogen-free N₂ soak scenario after a second attempt. The SIR boards for the first test of this scenario were found to be contaminated. A fresh set of boards for both this scenario and controls were then prepared and submitted for SIR testing. This is the reason why there are SIR results for a second set of controls (Figure 17).

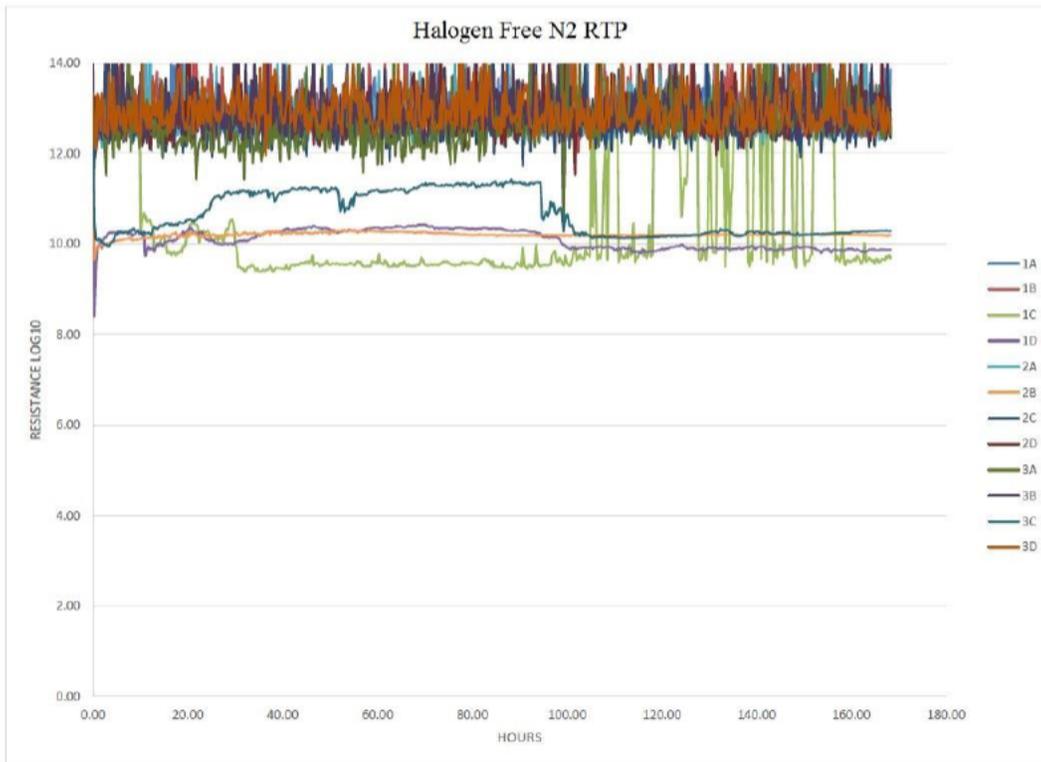


Figure 4 – Halogen-Free N₂ RTP

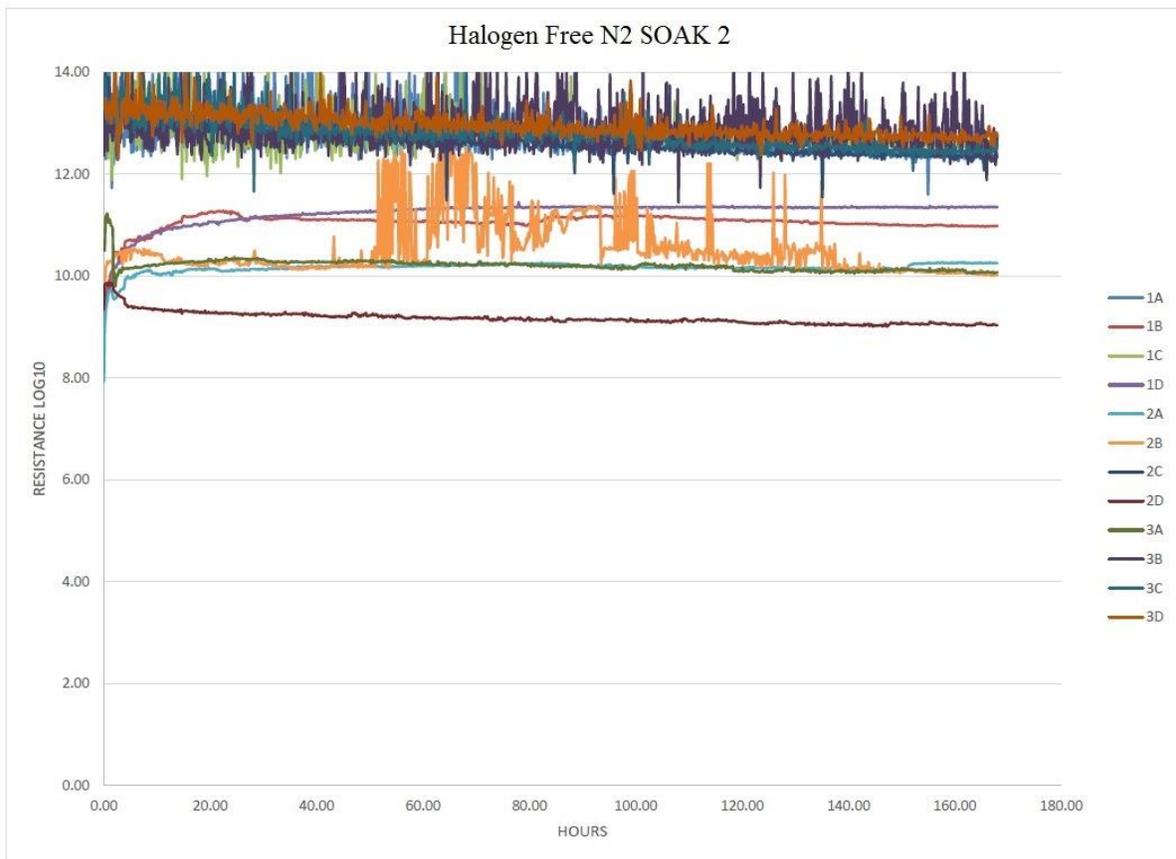


Figure 5 - Halogen-Free N₂ Soak (second test)

