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Can Age and Storage Conditions Affect the SIR Performance of a No-Clean Solder Paste Flux Residue?

Eric Bastow
Indium Corporation
Clinton, New York

The SMT assembly world, especially within the commercial electronics realm, is dominated by no-clean solder paste technology. A solder paste flux residue that does not require removal is very attractive in a competitive world where every penny of assembly cost counts. One important aspect of the reliability of assembled devices is the nature of the no-clean solder paste flux residue. Most people in this field understand the importance of having a process that renders the solder paste flux residue as benign and inert as possible, thereby ensuring electrical reliability. But, of all the factors that play into the electrical reliability of the solder paste flux residue, is there any impact made by the age of the solder paste and how it was stored? This paper uses J-STD-004B SIR (Surface Insulation Resistance) testing to examine this question. Two commercially available SAC305, Type 4, no-clean solder pastes—one with a ROL0 and the other with a ROL1 J-STD-004B classification—were subjected to two different storage conditions (room temperature and refrigeration) and aged for varying lengths of time. After aging, the solder pastes were printed and reflowed using the same common reflow profile and then submitted to SIR testing to see what, if any, difference could be detected in their SIR performances. The reason for testing both a ROL0 and a ROL1 was to see if differences in chemistry could have an impact on how a solder paste ages relative to SIR performance.

The flux ingredients responsible for removing oxidation from the PCB pad, component leads and terminations, and the solder powder in the paste are commonly referred to as “activators.” Activators are typically weak organic acids or halogenated compounds. Because solder paste is a blend of solder particles/powder and flux, the flux is in direct contact with the solder powder. This gives the activators access to the solder powder from the moment that the paste is blended until the time that it is reflowed, at which point most of the activators will be consumed or encapsulated in the residue. This means that while the solder paste is stored in the jar, cartridge, or syringe, the flux and, therefore, the activators are in contact with the solder particles. Normally, activators require heat to interact with metal oxides, but some interaction can and does take place at lower temperatures. This is why it is almost always required that solder paste be refrigerated for long-term storage. Nevertheless, solder paste can change with time. One tangible manifestation of a solder paste that has undergone this sort of flux/solder powder interaction is a rise in viscosity (Figure 1). Other changes may be too subtle to detect without performing attribute-specific tests.

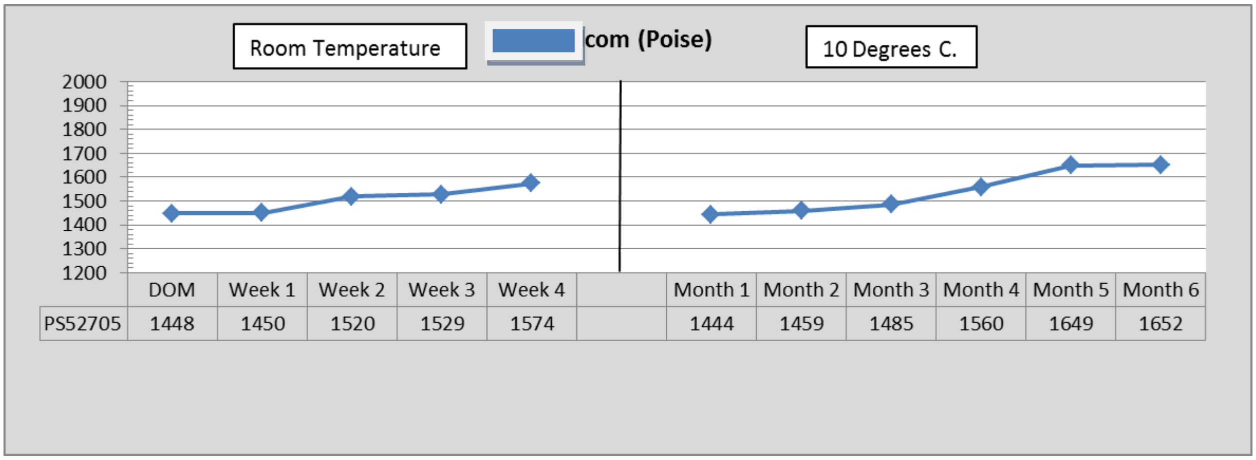


Figure 1: Viscosity vs. Time at Two Different Storage Conditions

Table 1 shows the test matrix that was used for this experiment. It should be noted that the age shown in Table 1 is defined as the time that elapsed from the date of manufacture (DOM) to the time that it was printed and reflowed onto the SIR test boards. The jars of paste were left sealed (never opened) until the time of use. The room temperature was typically between 71°F and 74°F (21.7°C – 23.3°C) and the refrigerator was set to 4°C (39°F). Both solder pastes have a 6-month shelf life when stored in refrigeration as specified by the paste manufacturer.

Table 1: Test Matrix

Solder Paste Type	Storage Condition	Age (Days)	Board Count
Halogen-Free	Room Temp	91	2
		61	2
		19	2
	Refrigerated	91	2
		61	2
		19	2
Halogen-Containing	Room Temp	91	2
		61	2
		19	2
	Refrigerated	91	2
		61	2
		19	2
Controls	N/A	N/A	2
		Total	26

On the day that the paste was opened and used, there were some obvious and varying degrees of flux separation. The greatest amount of flux separation was observed among the older pastes that were stored at room temperature. Figure 2 shows the appearance of the opened jar of the halogen-free paste that was stored at room temperature for 91 days. Figure 3 shows the appearance of the opened jar of the halogen-containing paste that was stored at room temperature for 91 days. Pools of dark (black) material were observed on the surface of the halogen-containing paste (see Figure 3). All solder pastes were gently hand-stirred with a plastic spatula to restore homogeneity prior to use. (The refrigerated solder paste jars were removed from the refrigerator ~2 hours prior to opening to allow the paste to warm up to room temperature before use.)

Figure 2: Flux Separation - Halogen-Free Paste Stored @ RT for 91 Days

Figure 3: Flux Separation - Halogen-Containing Paste Stored @ RT for 91 Days

The solder pastes were stencil printed onto standard IPC-B-24 SIR test boards using a 150µ (~.006”) thick stencil, as prescribed by IPC-TM-650 2.6.3.3. Each board had four SIR patterns, “A” through “D”. Each SIR pattern or comb pattern was comprised of 0.4mm bare copper traces with 0.5mm spacing between each trace. Figure 4 shows an image of an IPC-B-24 SIR board.

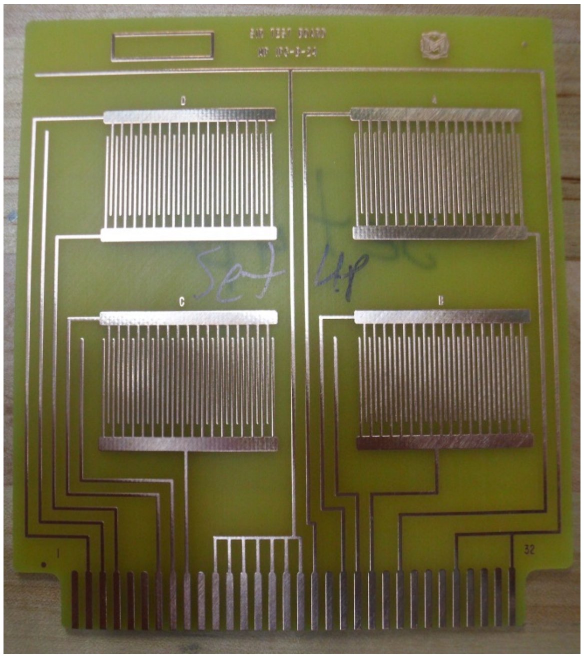
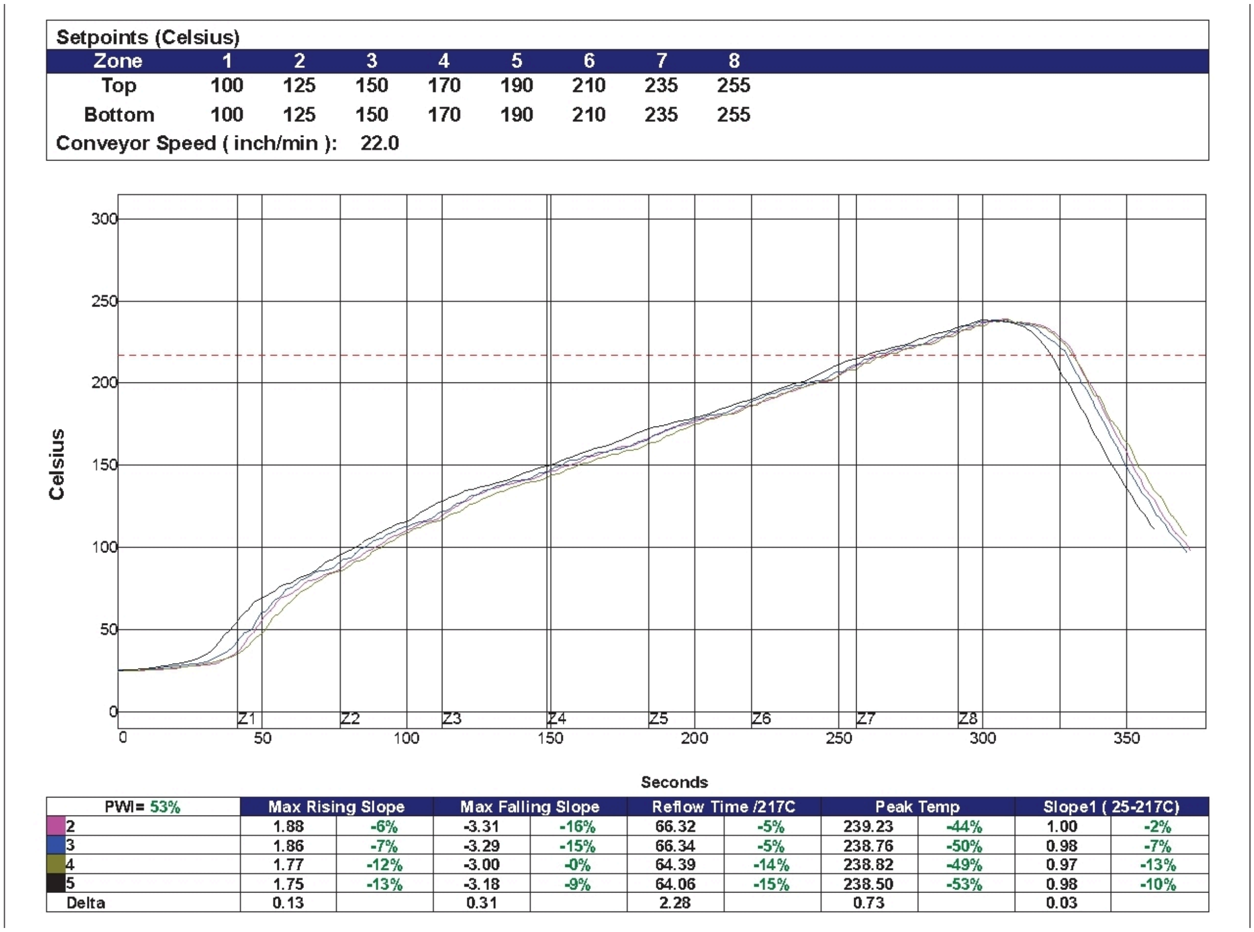


Figure 4: IPC-B-24 SIR Board

After the solder pastes were printed onto the SIR boards, they were reflowed using the profile shown in Figure 5. The profile is a linear “ramp to peak” with a peak temperature of ~239°C and a Time Above Liquidus (TAL) of ~65 seconds. It was intended to have a peak temperature of ~245°C, but because the profiler that was used to create the profile was out of calibration, the end result was a peak temperature of ~239°C. This discrepancy was discovered when the profile was later verified with a calibrated profiler. The unintended cooler peak temperature may actually have been a benefit for this study as it could have accentuated any differences in the SIR performance of the various solder pastes. This is because a cooler profile may not produce the same level of flux activation as a hotter profile.



Setpoints (Celsius)								
Zone	1	2	3	4	5	6	7	8
Top	100	125	150	170	190	210	235	255
Bottom	100	125	150	170	190	210	235	255
Conveyor Speed (inch/min):	22.0							

PWI= 53%	Max Rising Slope		Max Falling Slope		Reflow Time /217C		Peak Temp		Slope1 (25-217C)	
🟪 2	1.88	-6%	-3.31	-16%	66.32	-5%	239.23	-44%	1.00	-2%
🟦 3	1.86	-7%	-3.29	-15%	66.34	-5%	238.76	-50%	0.98	-7%
🟩 4	1.77	-12%	-3.00	-0%	64.39	-14%	238.82	-49%	0.97	-13%
⬛ 5	1.75	-13%	-3.18	-9%	64.06	-15%	238.50	-53%	0.98	-10%
Delta	0.13		0.31		2.28		0.73		0.03	

Figure 5: Solder Paste Reflow Profile

The boards were then submitted to SIR testing per IPC-TM-650 2.6.3.7 for 168 hours (7 days, or 1 week). The conditions inside the test chamber were 40°C and 90% relative humidity (RH). A bias was applied to the SIR boards to create an electric field strength of 25V/mm between adjacent parallel traces on the SIR patterns. A resistivity (SIR) reading was taken at least once every 20 minutes on each SIR pattern. Because there were two boards for each scenario, the result was an accumulation of 4032 SIR readings per scenario. According to IPC J-STD-004B, in order to constitute a "pass," all SIR readings after 24 hours must be 1 x 10^8 ohms per square or higher over the remaining duration of the test. However, passing or failing IPC SIR is not the main focus of this study. Rather, IPC SIR is used as the method to detect and measure the differences in the SIR performance of the various solder pastes evaluated in this work.

Figures 6 through 18 show plots of the SIR results.

Figure 18 is the SIR results from the control boards. The control boards were cleaned SIR boards on which no solder paste was applied. It can be seen that at ~90 hours into the test, there was a dramatic drop in the SIR readings on one of the patterns—board 26, pattern D. Figure 19 shows a photograph of some debris that was found on this pattern. This debris was identified as the likely cause for the dramatic drop in the SIR values for this pattern.