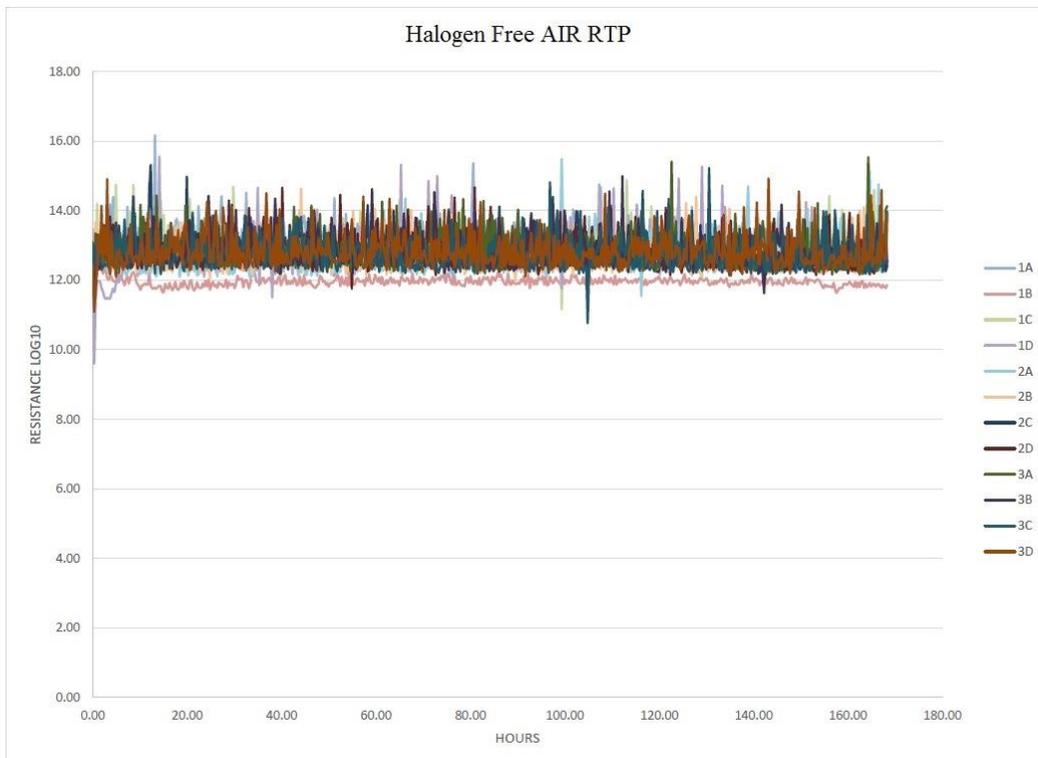


Figure 6 – Halogen-Free Air RTP

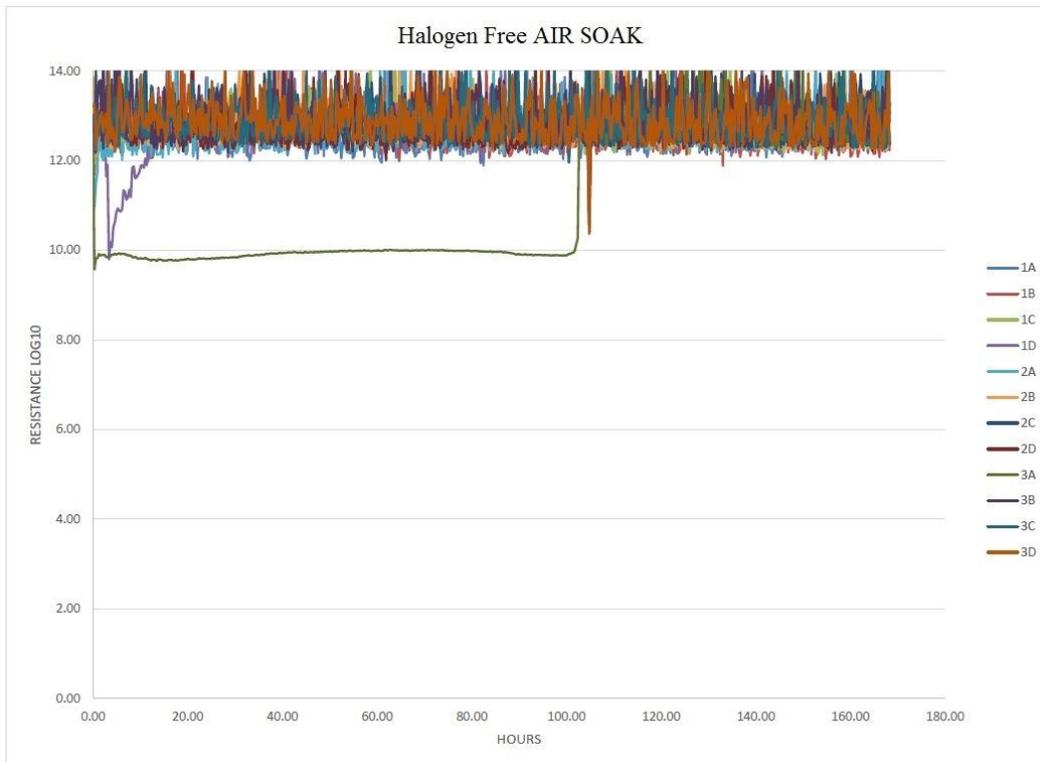


Figure 7 – Halogen-Free Air Soak

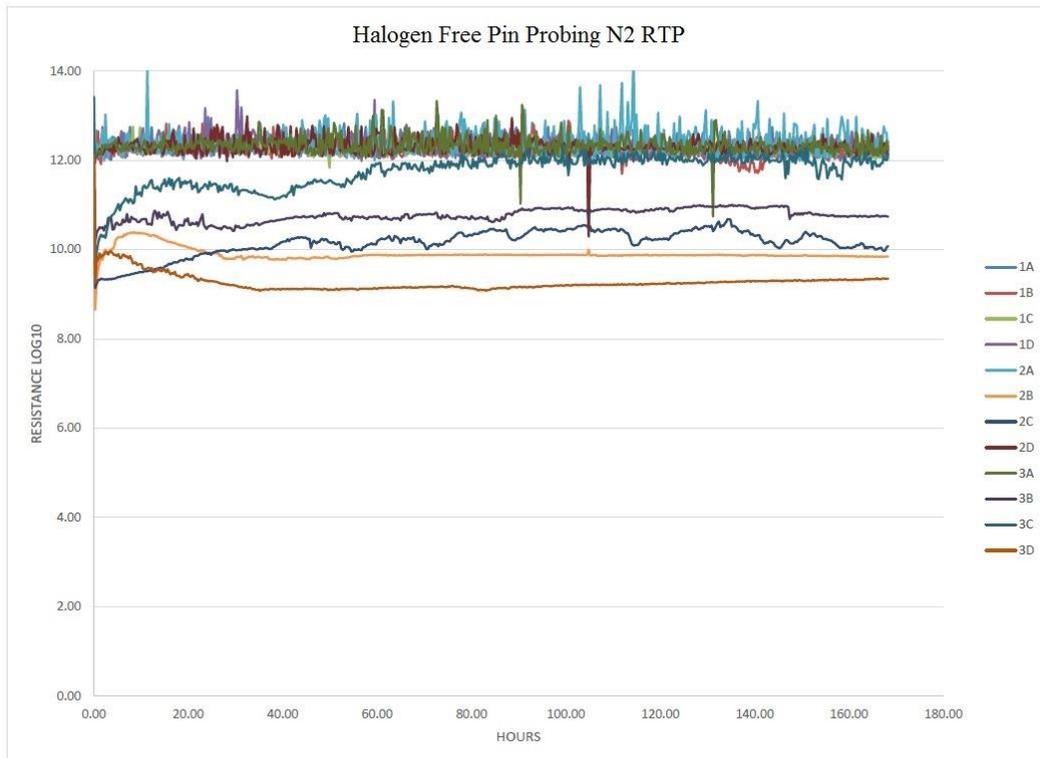


Figure 8 – Halogen-Free Pin-Probing N₂ RTP

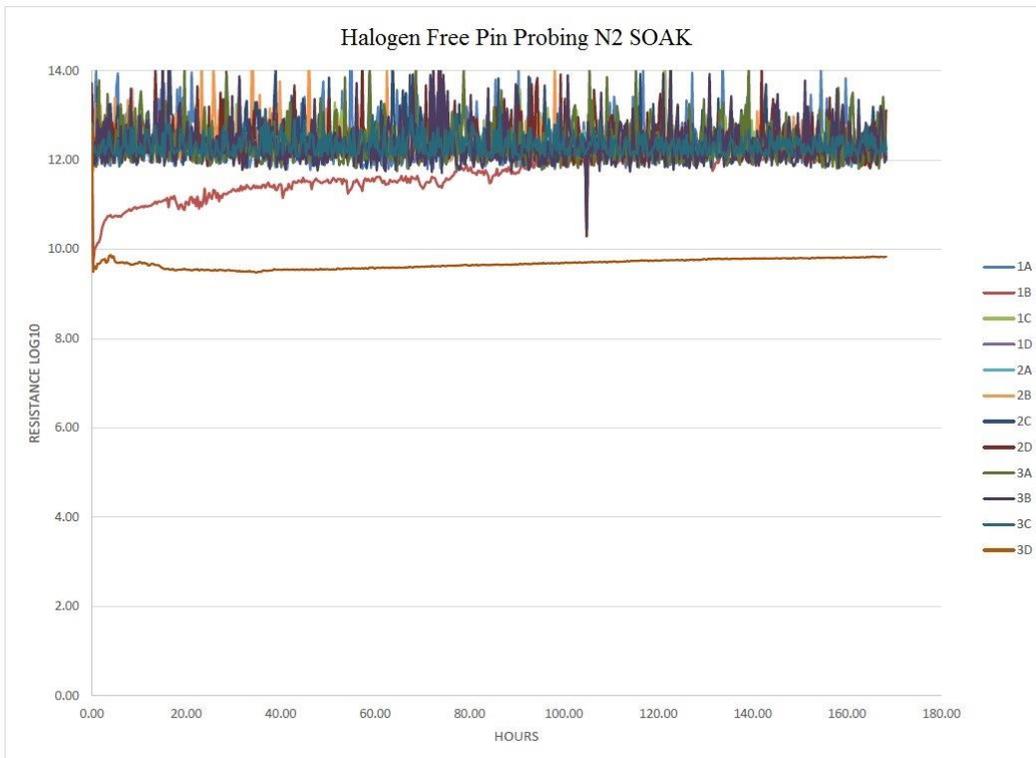


Figure 9 – Halogen-Free Pin-Probing N₂ Soak

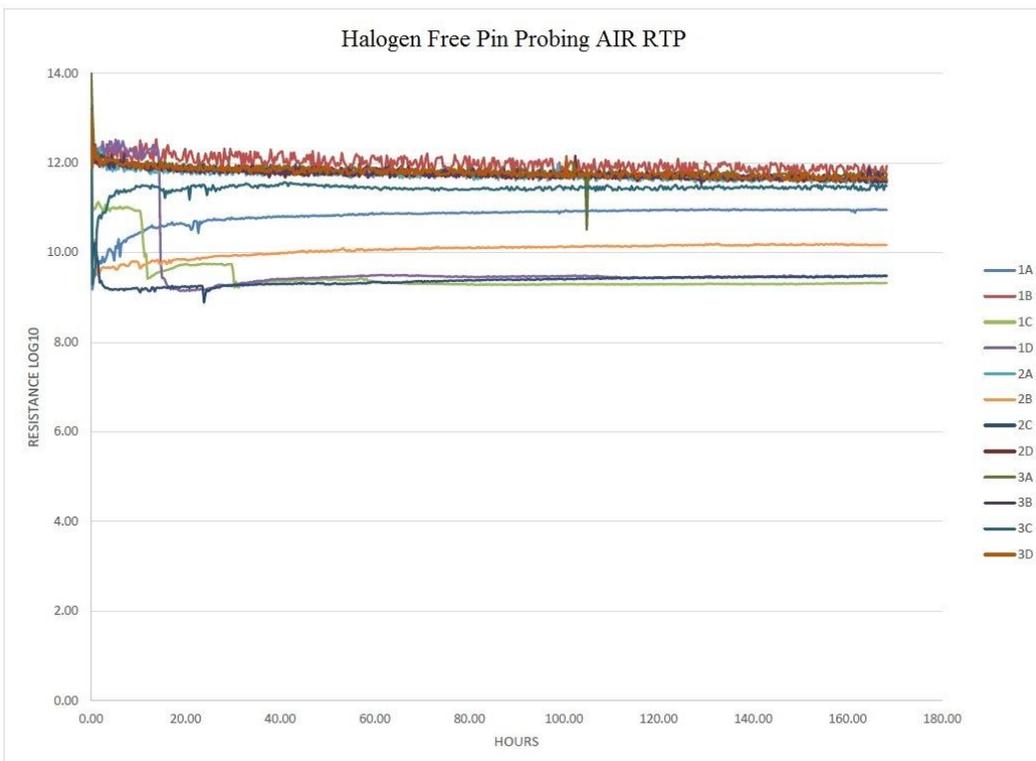


Figure 10 – Halogen-Free Pin-Probing Air RTP

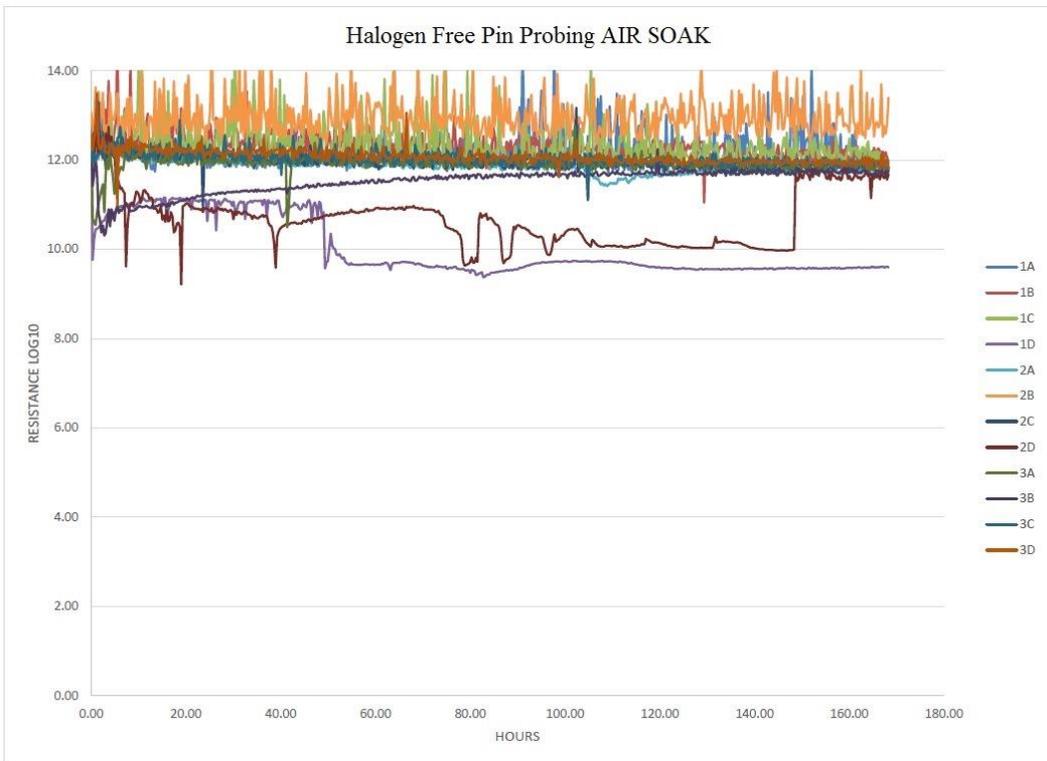


Figure 11 – Halogen-Free Pin-Probing Air Soak

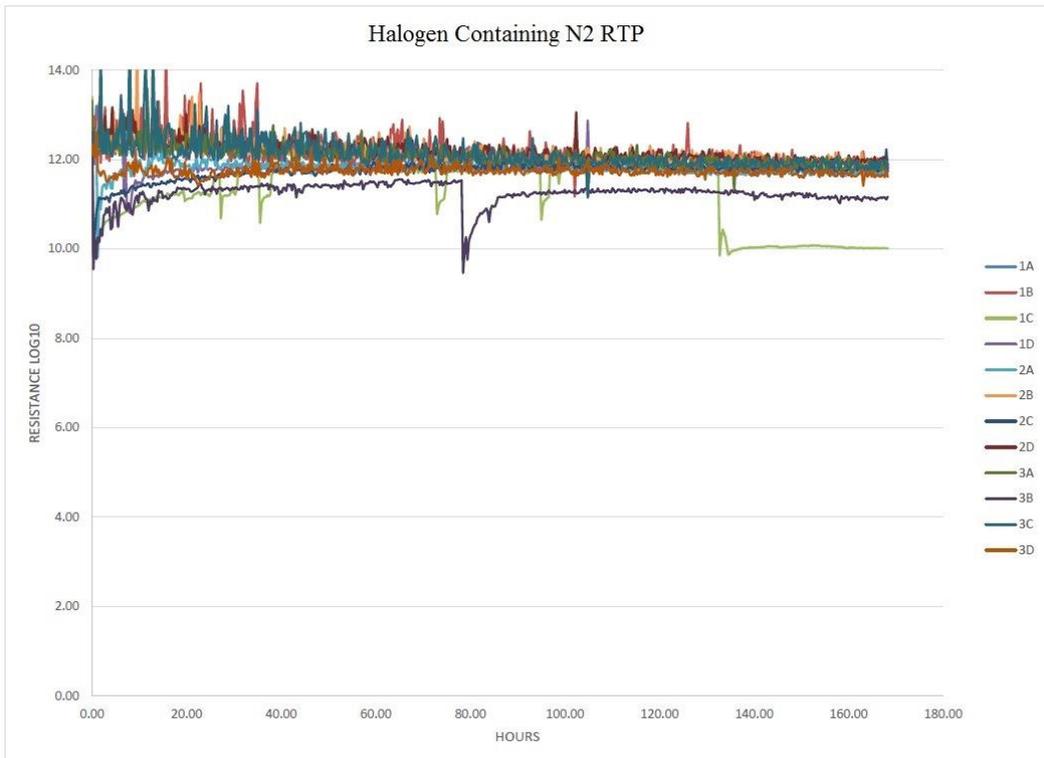


Figure 12 – Halogen-Containing N₂ RTP

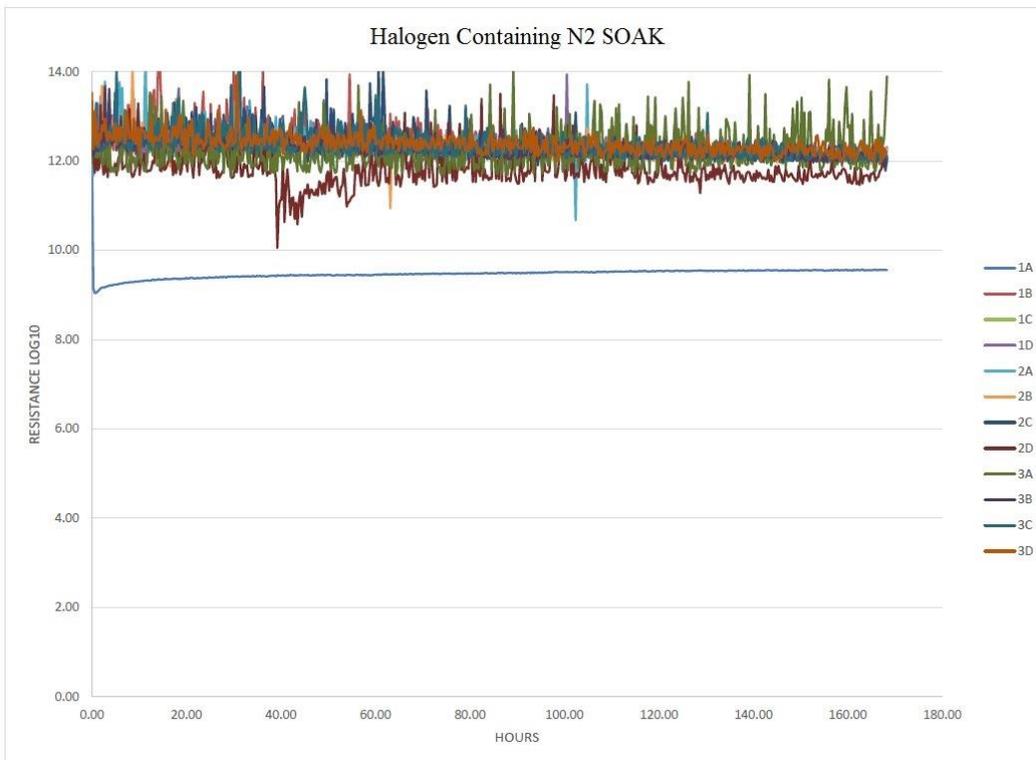


Figure 13 – Halogen-Containing N₂ Soak

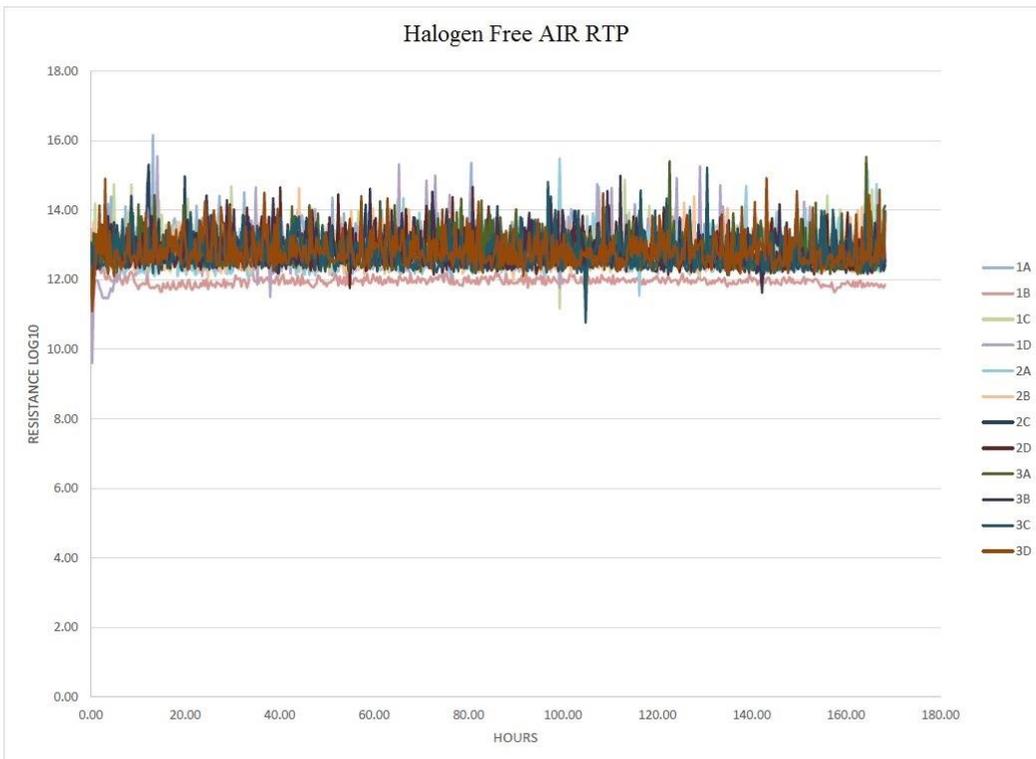


Figure 14 – Halogen-Free Air RTP

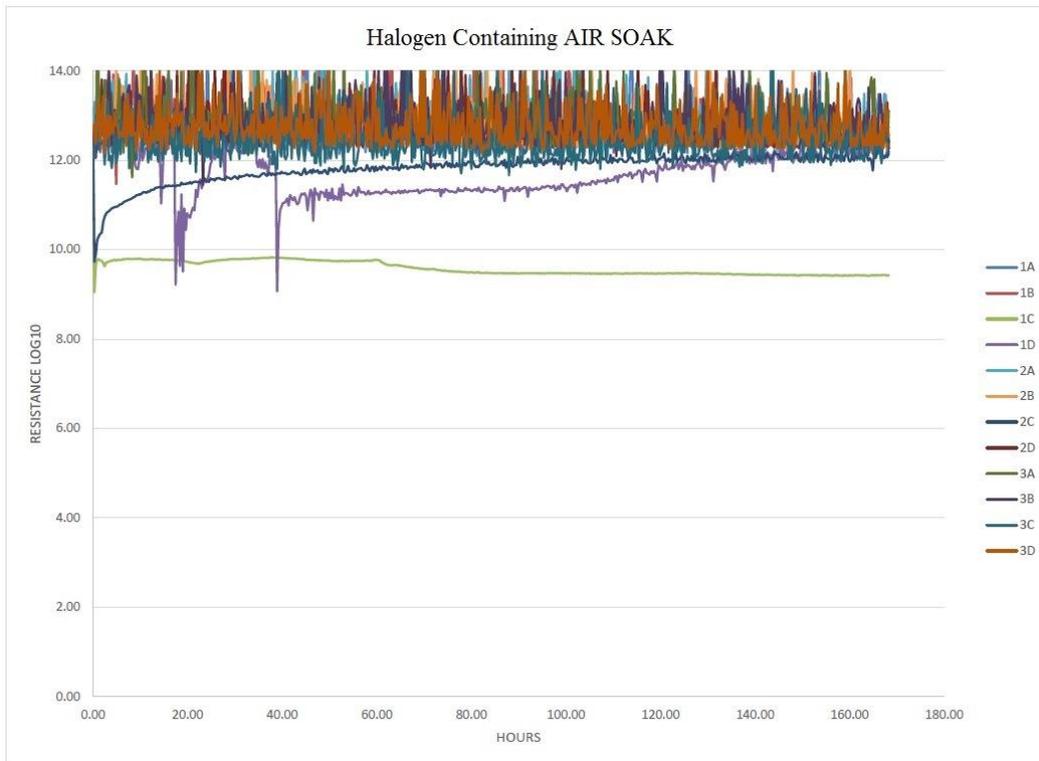


Figure 15 – Halogen-Containing Air Soak

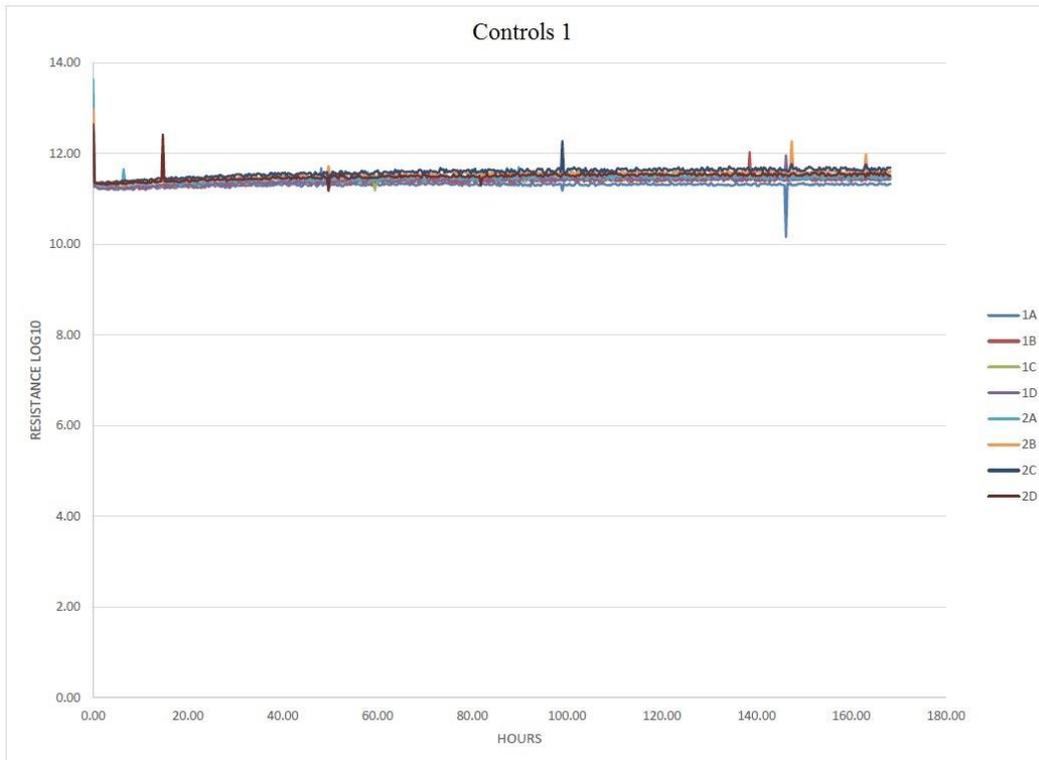


Figure 16 – Controls

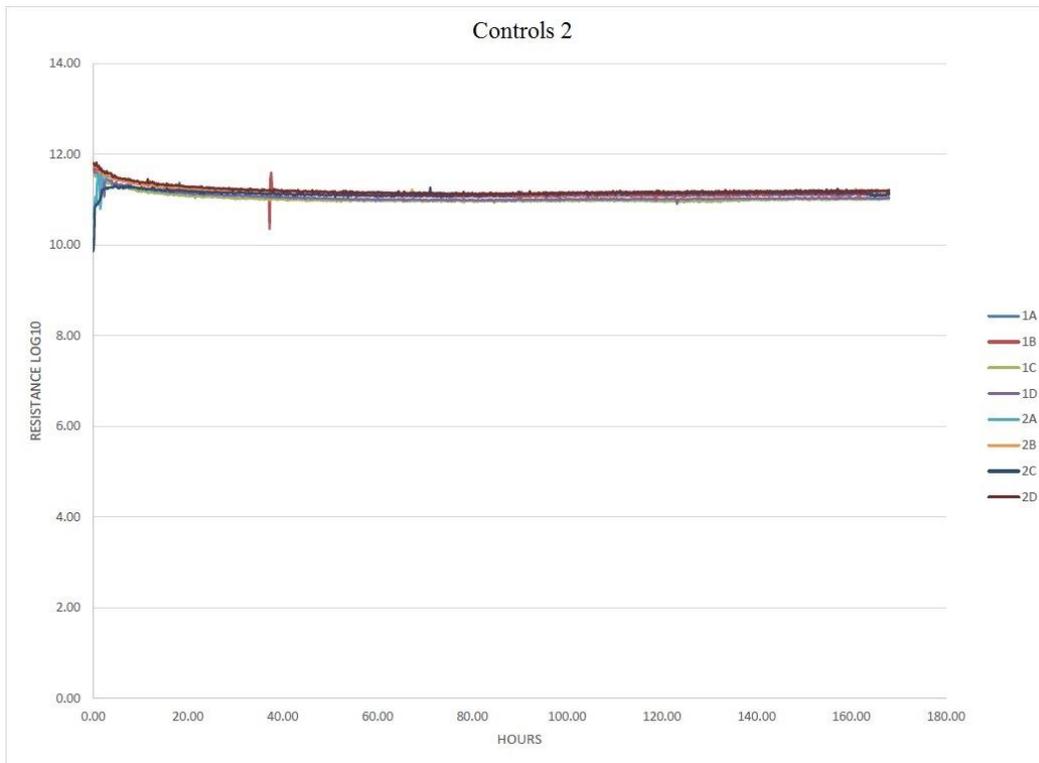


Figure 17 – Controls from Second Test of Halogen-Free N₂ Soak Second Test (Figure 5)

The SIR graphs in Figures 4 through 17 show all the readings in the interest of full disclosure. However, this may not be the best way to look at the general effect of reflowing solder paste in nitrogen versus air. To better detect any sort of trend related to the reflow environment, the SIR values of each scenario were averaged. The averaged SIR values for a given solder paste with each profile and reflow environment are plotted on a single graph in Figures 18 through 20. This allows us to examine how each unique solder paste flux chemistry responds. Figure 21 shows all of the averaged SIR values for all the pastes and scenarios plotted on the same graph.

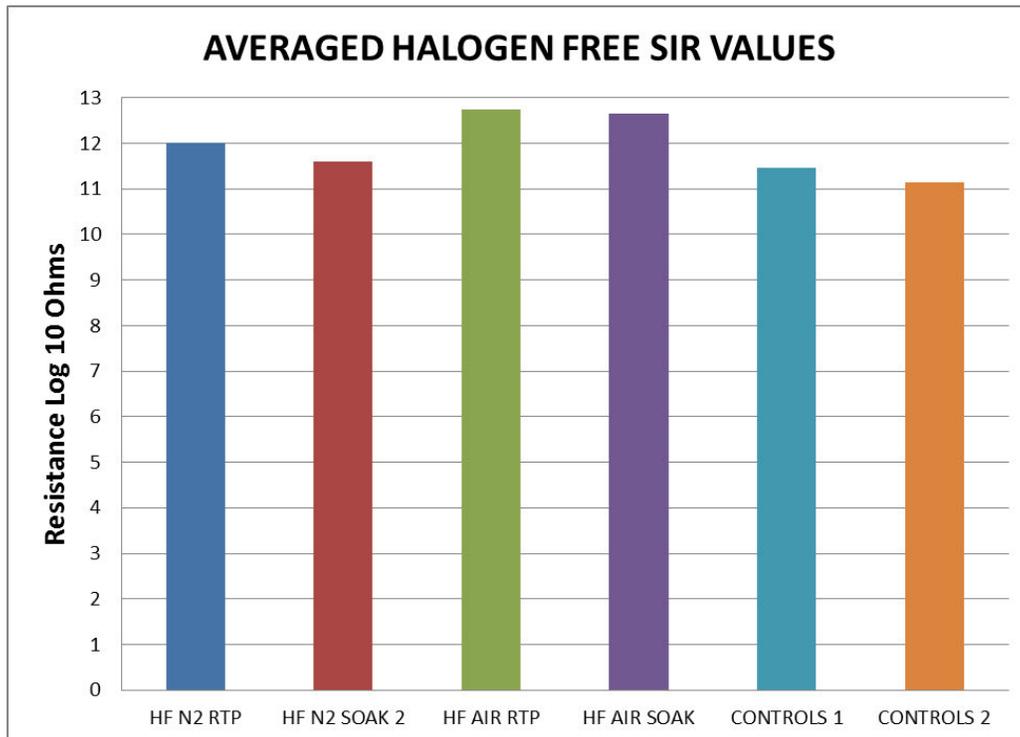


Figure 18 – Averaged Halogen-Free SIR Values

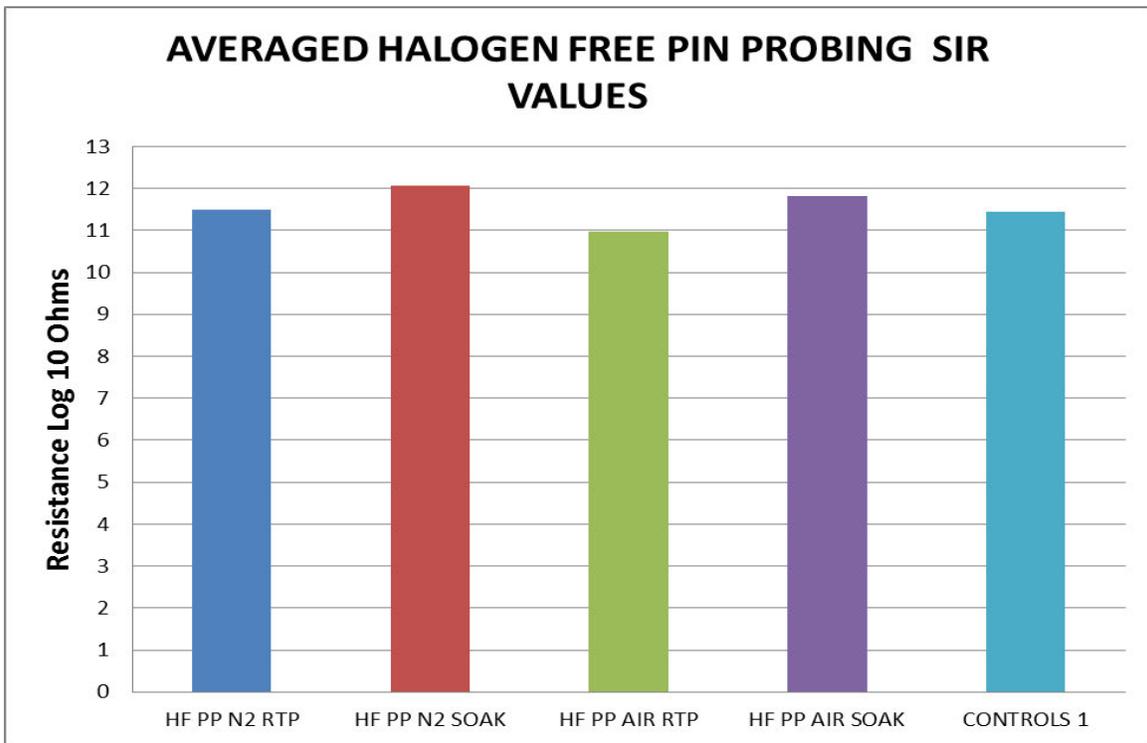


Figure 19 – Averaged Halogen-Free Pin-Probing SIR Values

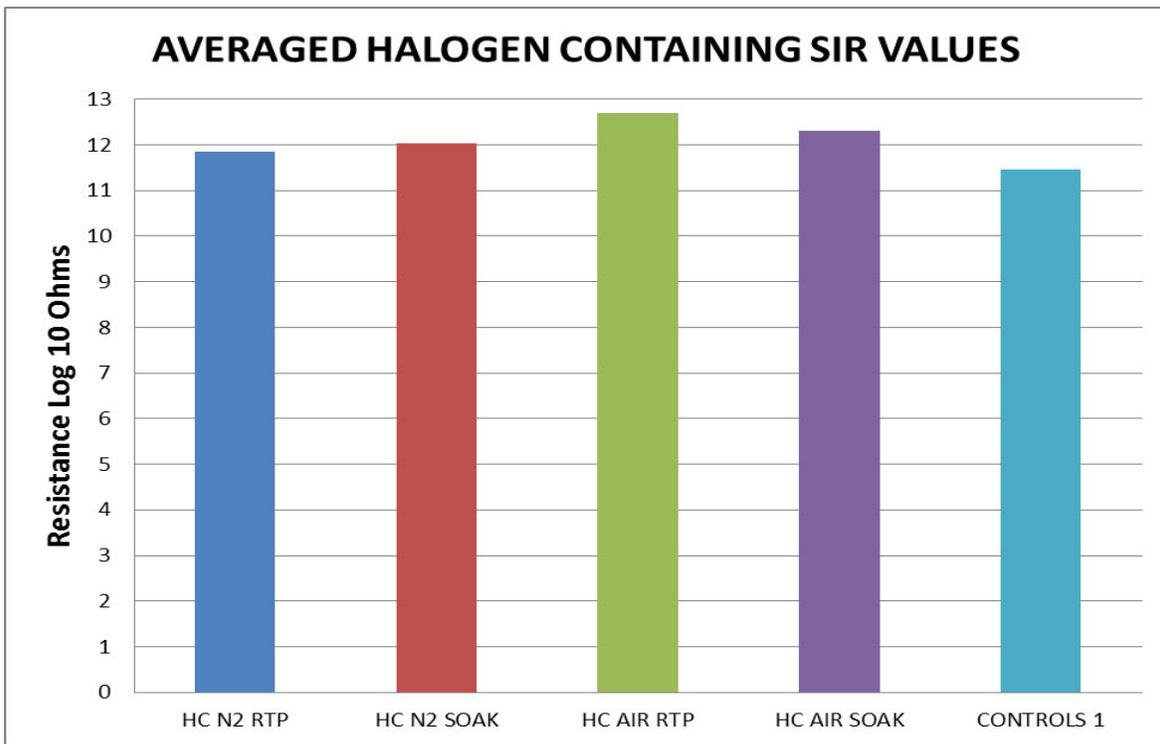


Figure 20 – Averaged Halogen-Containing SIR Values

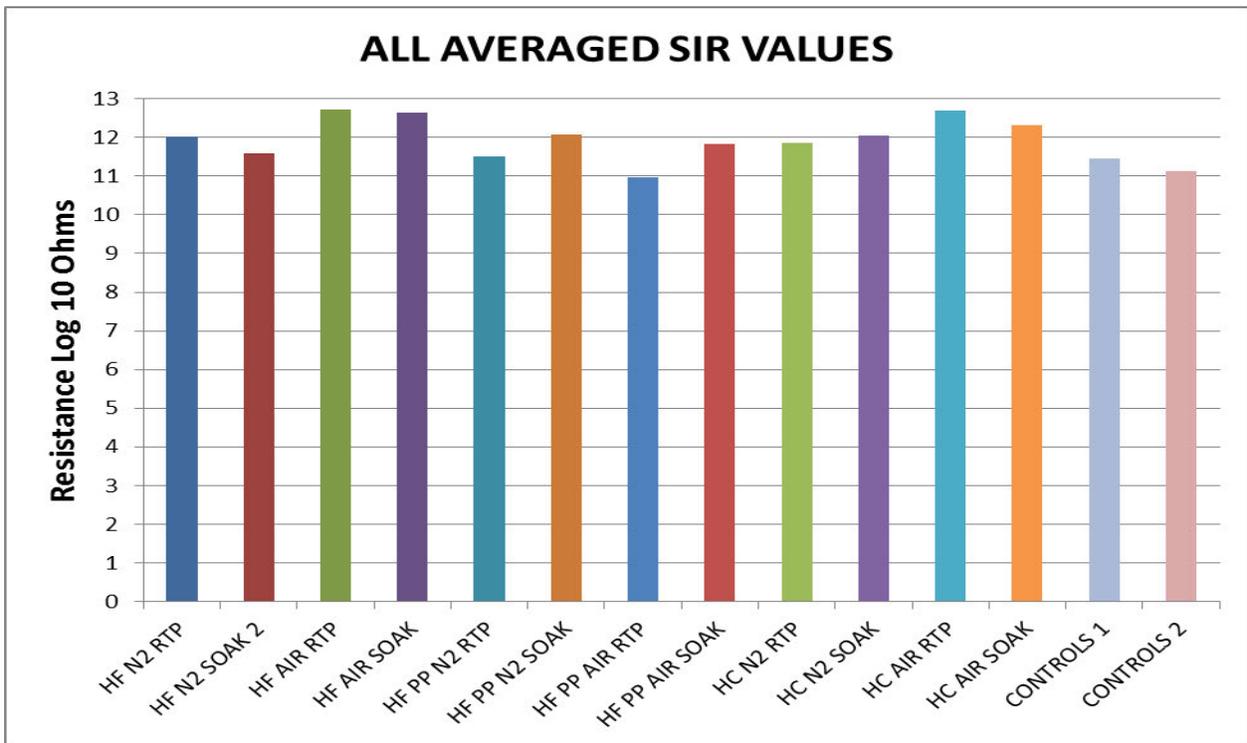


Figure 21 – All Averaged SIR Values

Of particular interest in the SIR performance of a solder paste flux residue is the lowest SIR value that was recorded during the test. Often, a single event such as a momentary reduction in the resistance of a flux residue can lead to catastrophic electrical failure of a PCBA. With that in mind, the lowest SIR values for each solder paste are graphed individually as well as in a graph showing all the solder pastes' lowest SIR values together. See Figures 22 through 25.

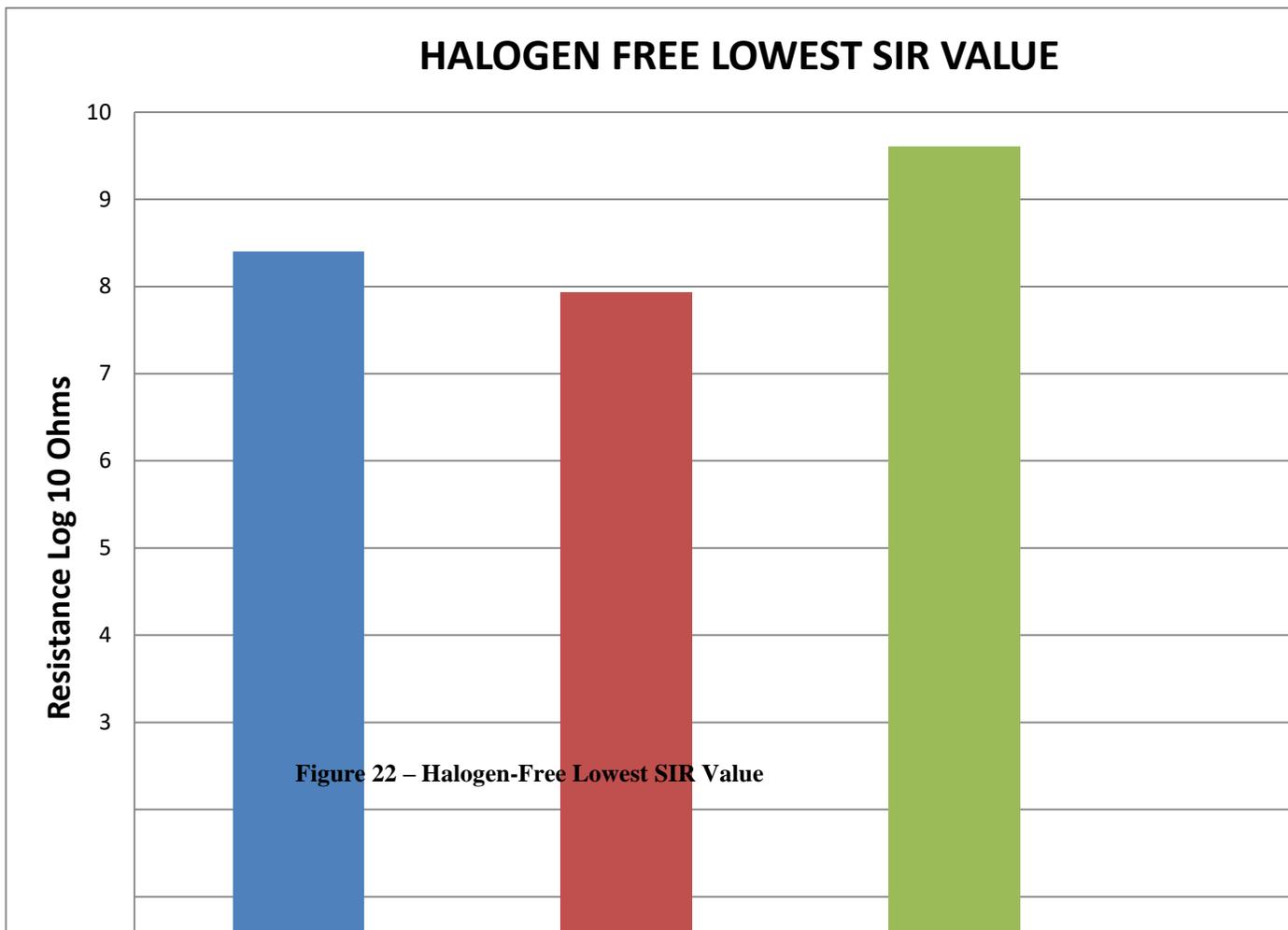
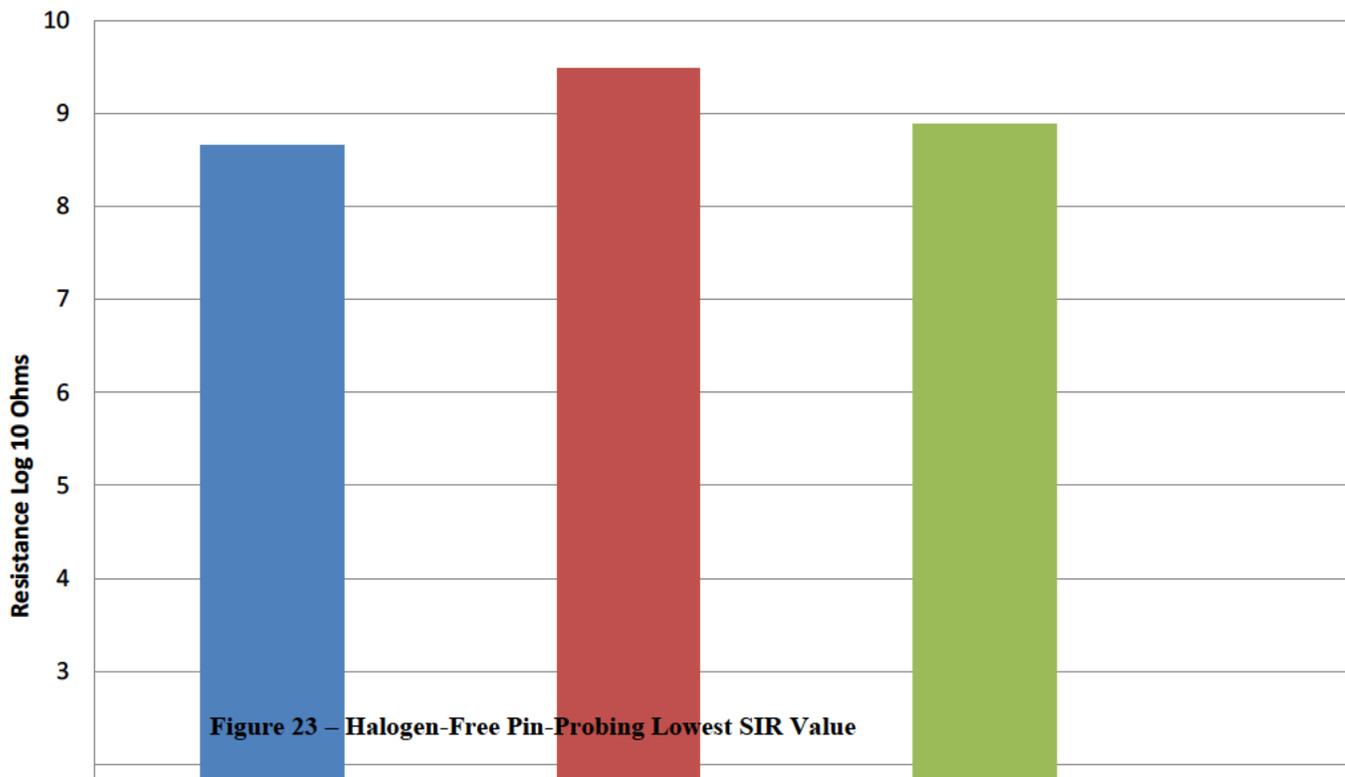
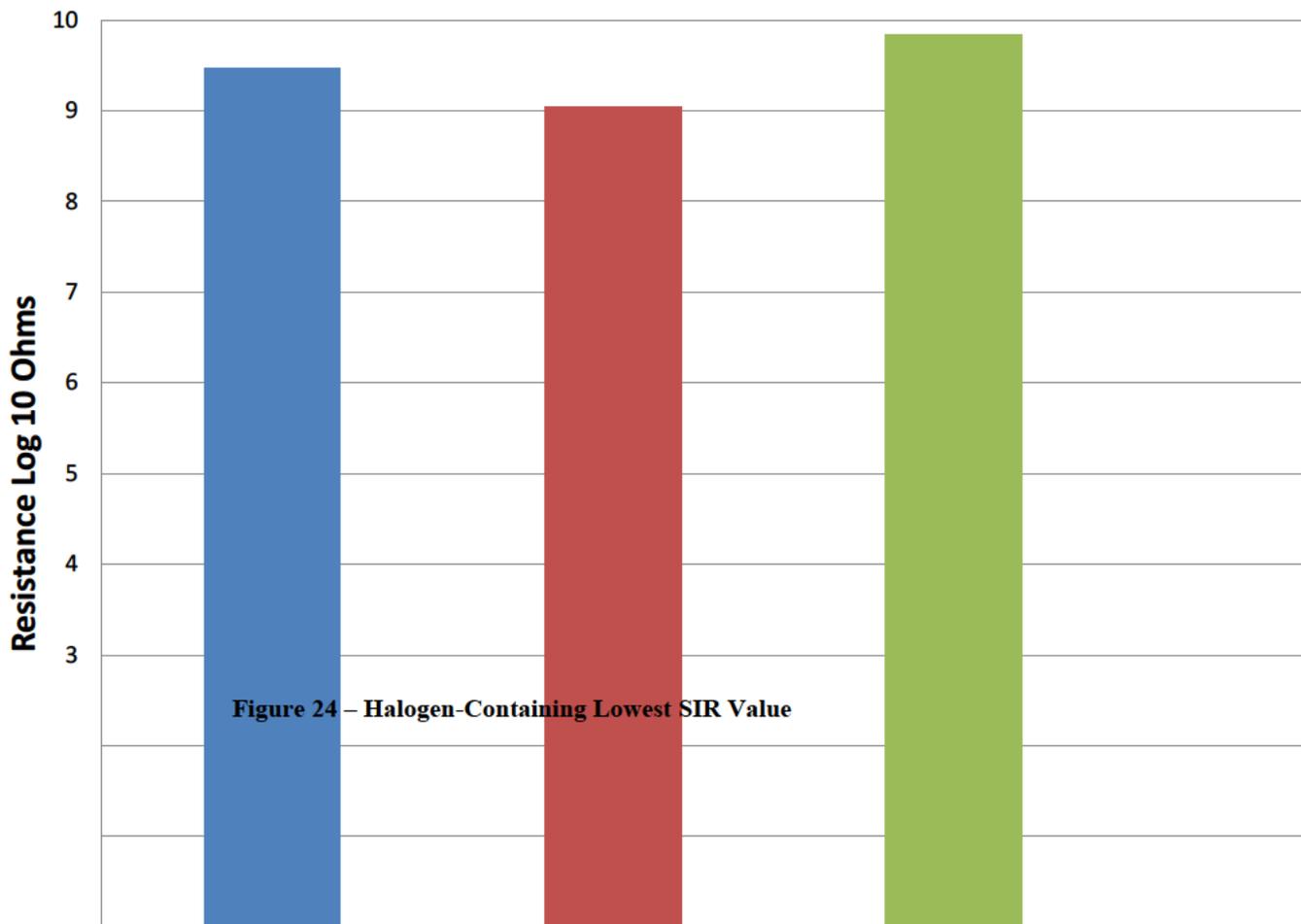