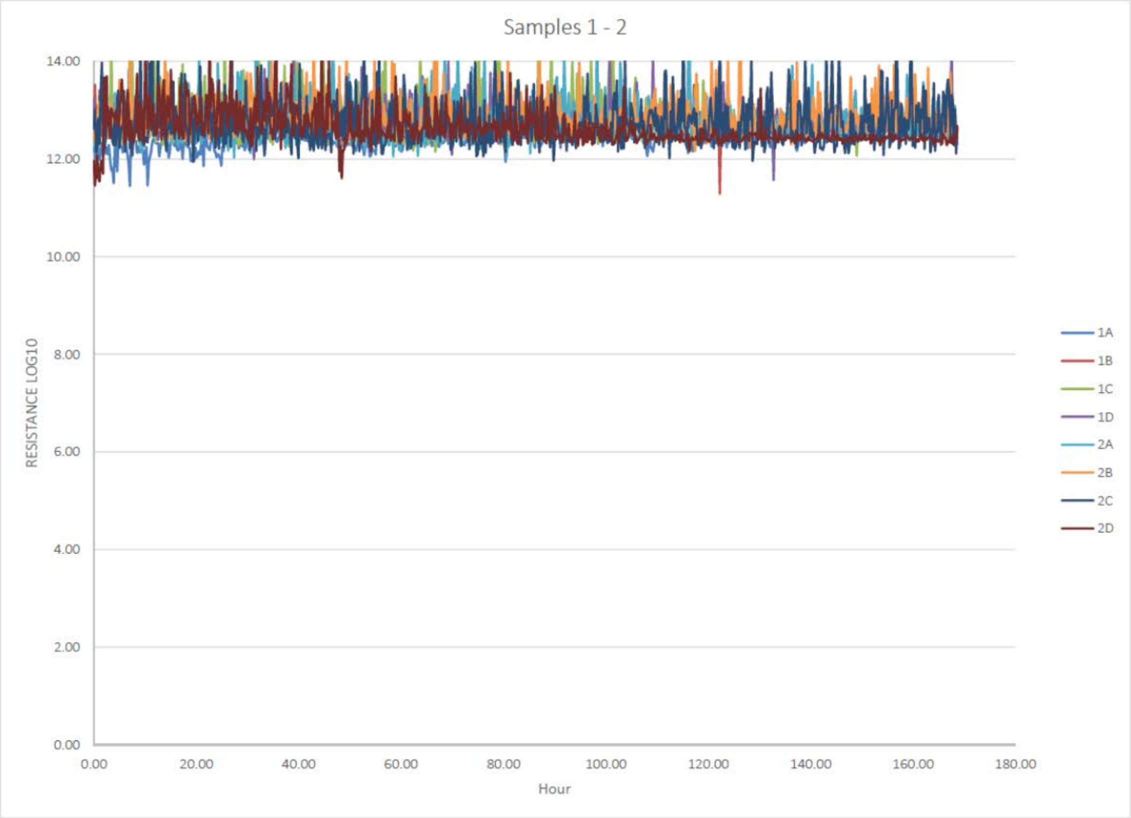


Figure 6: Halogen-Free – Room Temp – 91 Days

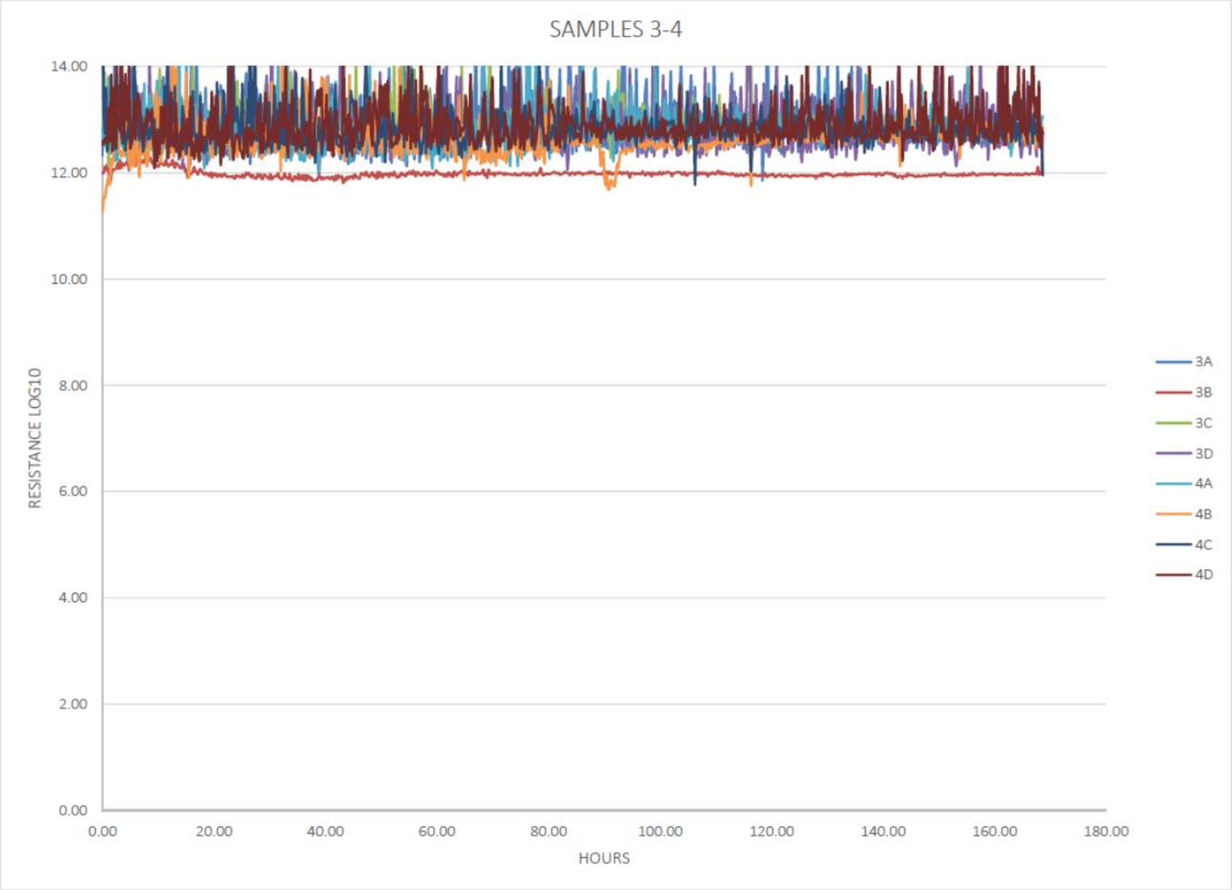


Figure 7: Halogen-Free – Room Temp – 61 Days

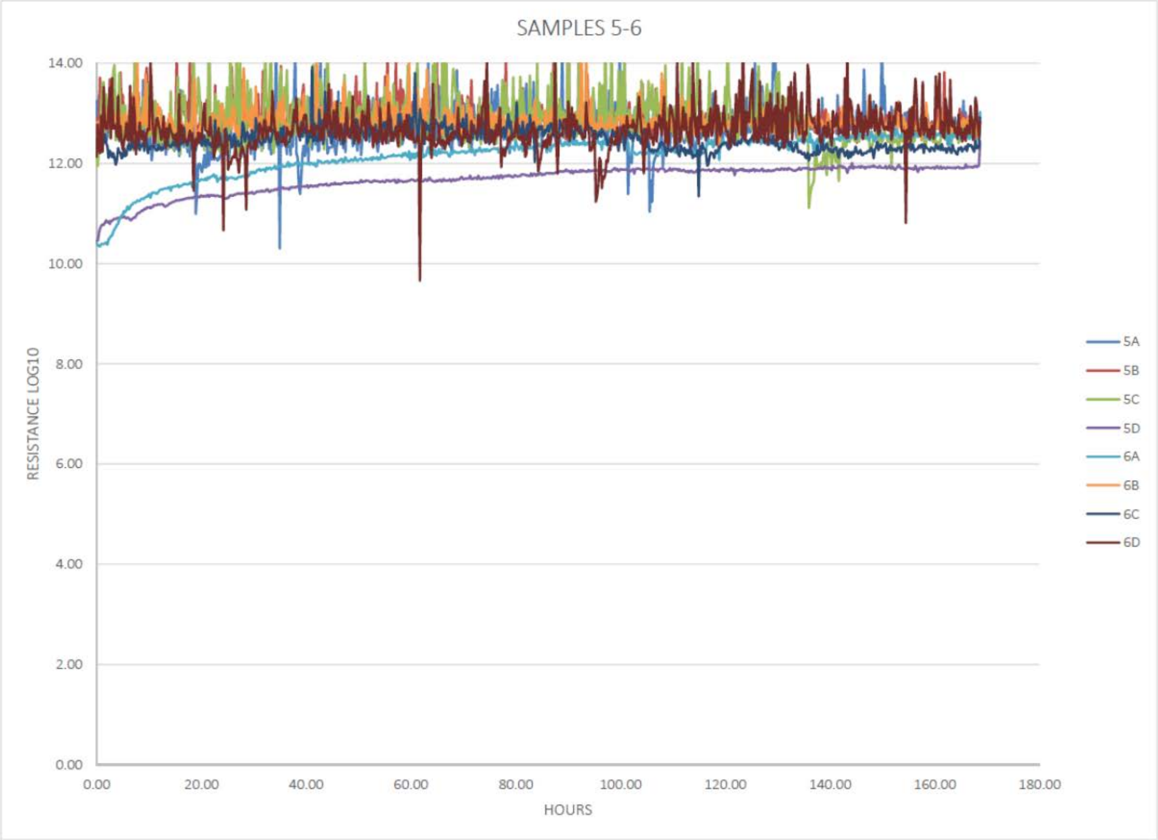


Figure 8: Halogen-Free – Room Temp – 19 Days

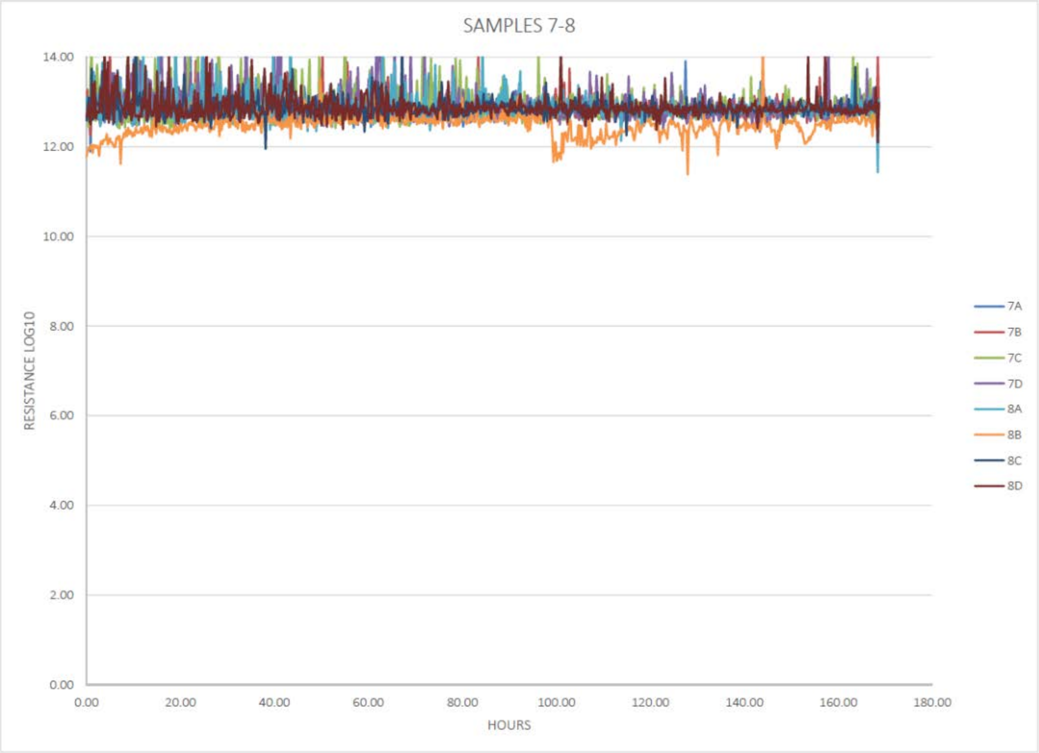


Figure 9: Halogen-Free – Refrigerated – 91 Days

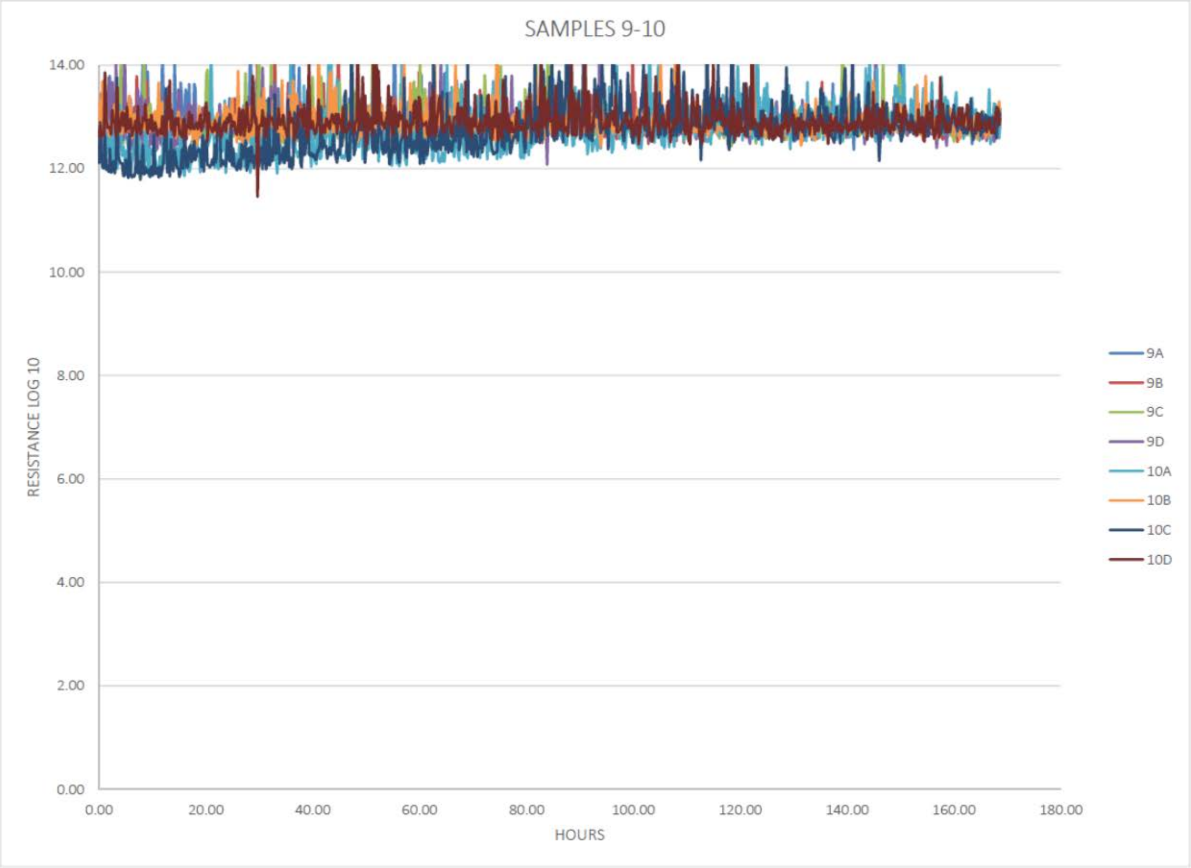