Figure 22 – Halogen-Free Lowest SIR Value

HALOGEN FREE PIN PROBING LOWEST SIR



HALOGEN CONTAINING LOWEST SIR VA



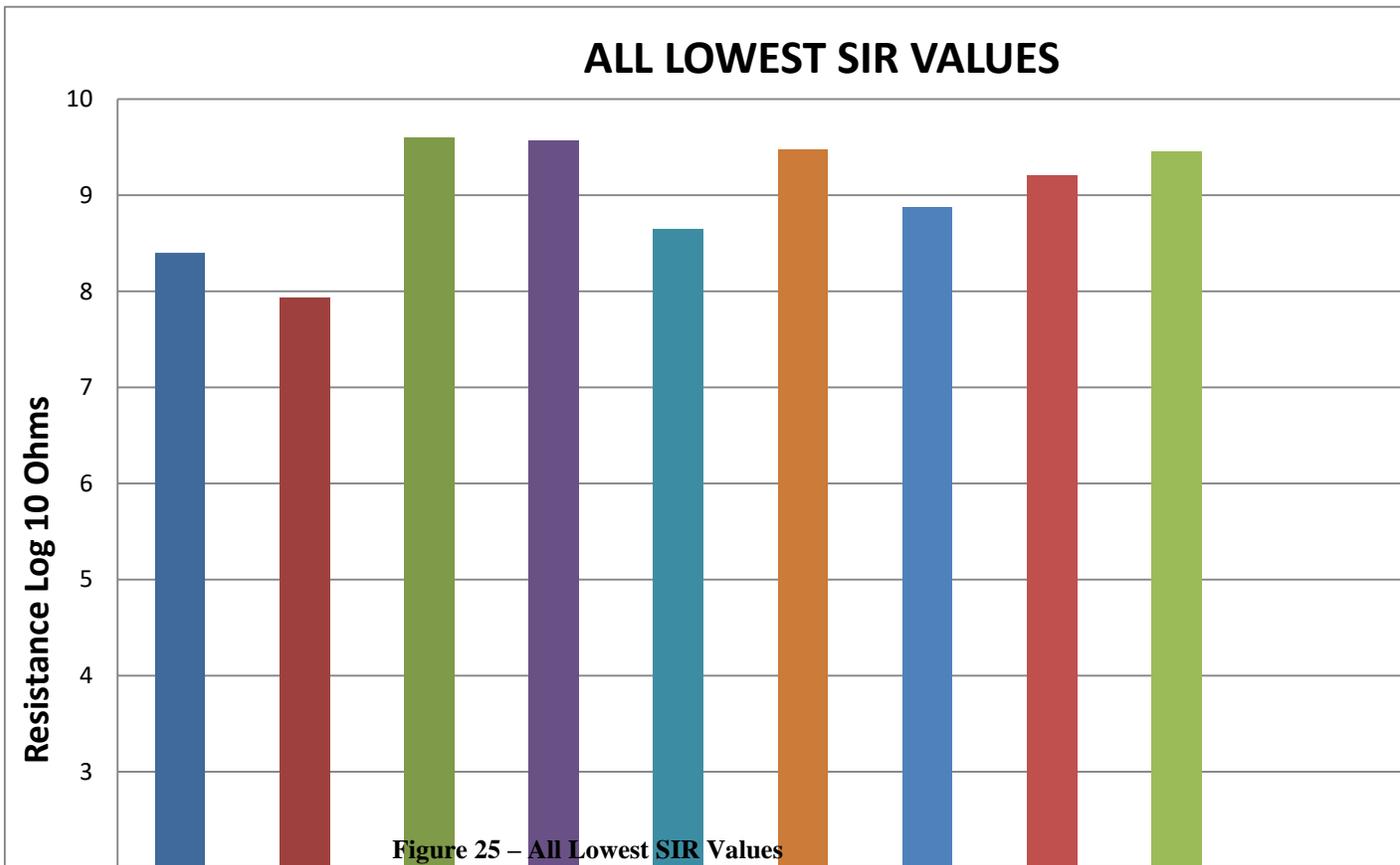


Figure 25 – All Lowest SIR Values

The lowest overall SIR value observed during this experiment was $1 \times 10^{7.93}$ Ohms, in which the halogen-free solder paste was submitted to a N_2 reflow environment along with the soak profile. See Figures 22 and 25. It should be noted that this value was recorded at time 0 (Figure 5). Per IPC J-STD-004B, because this value was recorded during the first 24 hours of the test, it does not constitute a failure even though it is below the 1×10^8 Ohms required minimum SIR value. Critical review of the SIR graphs of each scenario will reveal that many of the lowest SIR values were realized during the first 24 hours of the test and, therefore, are not relevant to making a “pass” or “fail” conclusion nor predicting the electrical reliability of the solder paste flux residue. With this in mind, this data is presented again showing the lowest SIR value for each scenario recorded after 24 hours into the test. See Figures 26 through 29.

HALOGEN FREE LOWEST SIR VALUE BEYOND 2

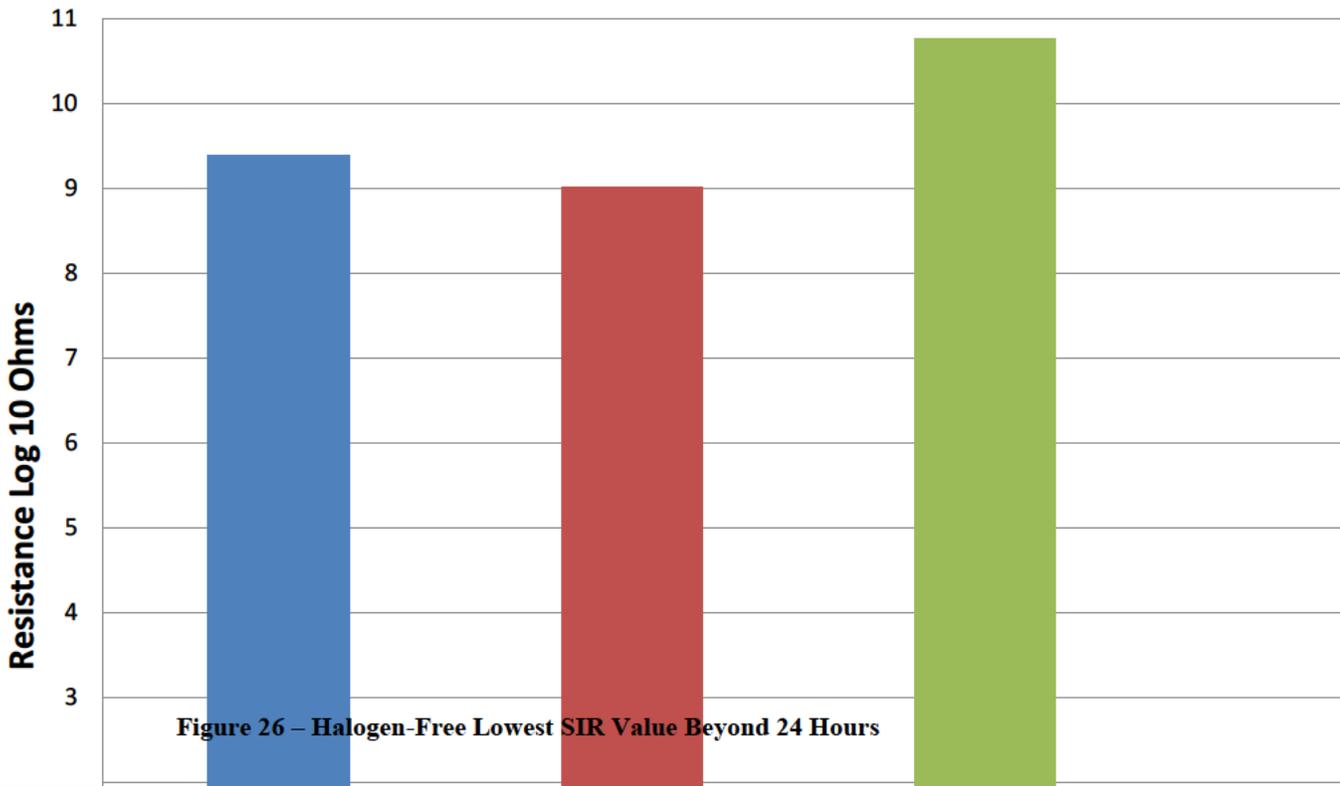


Figure 26 – Halogen-Free Lowest SIR Value Beyond 24 Hours

HALOGEN FREE PIN PROBING LOWEST SIR VALUE HOURS

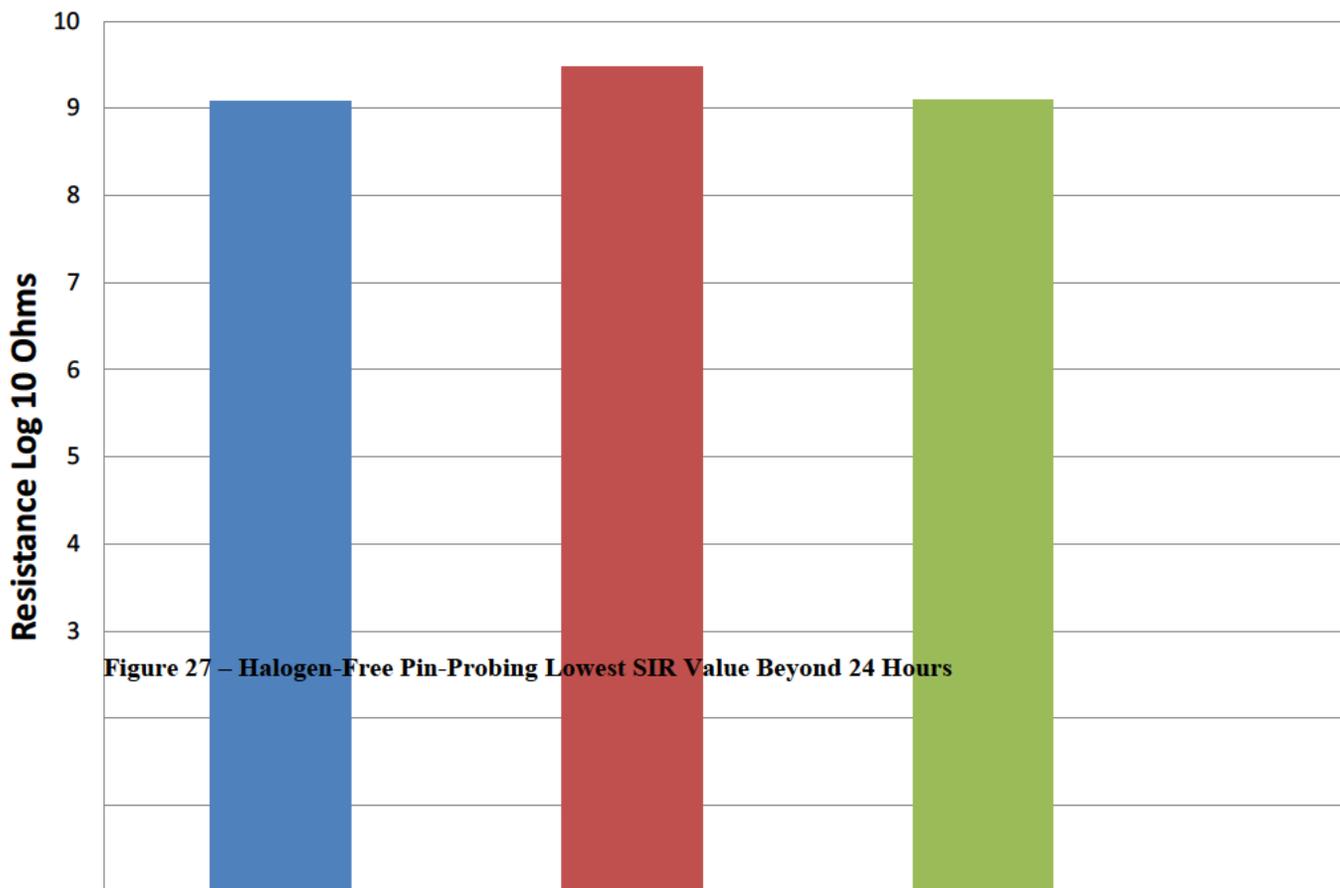
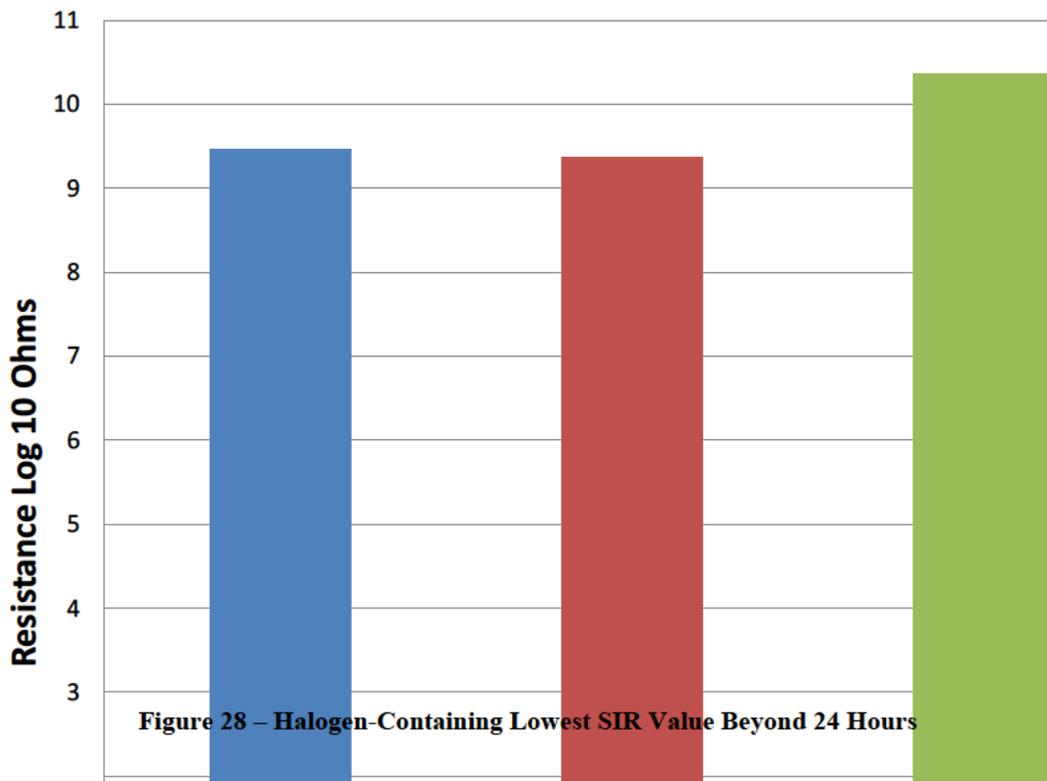
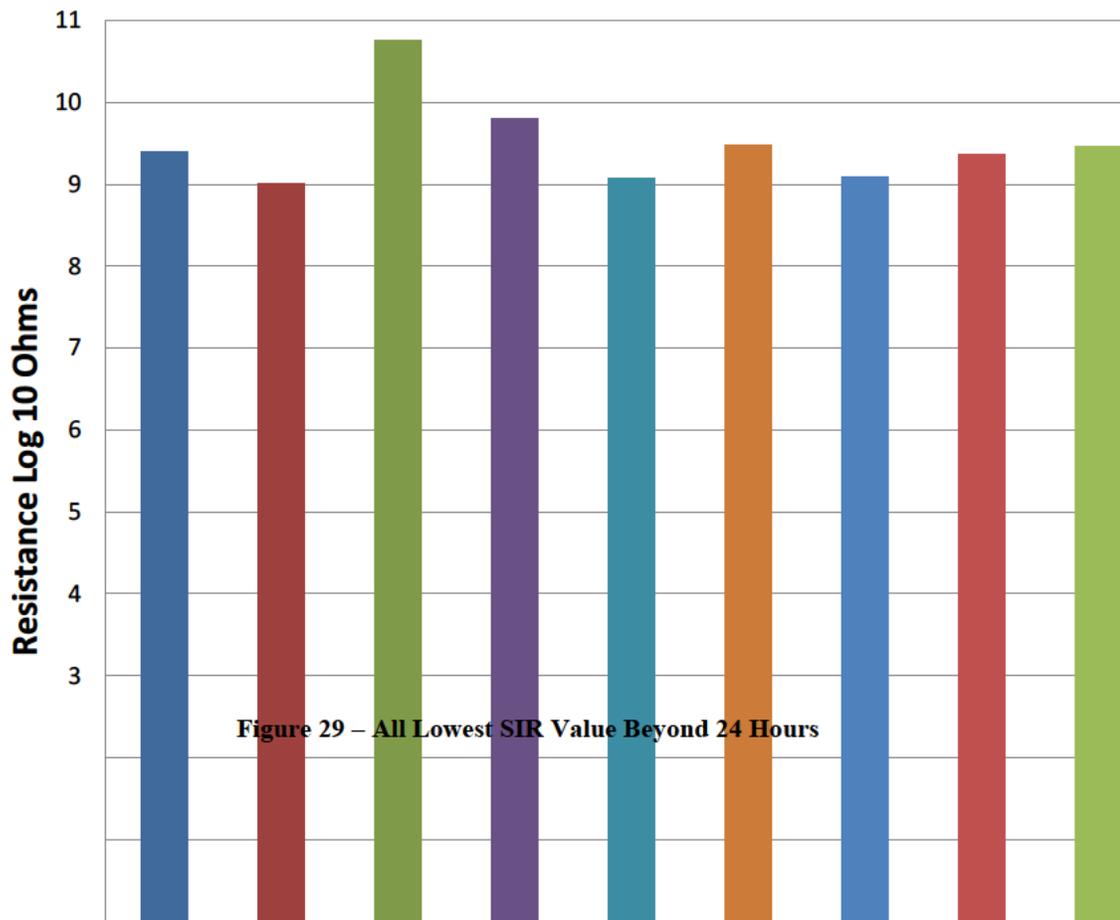


Figure 27 – Halogen-Free Pin-Probing Lowest SIR Value Beyond 24 Hours

HALOGEN CONTAINING LOWEST SIR VALUE BEYO



ALL LOWEST SIR VALUE BEYOND 24 HOU



The lowest SIR value recorded beyond 24 hours is $1 \times 10^{9.01}$ Ohms and was seen with the halogen-free solder paste when submitted to a N_2 reflow environment with the soak profile. See Figures 26 and 29. Interestingly, this is the same scenario that produced the overall lowest SIR value of $1 \times 10^{7.93}$ Ohms. See Figures 22 and 25.

Below are some observations about the results:

1. Averaged Halogen-Free Paste SIR Values (Figure 18)
 - a. The SIR values of the solder paste are higher than the bare boards (controls).
 - b. The SIR values achieved with the N_2 reflow environment are slightly lower than those achieved in air.
2. Averaged Halogen-Free Pin-Probing Paste SIR Values (Figure 19)
 - a. The SIR values achieved in air are slightly lower than those achieved in N_2 .
 - b. The SIR values are higher with a soak reflow profile than those achieved with a ramp-to-peak profile.
 - c. Only the air RTP profile produced slightly lower values than the controls.
3. Averaged Halogen-Containing Paste SIR Values (Figure 20)
 - a. The SIR values achieved in N_2 are slightly lower than those achieved in air.
 - b. All of the scenarios produced higher SIR values than the controls.
4. All Averaged Paste SIR Values (Figure 21)
 - a. The halogen-free and halogen-containing pastes when reflowed in air show the overall highest SIR values.
 - b. The halogen-free pin-probing paste when reflowed in air showed the overall lowest SIR performance.
 - c. All values are comparable to or higher than the controls.
 - d. All values are 3 or more magnitudes higher than the IPC required minimum of $100M\Omega$.
5. Halogen-Free Lowest SIR Value (Figure 22)
 - a. N_2 produced lower SIR values than air.
6. Halogen-Free Pin-Probing Lowest SIR Value (Figure 23)
 - a. The RTP profile produced lower SIR values than the soak profile.
7. Halogen-Containing Lowest SIR Value (Figure 24)
 - a. The soak profile produced lower SIR values than the RTP profile.
8. All Lowest AIR Values (Figure 25)
 - a. The halogen-free paste when reflowed in N_2 produced the lowest values.
 - b. The halogen-free paste when reflowed in air produced the highest values.
9. Halogen-Free Lowest SIR Value Beyond 24 Hours (Figure 26)
 - a. N_2 produced lower SIR values than air.
 - b. The RTP profile produced slightly higher values than the soak profile.
10. Halogen-Free Pin-Probing Lowest SIR Value Beyond 24 Hours (Figure 27)
 - a. The soak profile produced slightly higher SIR values than the RTP profile.
11. Halogen-Containing Lowest SIR Value Beyond 24 Hours (Figure 28)
 - a. The air RTP profile produced a higher SIR value than the other scenarios.
12. All Lowest SIR Value Beyond 24 Hours (Figure 29)
 - a. The lowest SIR value after 24 hours is an order of magnitude higher than the IPC minimum of $100M\Omega$ (1×10^8 Ohms).

Conclusions

By looking at the averaged SIR values, it appears that there is no clear cut, meaningful trend. Regardless of the reflow conditions, all of the averaged SIR values are comparable to or higher than the bare control coupons and are 3 or more orders of magnitude higher than the minimum IPC value of $100M\Omega$ (1×10^8 Ohms). The lowest SIR value measured after 24 hours is ~1 order of magnitude higher than the IPC minimum. The differences that have been highlighted throughout the observations and elsewhere in this paper are likely too insignificant to make any definitive statements that one paste or reflow scenario is better than another. For all intents and purposes, modern no-clean solder pastes seem to be relatively immune to differences in the reflow environment where SIR is concerned and should create no concern that one reflow environment is more likely to create an SIR failure than another.

The Effect of a Nitrogen Reflow Environment on the Electrical Reliability of Rosin Based No-Clean Solder Paste Flux Residues

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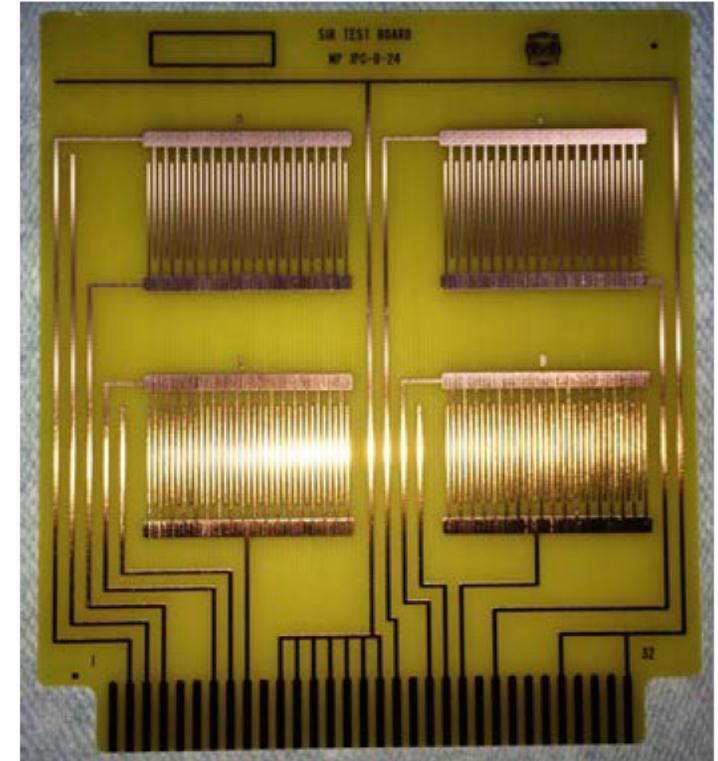
ebastow@indium.com

Background and Theory

- “No-Clean” Flux Technology
 - Rosin Encapsulation
 - Activator Selection
 - Oxide Removal
 - “Corrosive” – Effect SIR Performance
- Air Reflow
 - Existing Oxides
 - Additional Oxide Formation
 - Greater Activator Consumption?
 - Effects SIR Performance?
- Nitrogen Reflow
 - Existing Oxides
 - Negligible Additional Oxide Formation
 - Lesser Activator Consumption?
 - Effects SIR Performance?

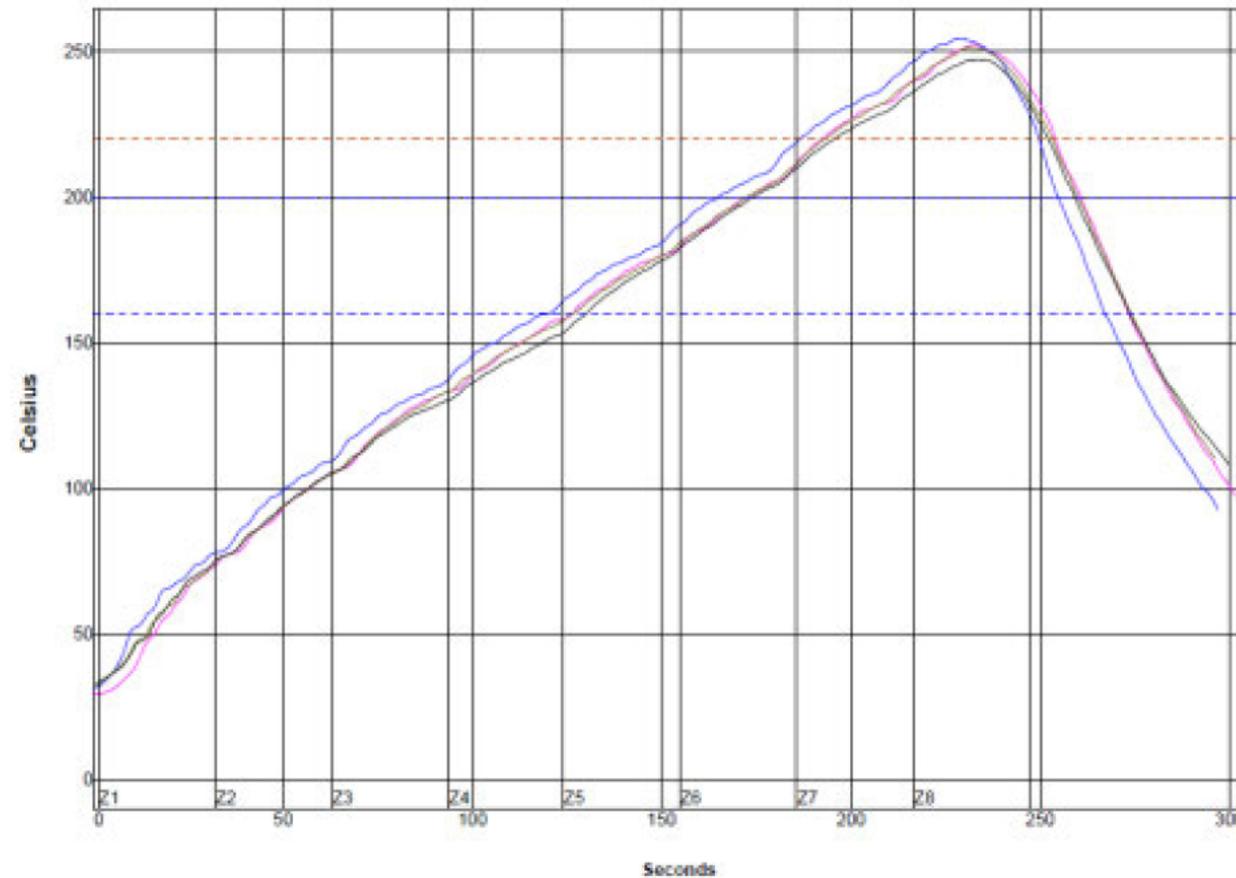
J-STD-004B SIR: IPC –TM-650 2.6.3.3 and 2.6.3.7

- Cleaned
- Printed (150 μ /.006")
- Reflowed
- 168 Hours
 - 40C/90%RH
 - 25V/mm Field Strength
 - Resistance Reading Every 20 Minutes
- All Readings After 24 Hours $\geq 100\text{M}\Omega$ (1×10^8 Ohms)



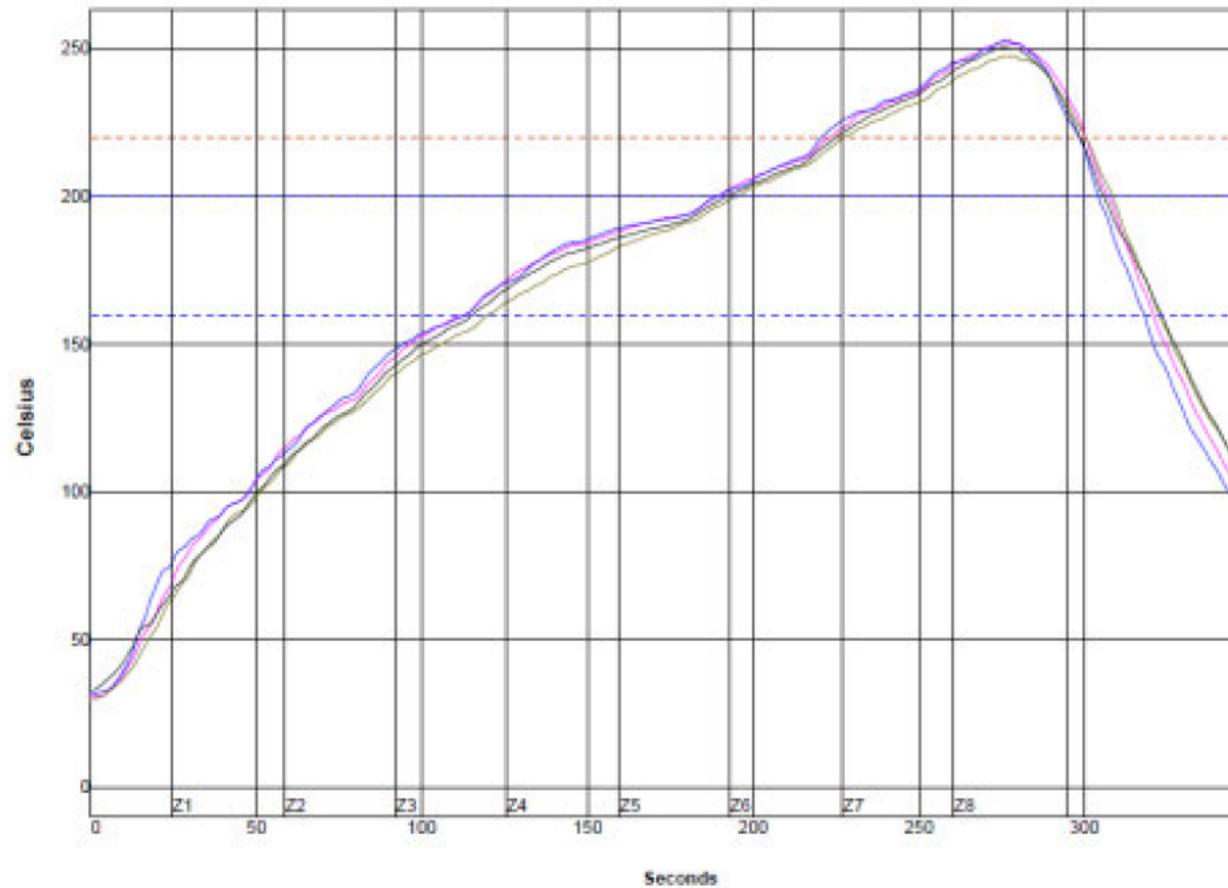
	Reflow Profile/Environment				
	N2		Air		
	RTP	Soak	RTP	Soak	
Solder Paste					
ROL0 Trad. Res.	3	3	3	3	
ROL0 Probe Res.	3	3	3	3	
ROL1 Trad. Res.	3	3	3	3	
Controls					2
Total					38