Figure 10: Halogen-Free – Refrigerated – 61 Days

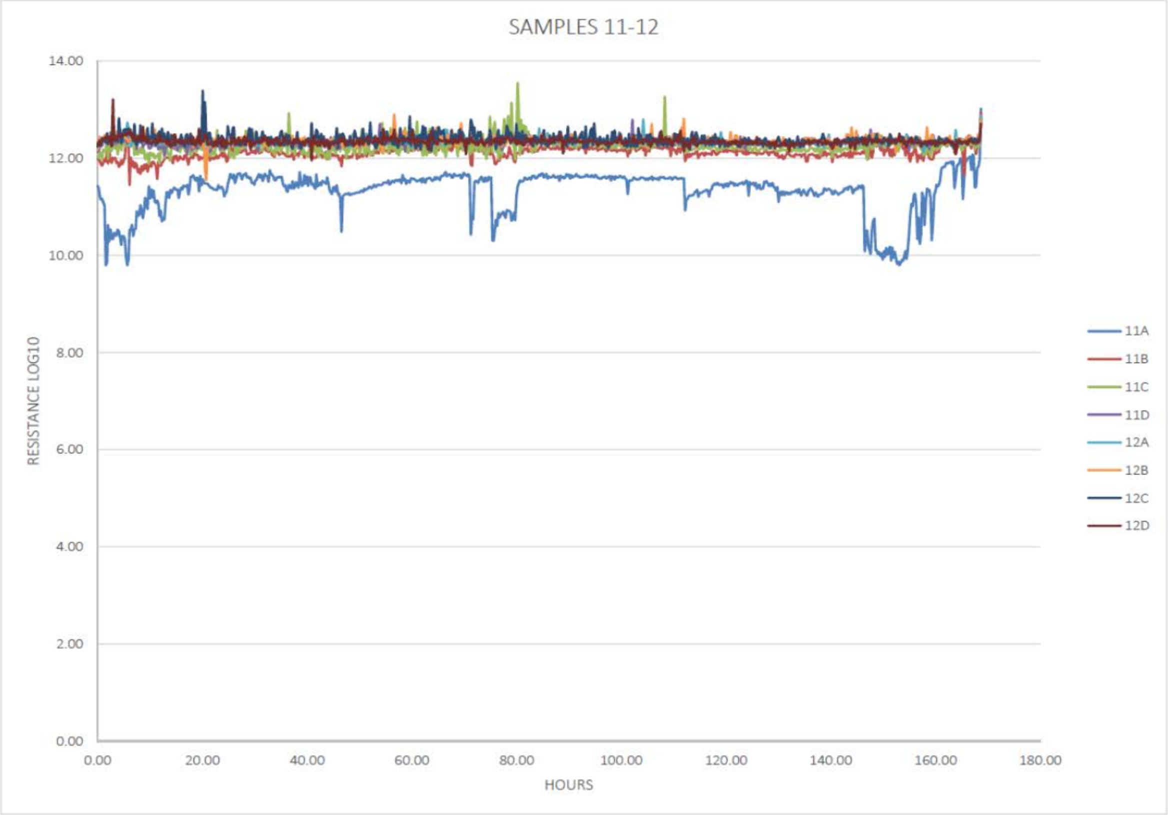


Figure 11: Halogen-Free – Refrigerated – 19 Days

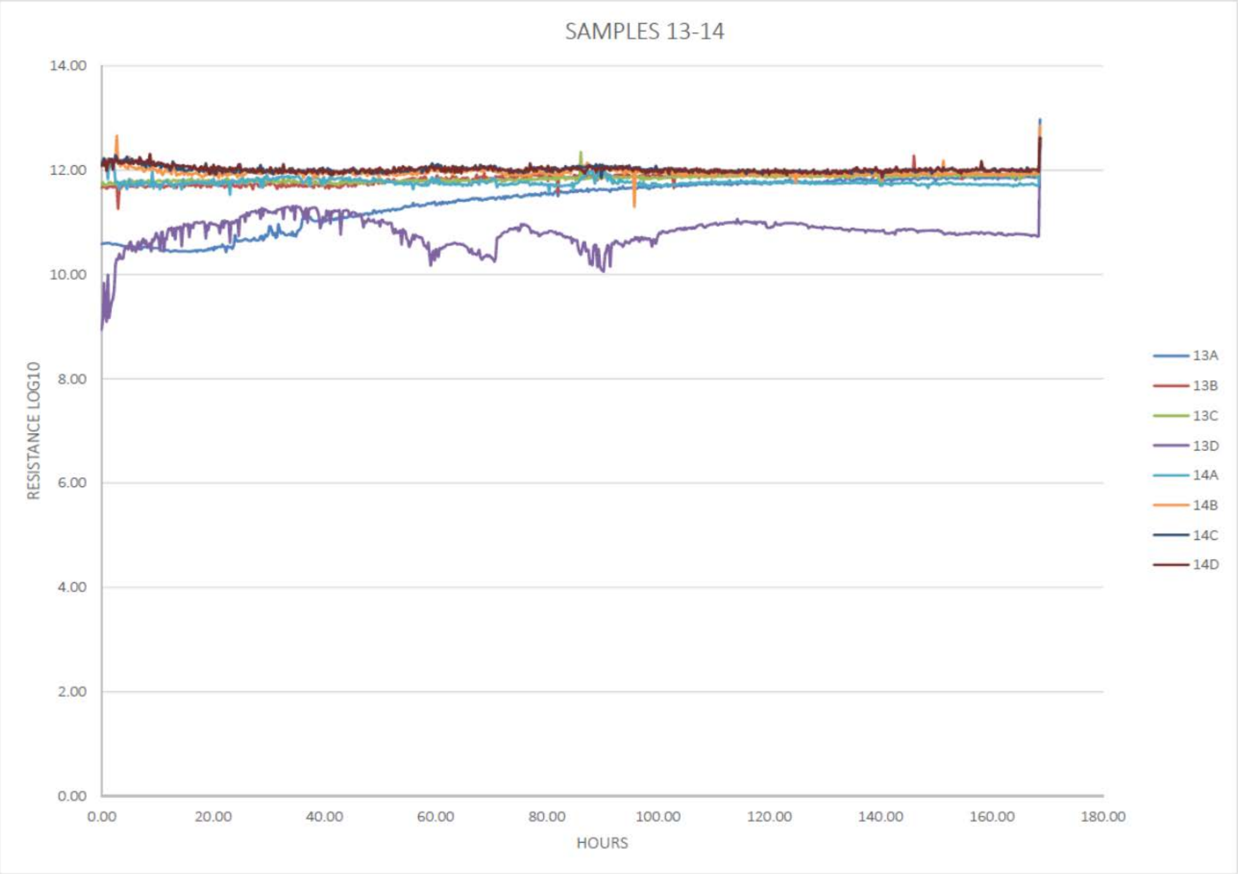


Figure 12: Halogen-Containing – Room Temp – 91 Days

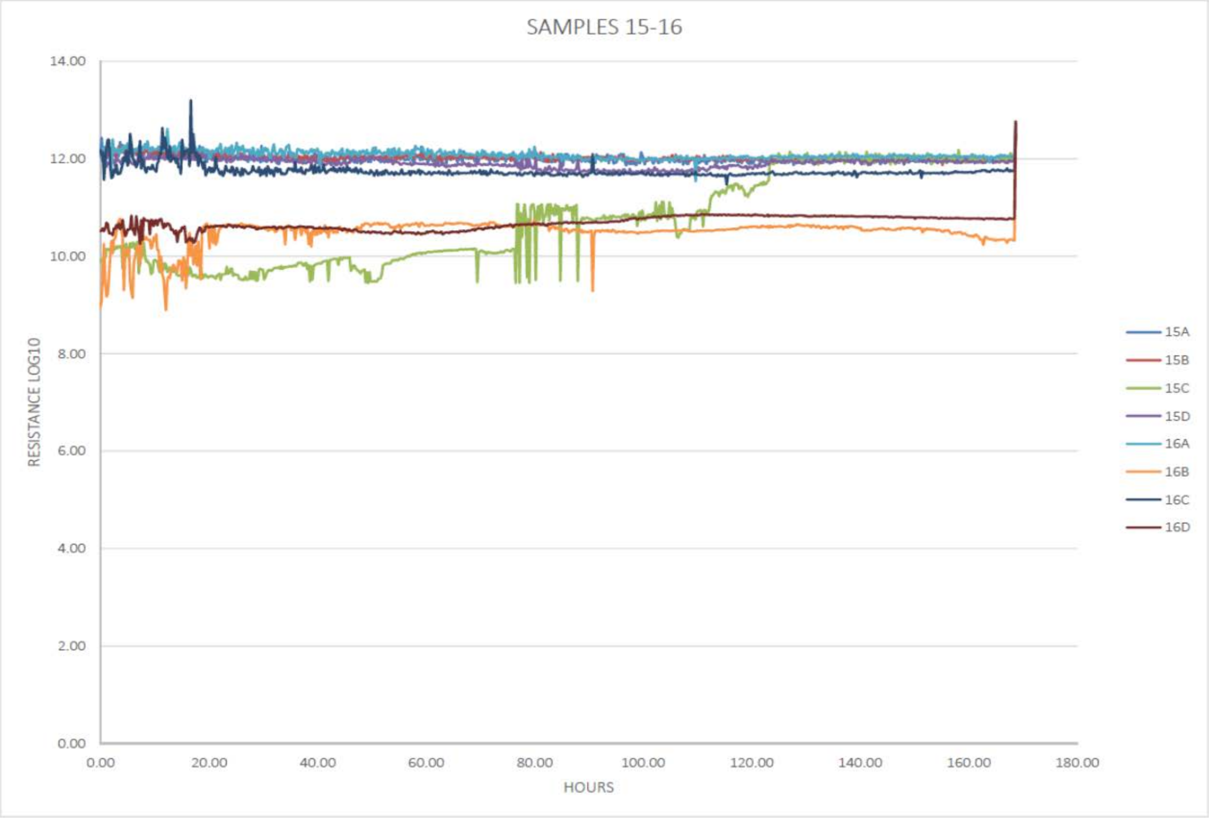


Figure 13: Halogen-Containing – Room Temp – 61 Days

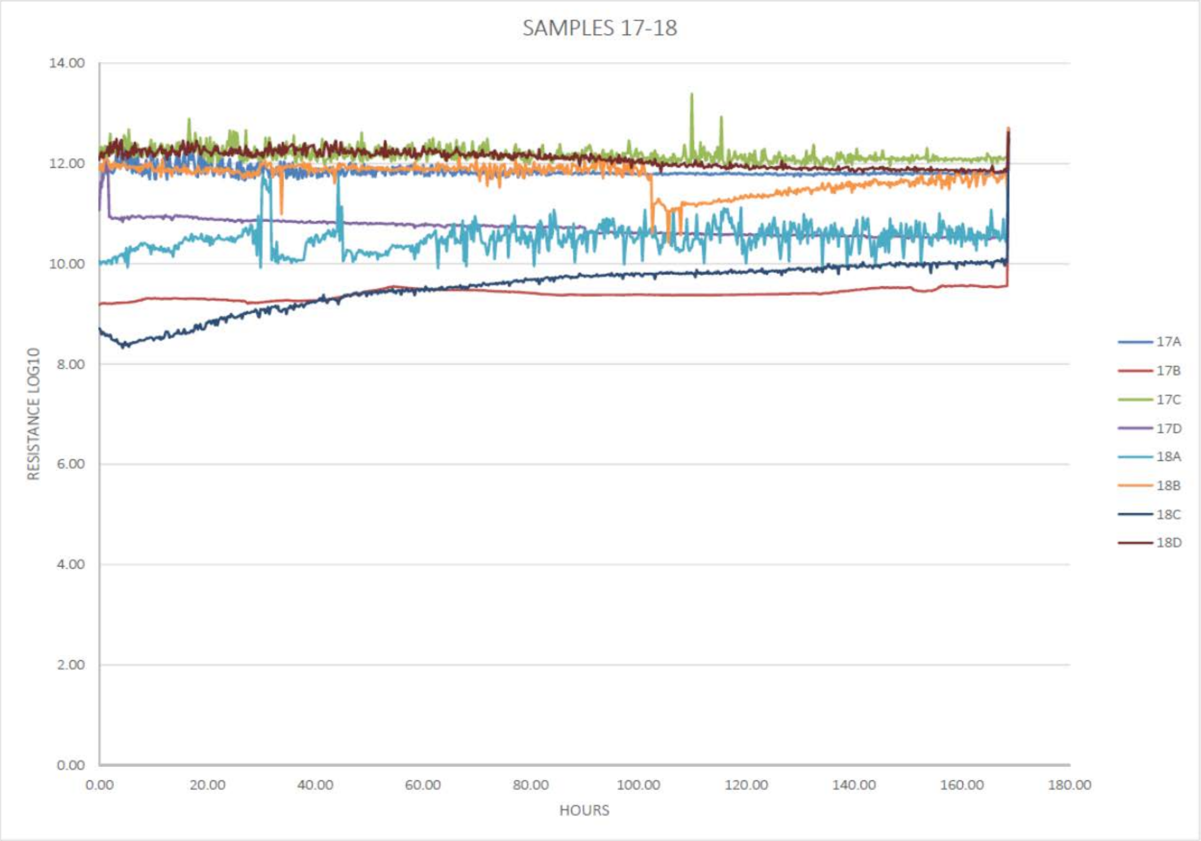