RAMP TO PEAK aka "RTP"



PWI= 160%	Soak Time 160/200C		Reflow Time /220C		Peak Temp		Tot Time /220C	
<TC2>	46.68	-120%	61.41	5%	252.51	133%	61.41	-91%
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<TC4>	45.98	-120%	60.36	1%	251.38	119%	60.36	-98%
<TC5>	43.76	-131%	56.55	-12%	247.34	85%	56.55	-123%
Delta	2.32		6.72		7.18		6.72	

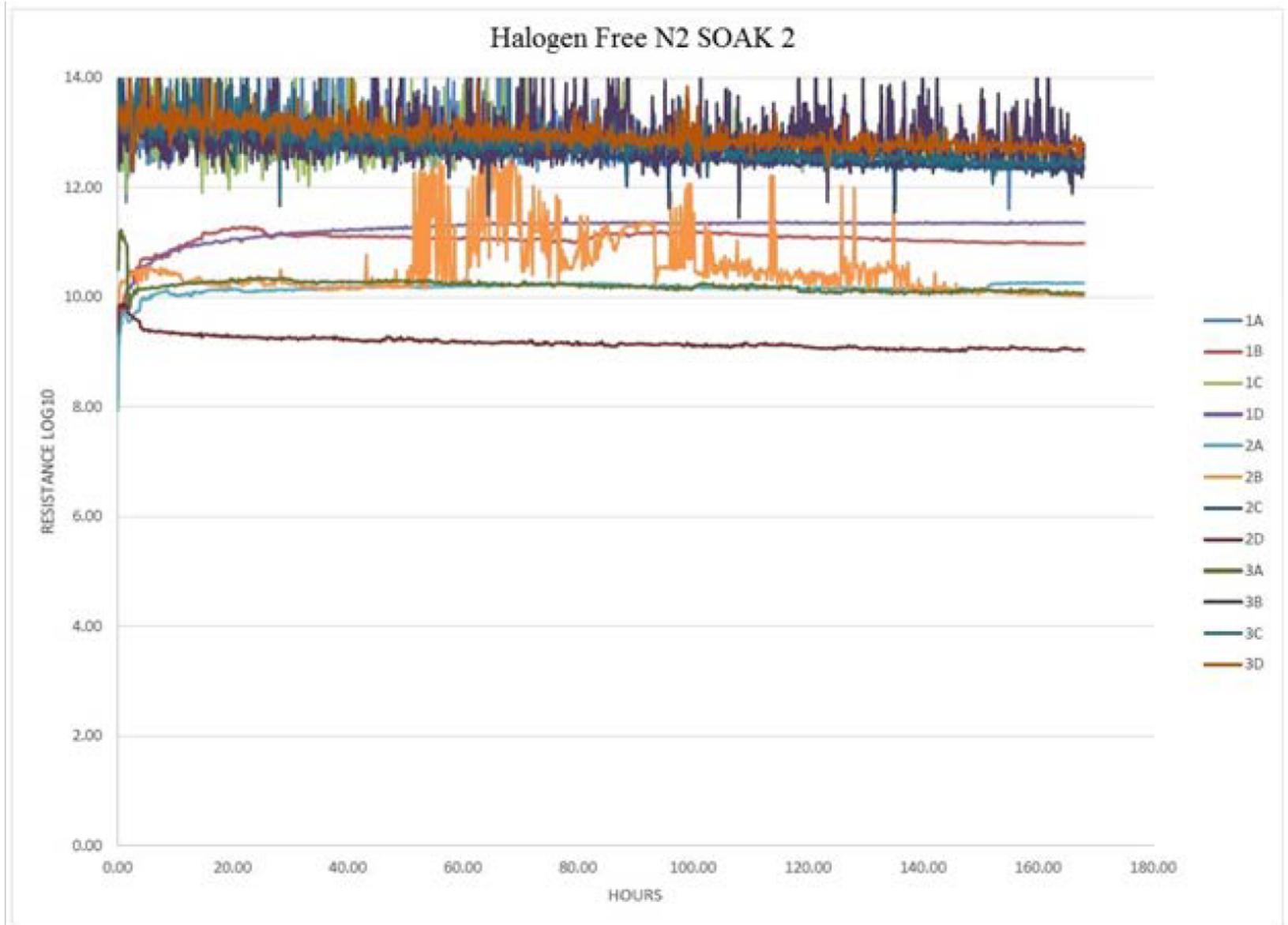
SOAK

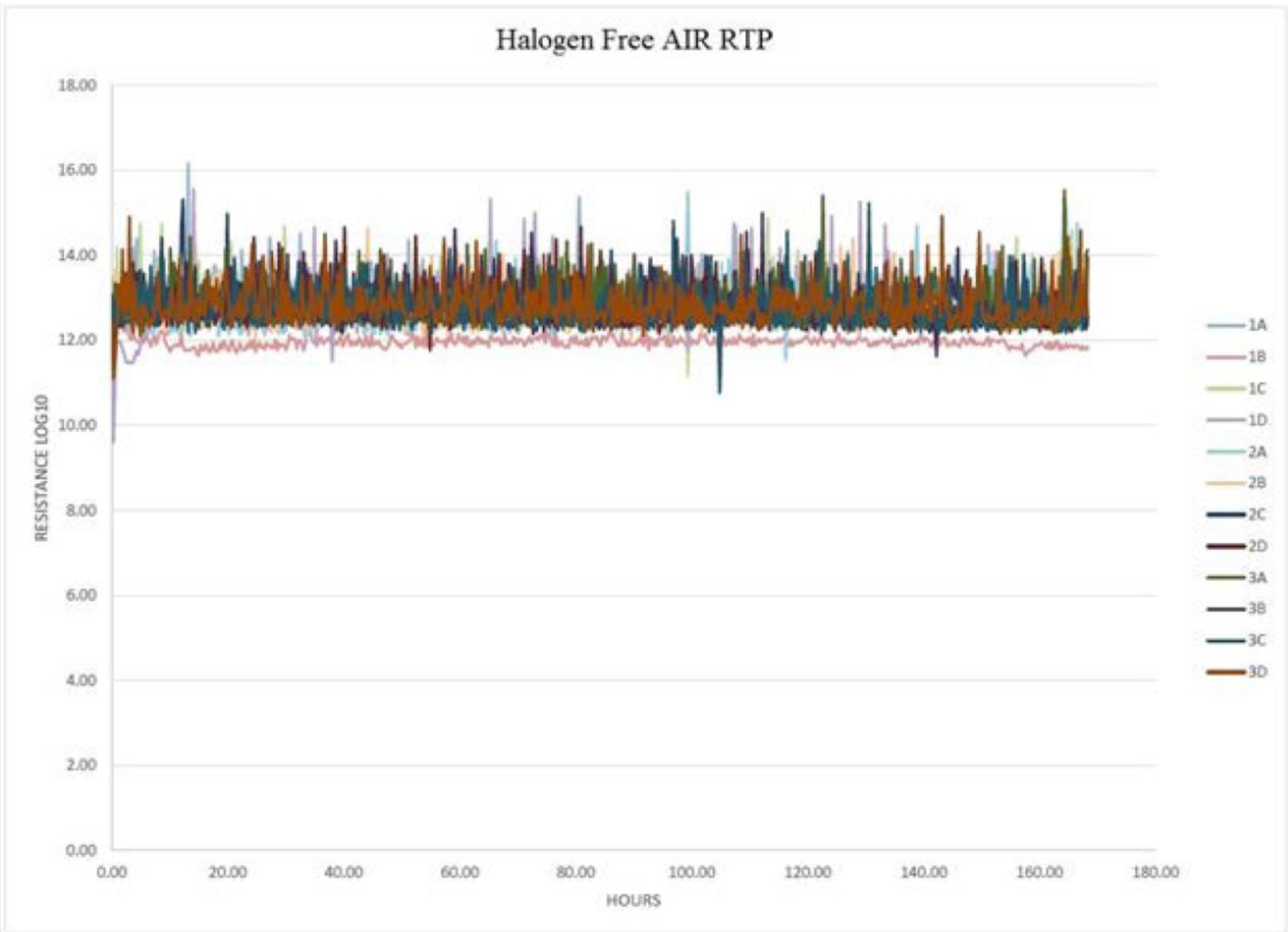


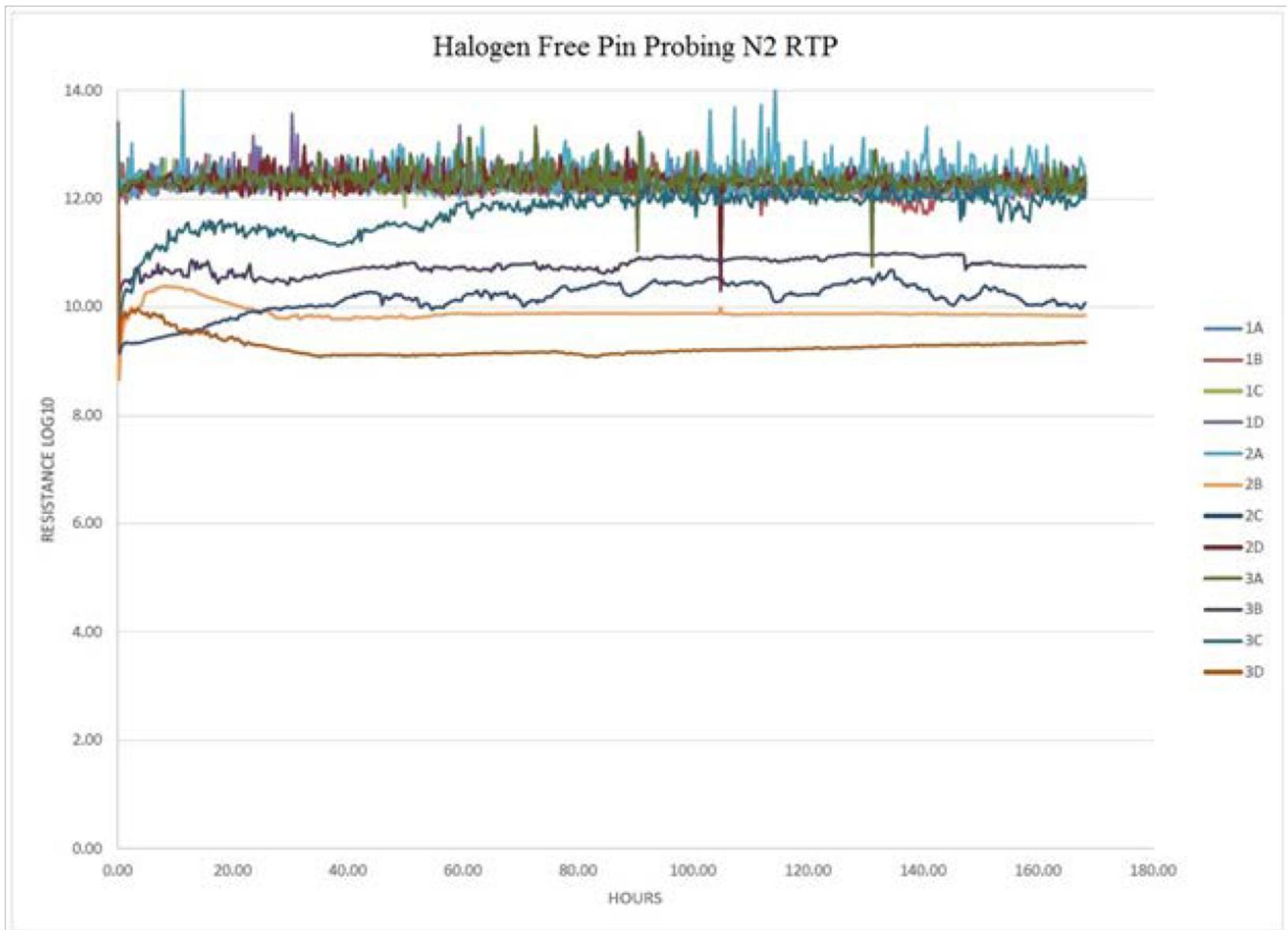
	Soak Time 160-200C		Reflow Time /220C		Peak Temp		Tot Time /220C	
<TC2>	76.06	30%	77.39	58%	252.17	129%	77.39	16%
<TC3>	75.83	29%	77.72	59%	253.06	141%	77.72	18%
<TC4>	75.59	28%	73.01	43%	247.50	67%	73.01	-13%
<TC5>	76.65	33%	73.70	46%	250.40	103%	73.70	-9%
Delta	1.06		4.71		5.56		4.71	

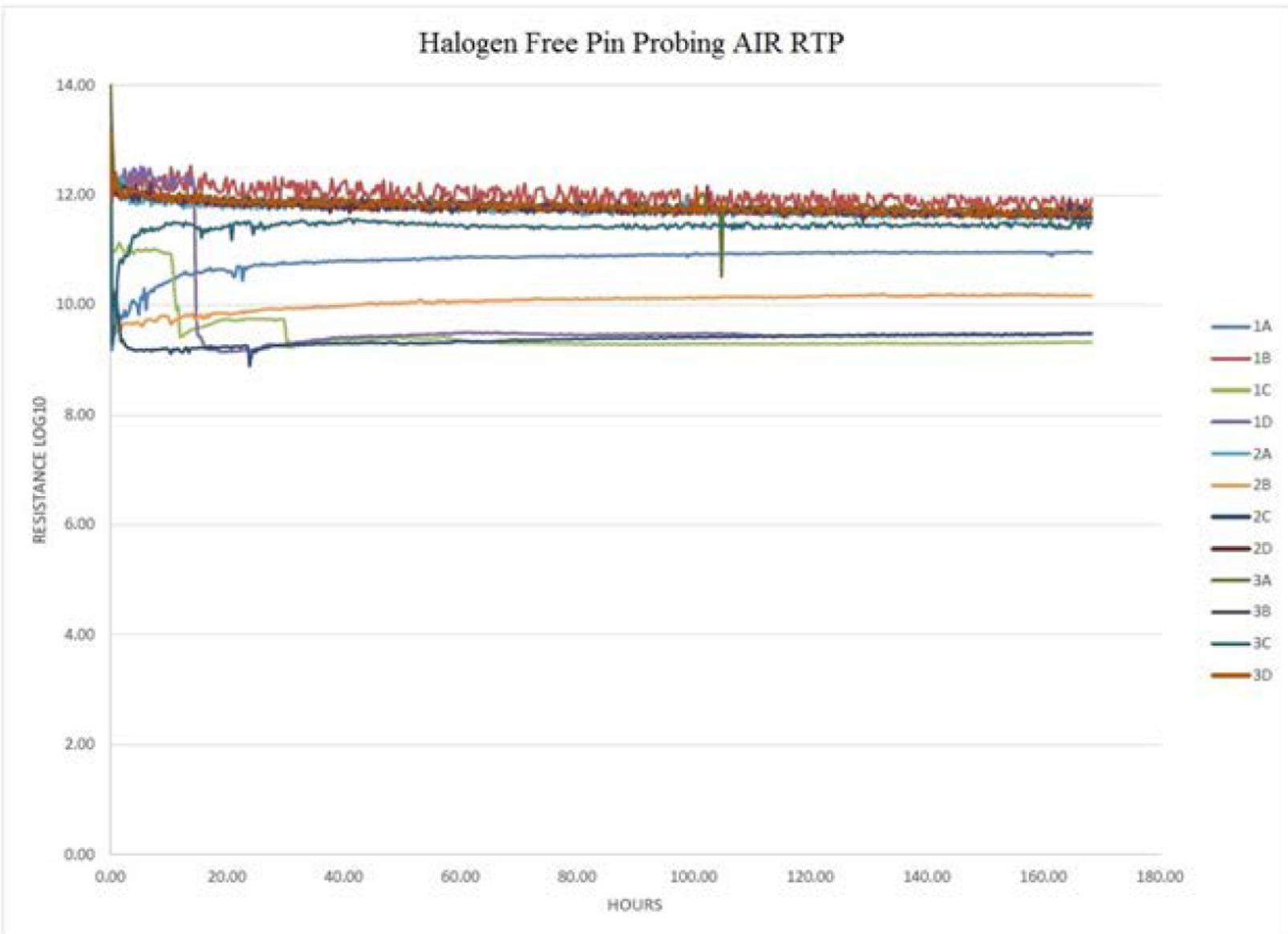
REFLOW PROFILE SUMMARY

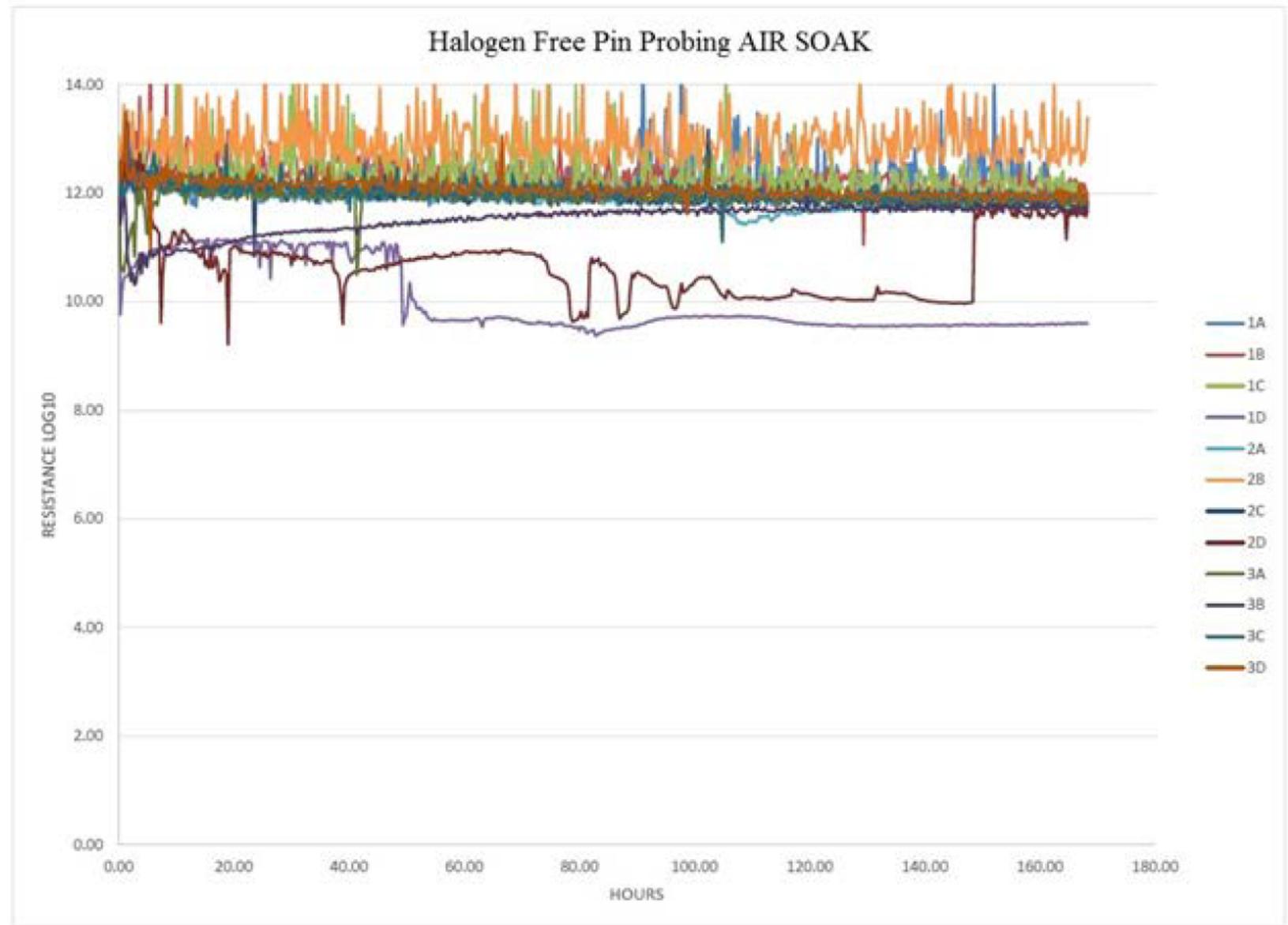
Profile Name	Avg. Soak Time (160C - 200C)	Avg. Reflow Time (220C)	Avg. Peak Temp.
Ramp to Peak	45.27 seconds	60.4 seconds	251.44 C
Soak	76.03 seconds	75.46 seconds	250.78 C

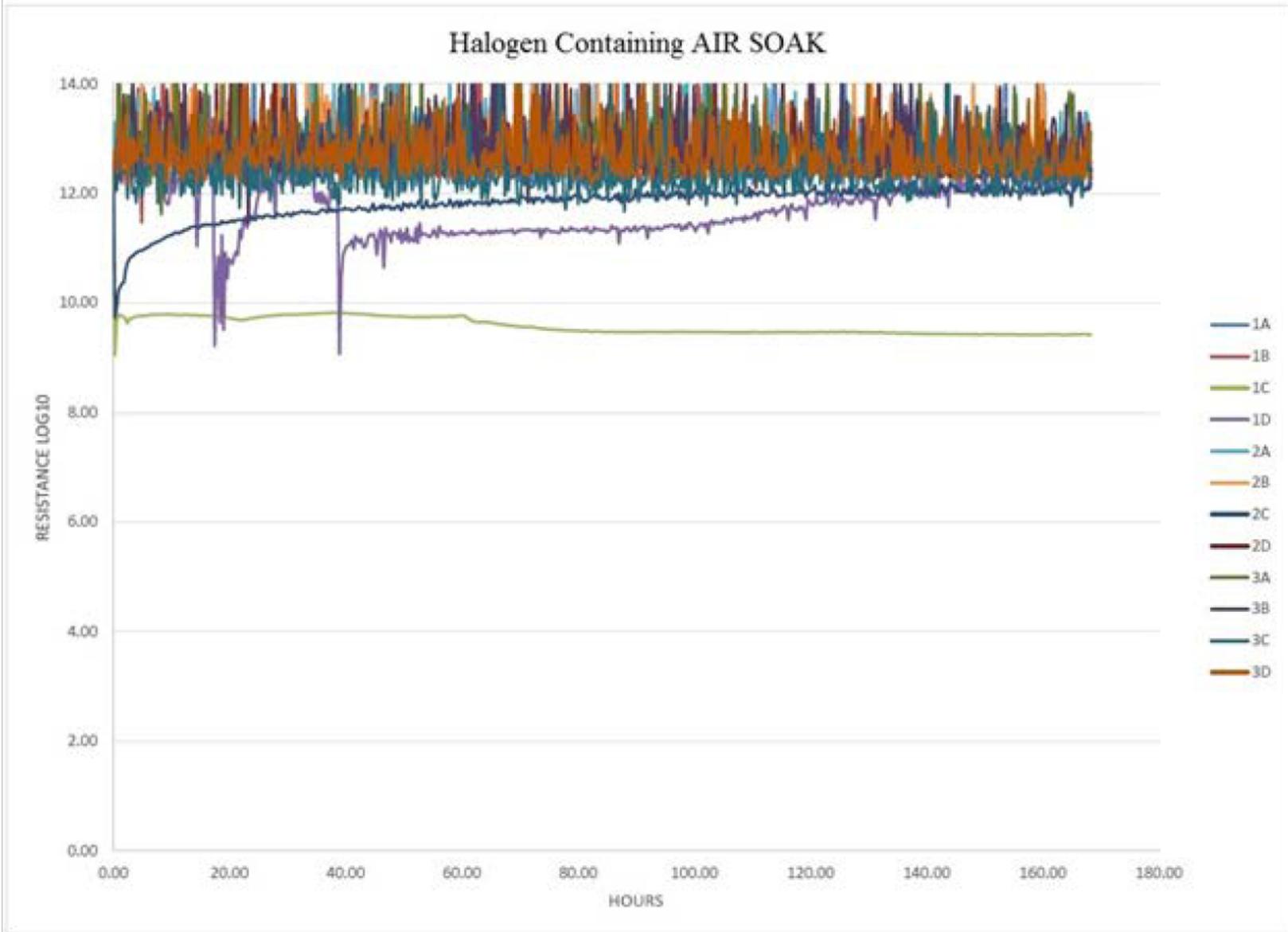




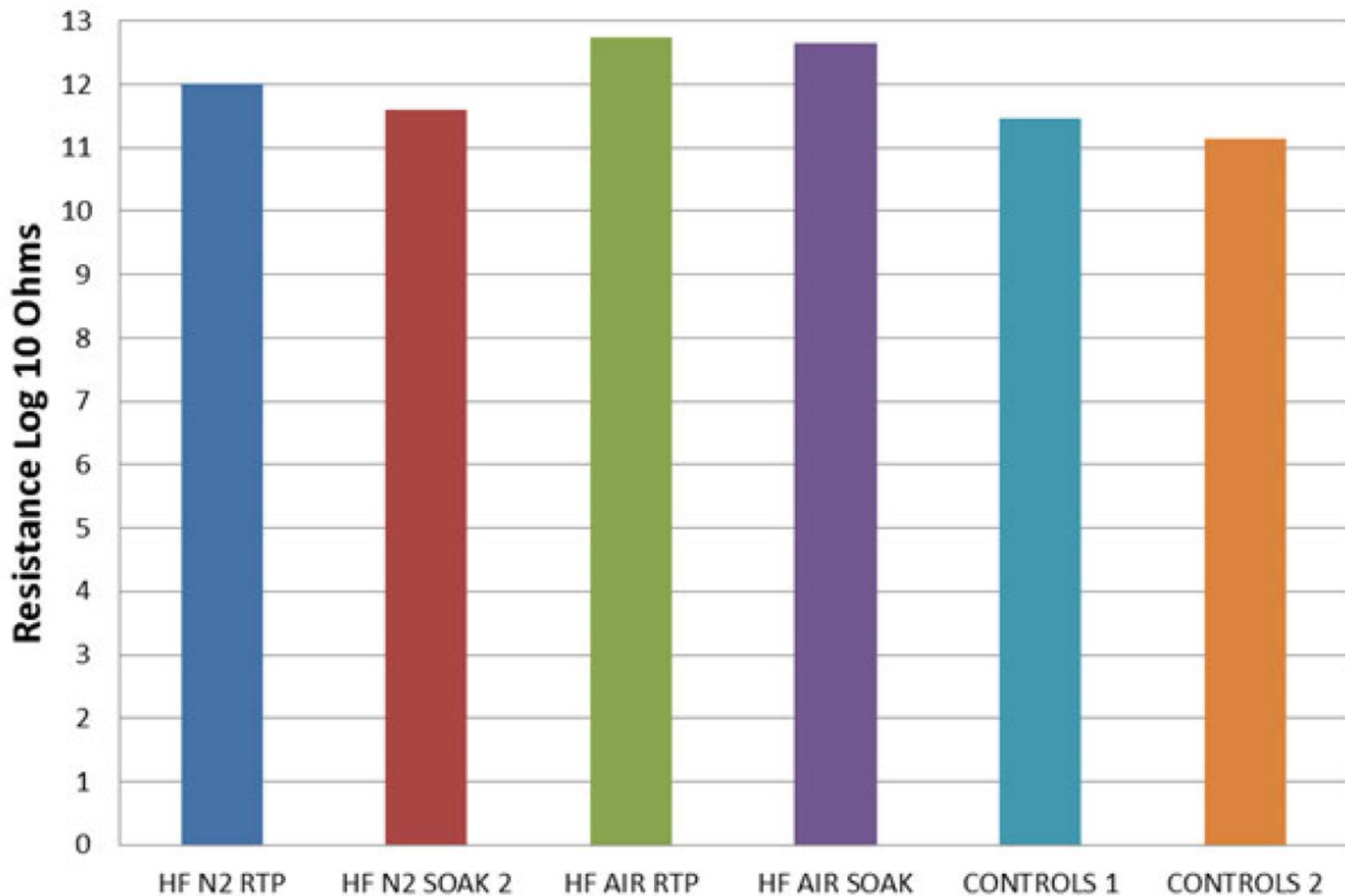




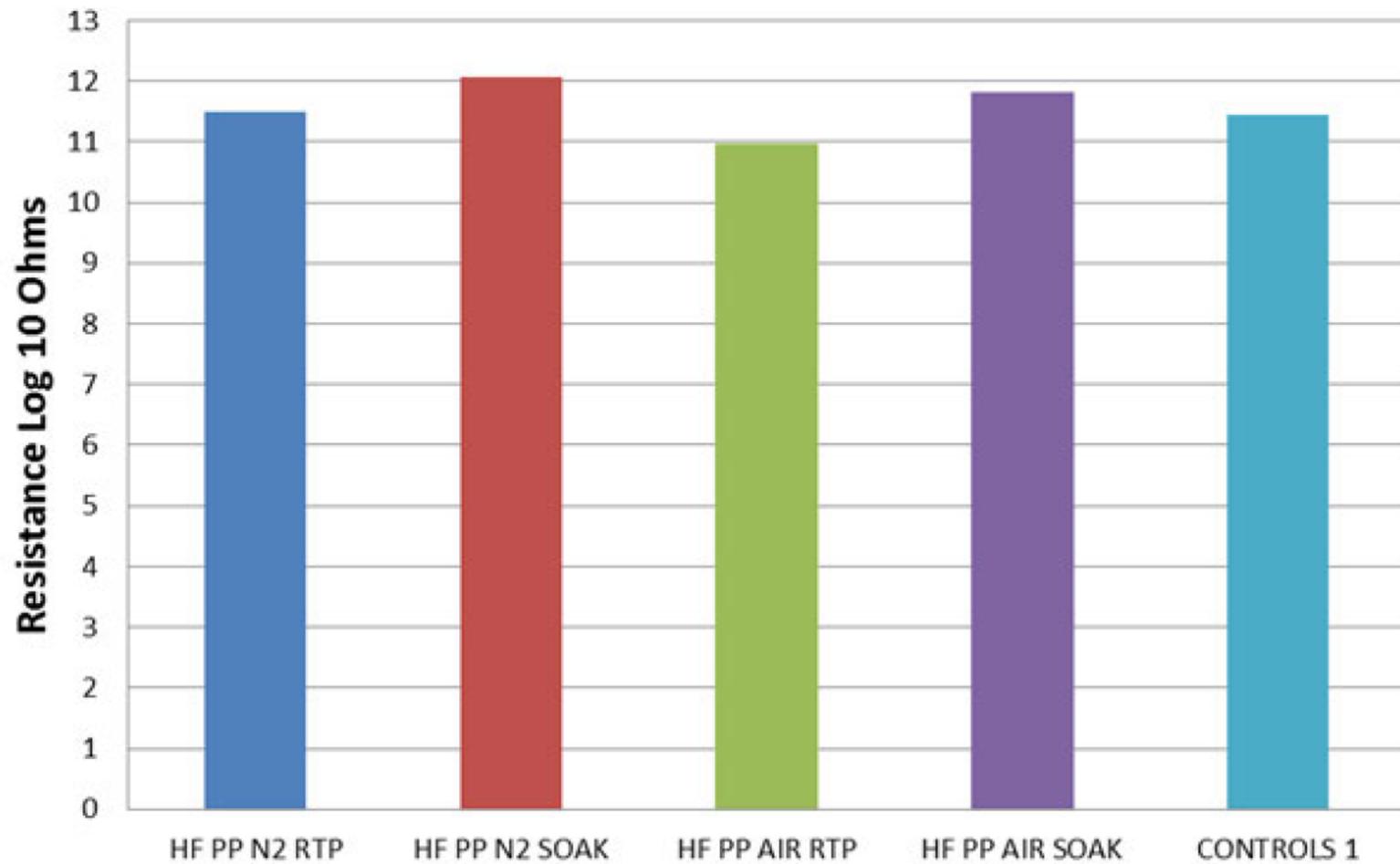




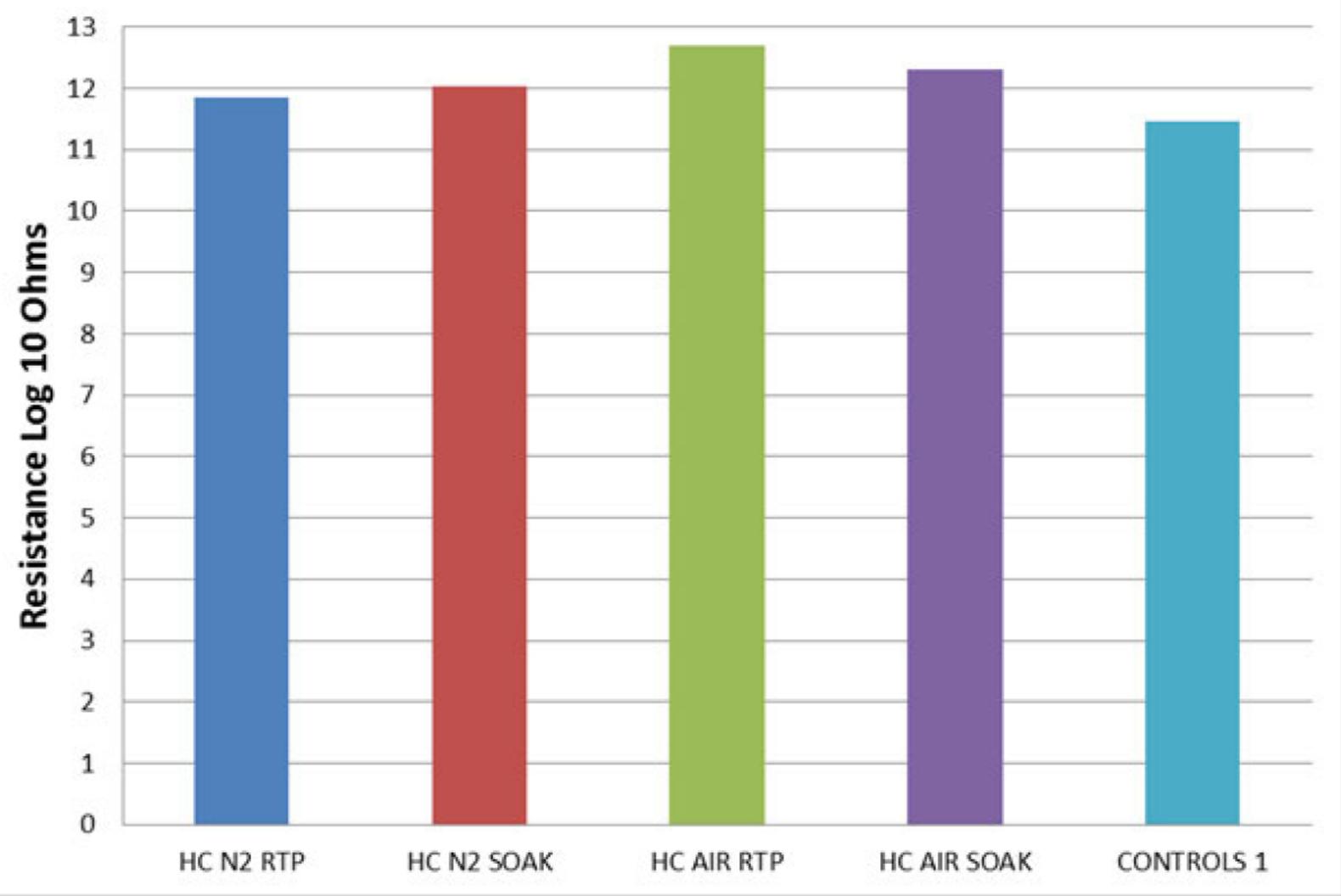
AVERAGED HALOGEN FREE SIR VALUES



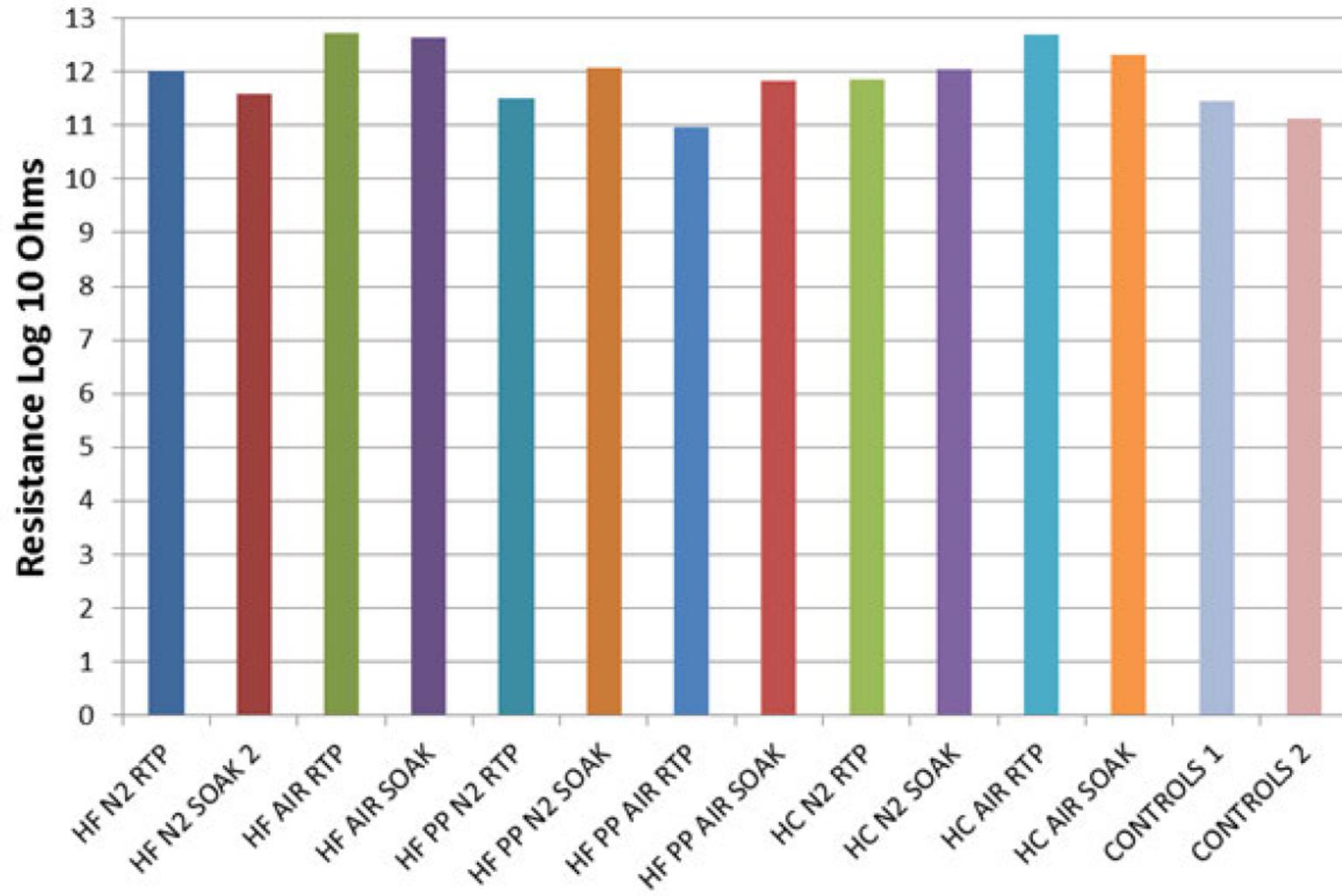
AVERAGED HALOGEN FREE PIN PROBING SIR VALUES



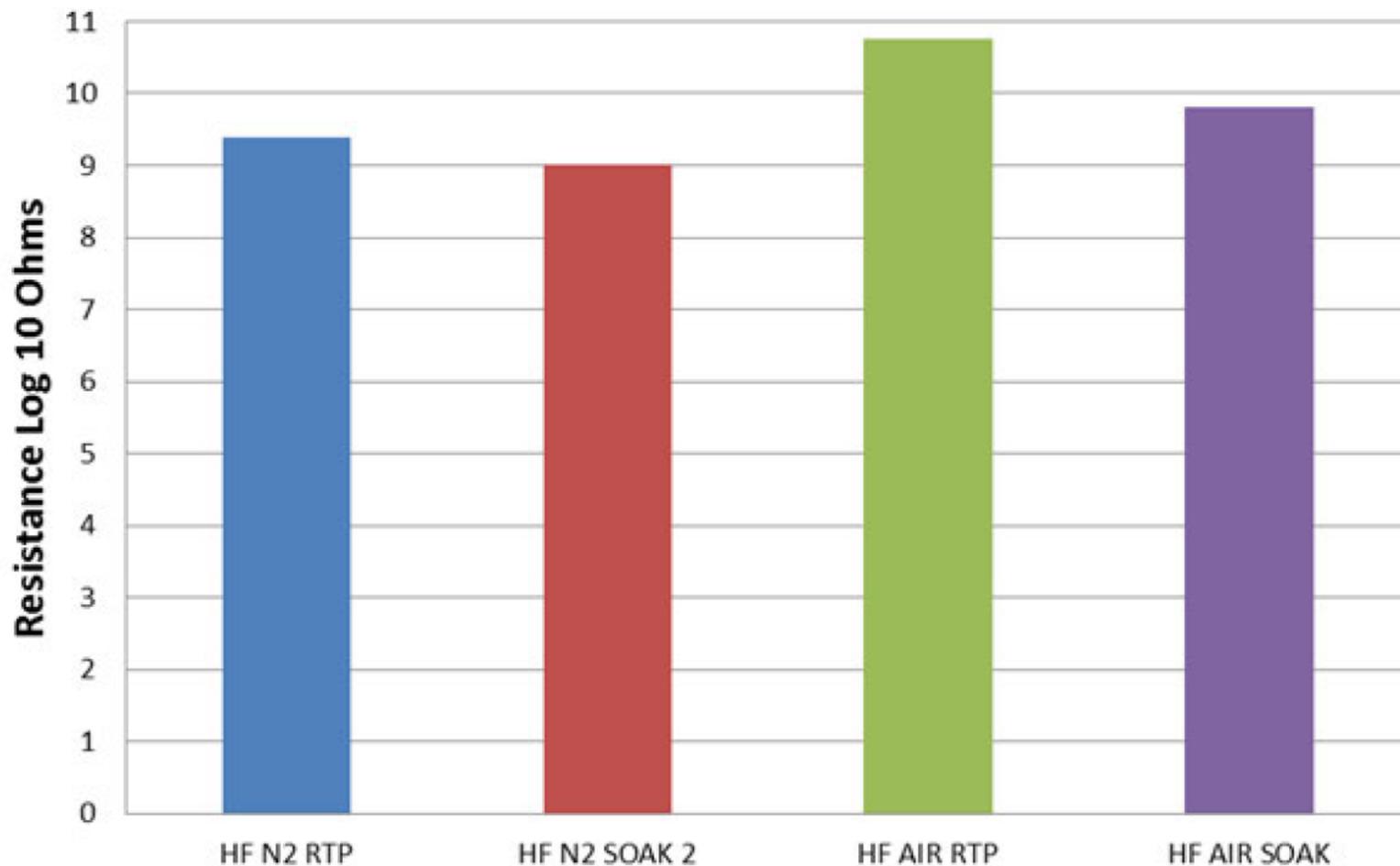
AVERAGED HALOGEN CONTAINING SIR VALUES



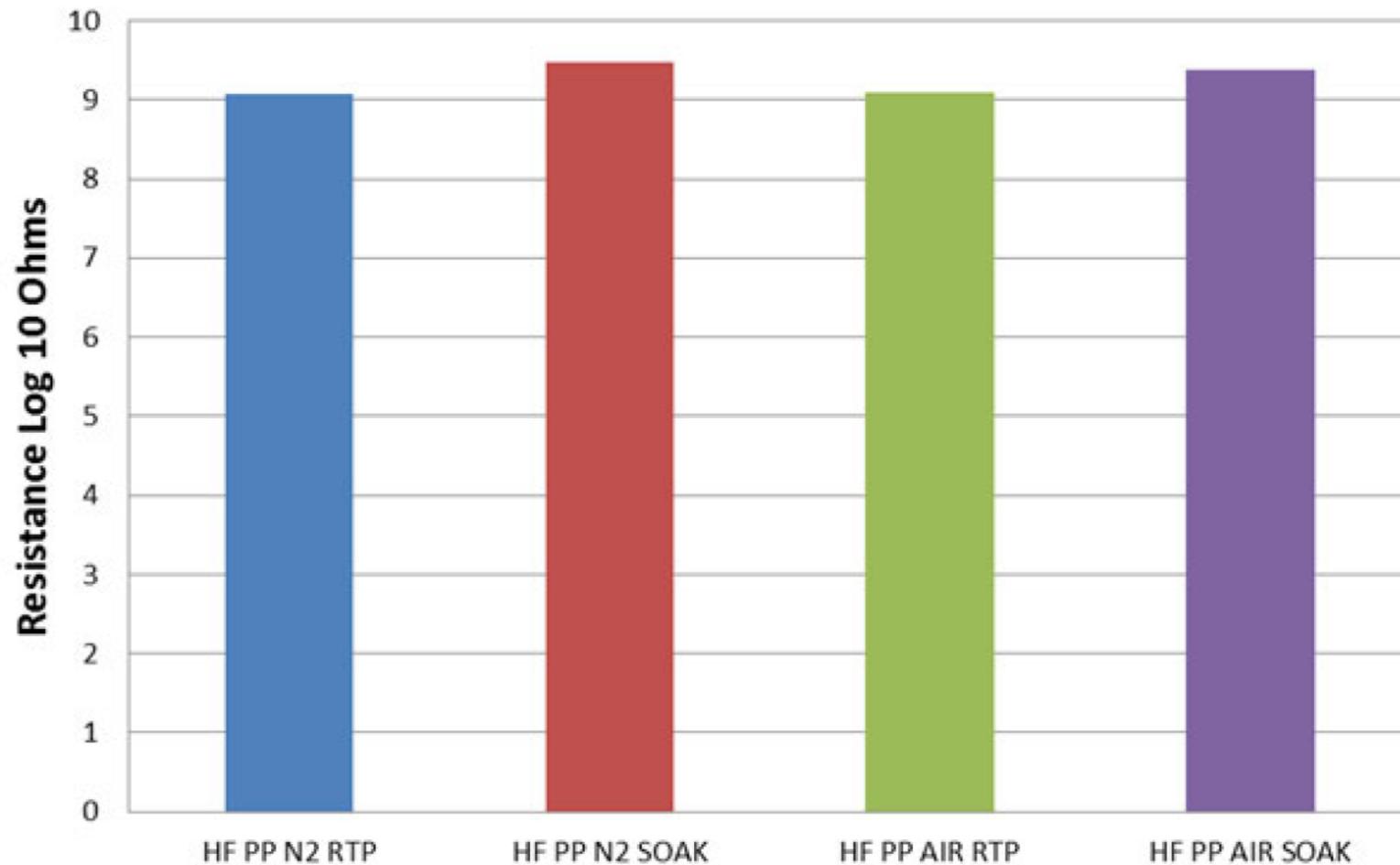
ALL AVERAGED SIR VALUES



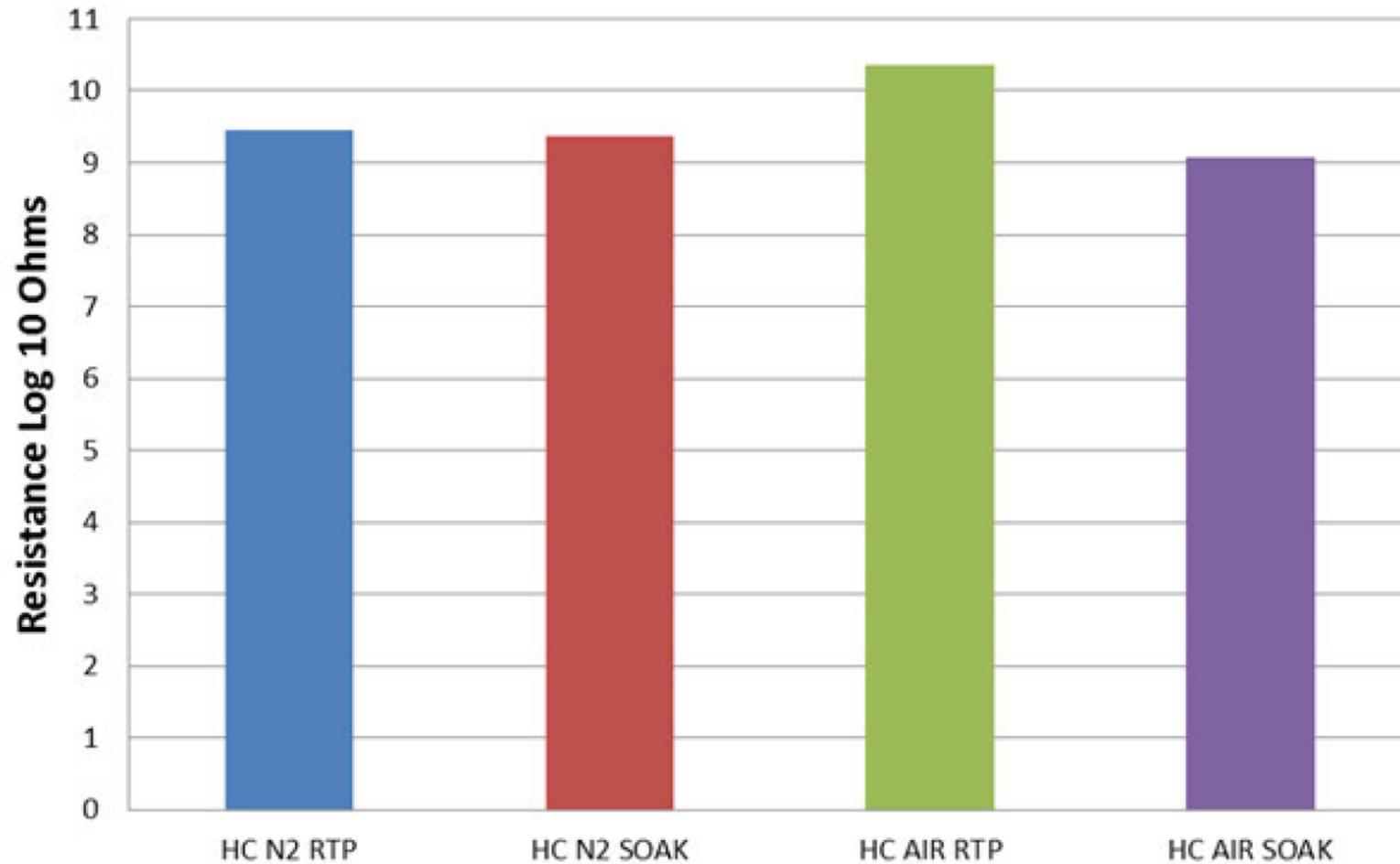
HALOGEN FREE LOWEST SIR VALUE BEYOND 24 HOURS



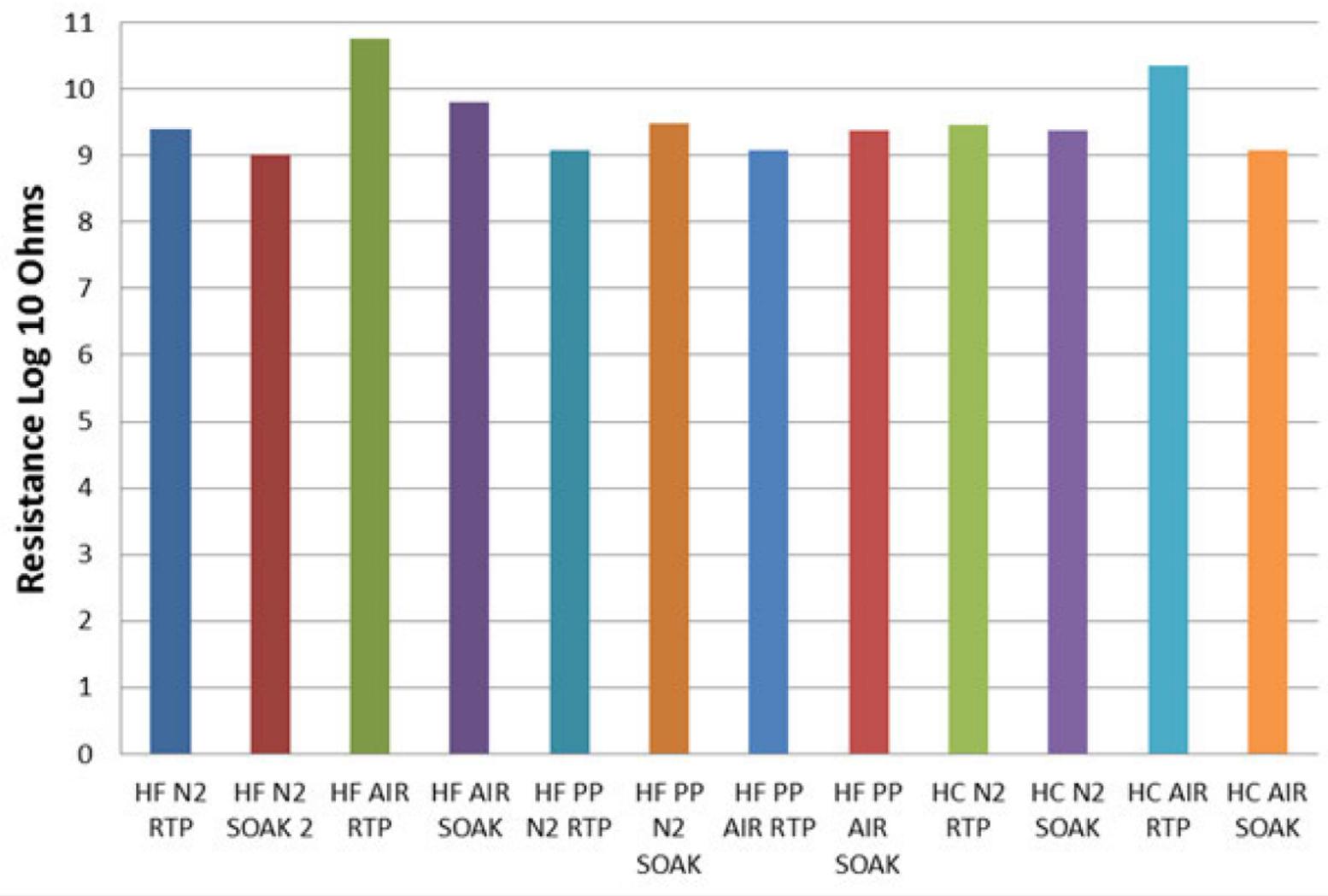
HALOGEN FREE PIN PROBING LOWEST SIR VALUE BEYOND 24 HOURS



HALOGEN CONTAINING LOWEST SIR VALUE BEYOND 24 HOURS



ALL LOWEST SIR VALUE BEYOND 24 HOURS



Observations and Conclusions

- No Clear Cut Meaningful Trend
- All Averaged SIR Values Comparable or Higher than Controls
- All Averaged SIR Values ≥ 3 Orders of Magnitude Higher than 100M Ω (1×10^8 Ohms)
- Lowest SIR Value After 24 Hours ~ 1 Order of Magnitude Higher than 100M Ω (1×10^8 Ohms)
- “Differences” Too Small
- Modern No-Clean Solder Pastes Immune to Reflow Environment
 - Activator Selection
 - Rosin Encapsulation