Figure 14: Halogen-Containing – Room Temp – 19 Days

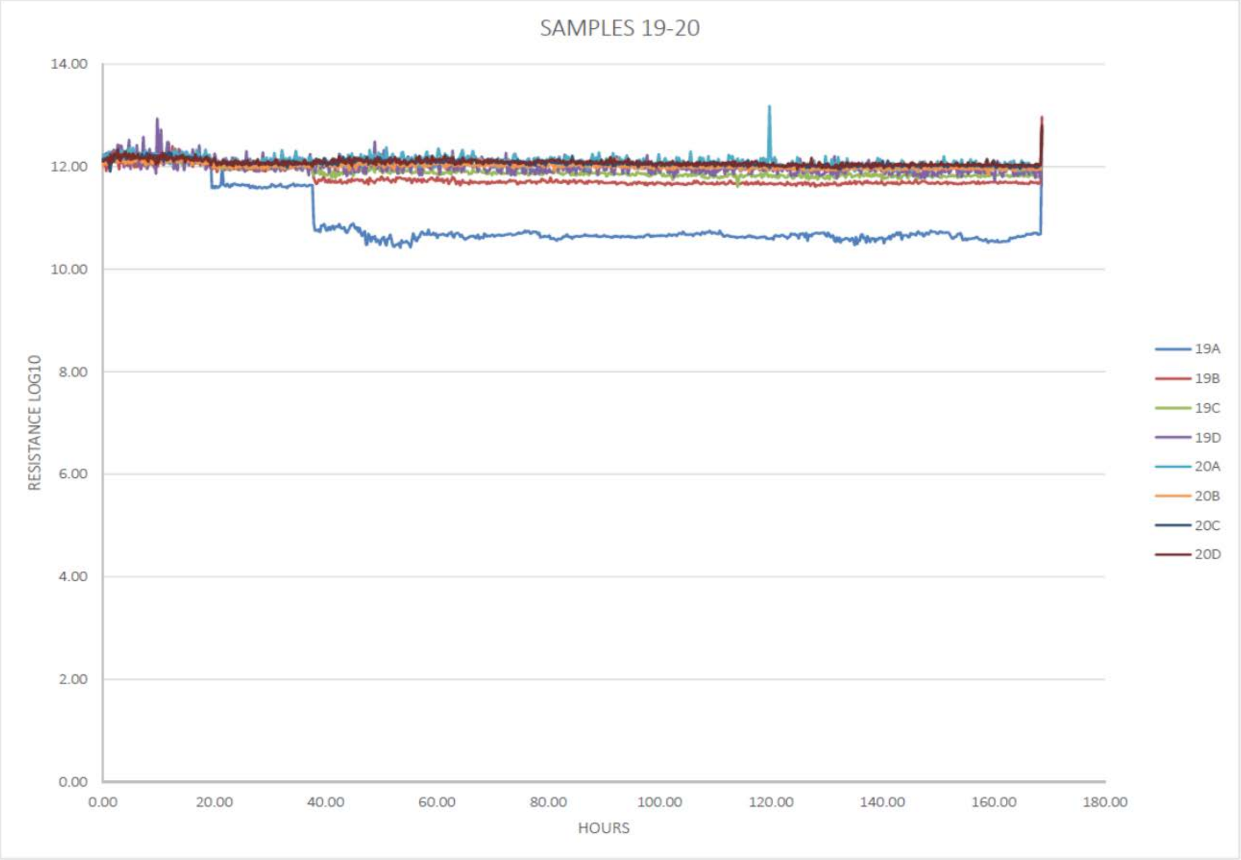


Figure 15: Halogen-Containing – Refrigerated – 91 Days

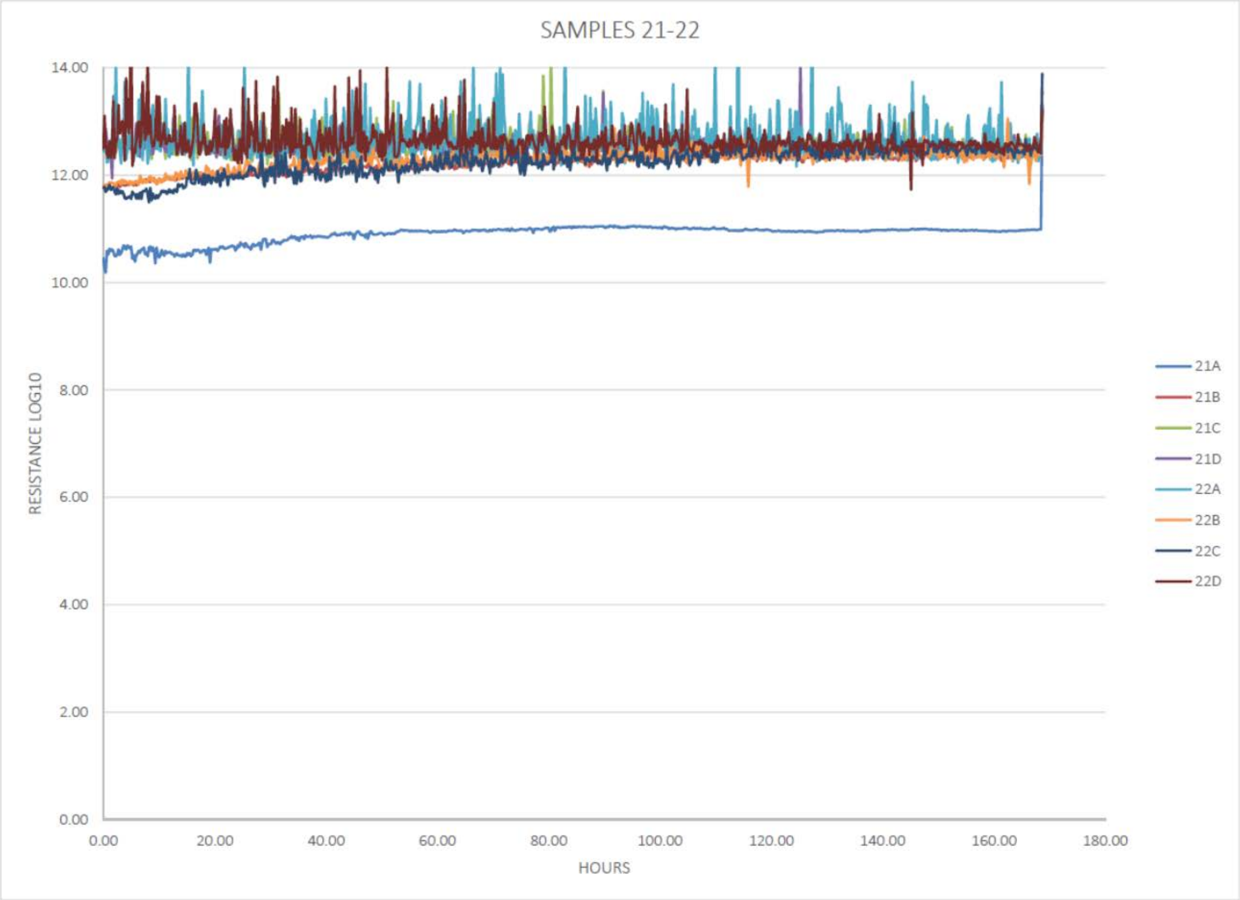


Figure 16: Halogen-Containing – Refrigerated – 61 Days

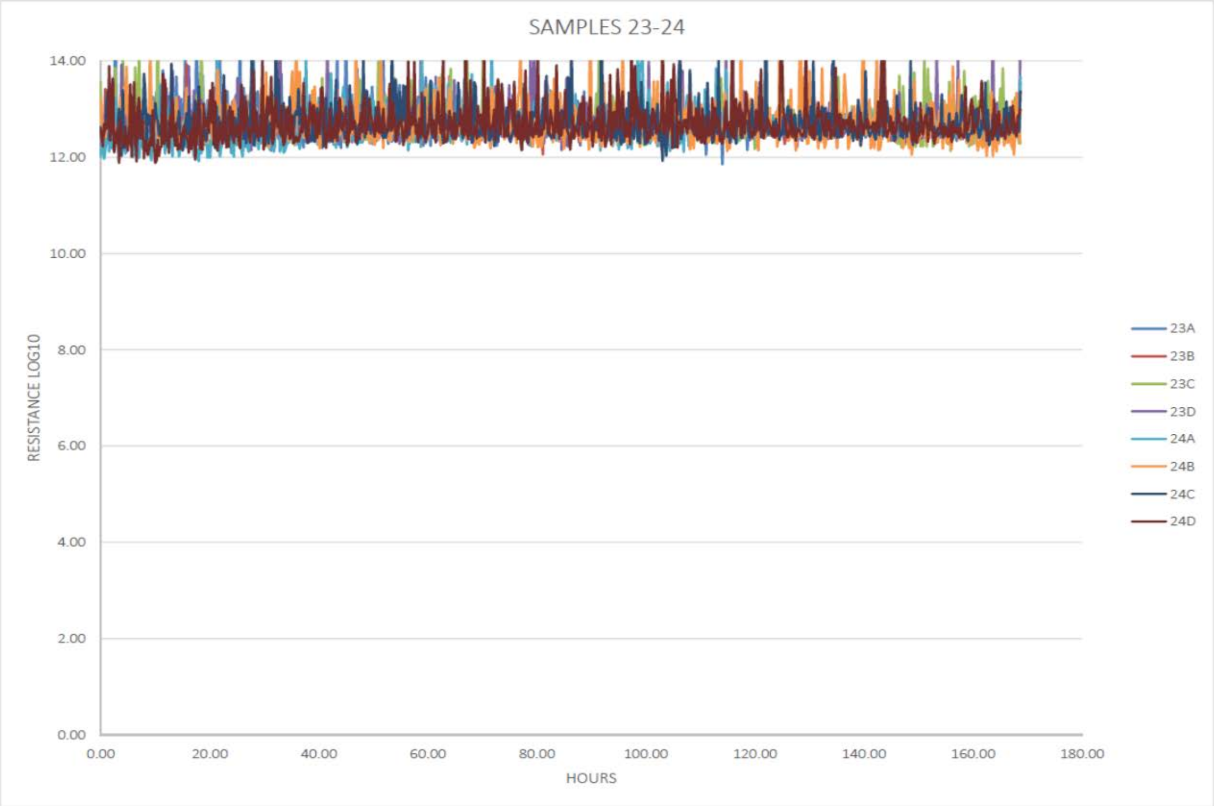


Figure 17: Halogen-Containing – Refrigerated – 19 Days

Figure 18: Controls

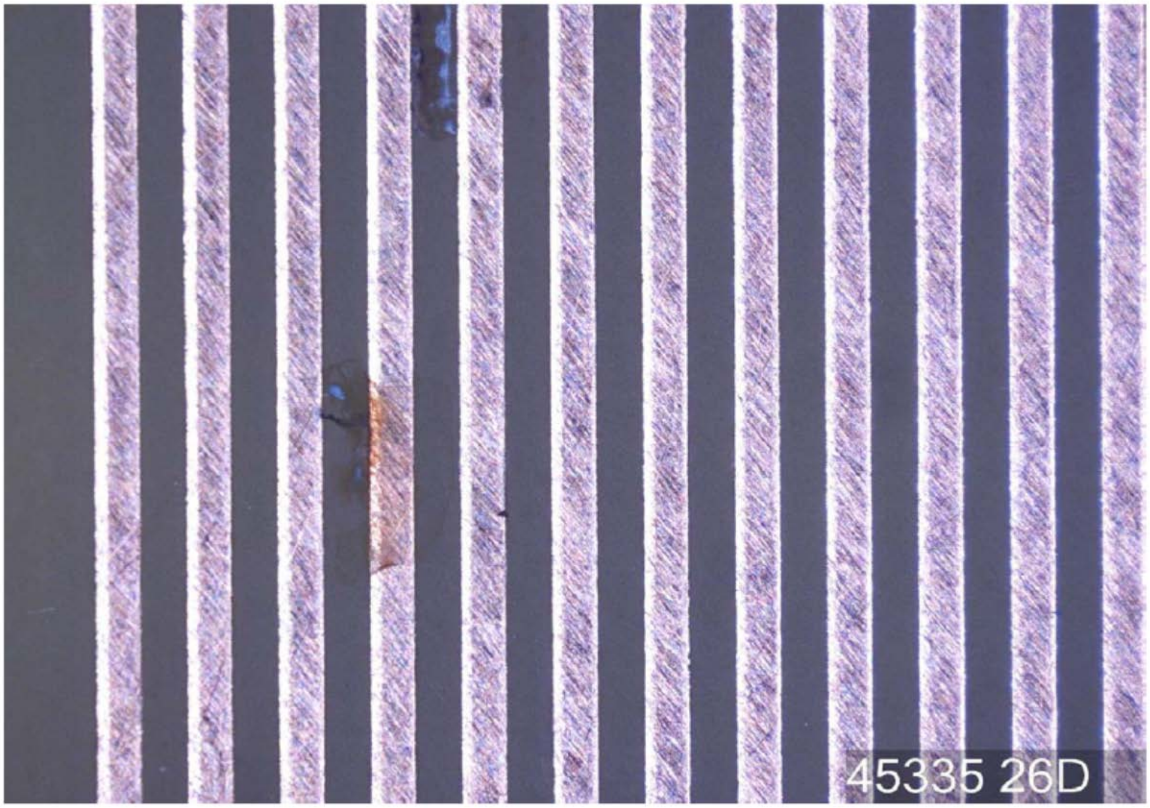


Figure 19: Debris on Control Board 26, Pattern D

As mentioned earlier, the intent of this work is to look for the possible impact of age and storage conditions on no-clean solder pastes as it relates to the SIR performance of their respective residues. In order to capture a general effect or trend, all the SIR data points for each individual scenario were averaged and plotted against each other (see Figure 20). Each average is derived from 4032 readings (2 boards, 4 patterns per board, a reading every 20 minutes over 168 hours). The average from the control boards is presented twice. The plot labeled “CTRL ALL” is the average of all the readings taken from the control boards. The plot labeled “CTRL Mod” is the average of all the readings taken except for the readings of the pattern that was identified to have debris on it. The averages are presented by Paste/Condition/Age (along the X axis of the graph). Below is the definition of the codes used in Figure 20.
HF = Halogen-Free Solder Paste
HC = Halogen-Containing Solder Paste
RT = Room Temperature Storage
Ref = Refrigerated Storage
CTRL = Control Boards
91, 61, or 19 = Number of Days in Storage

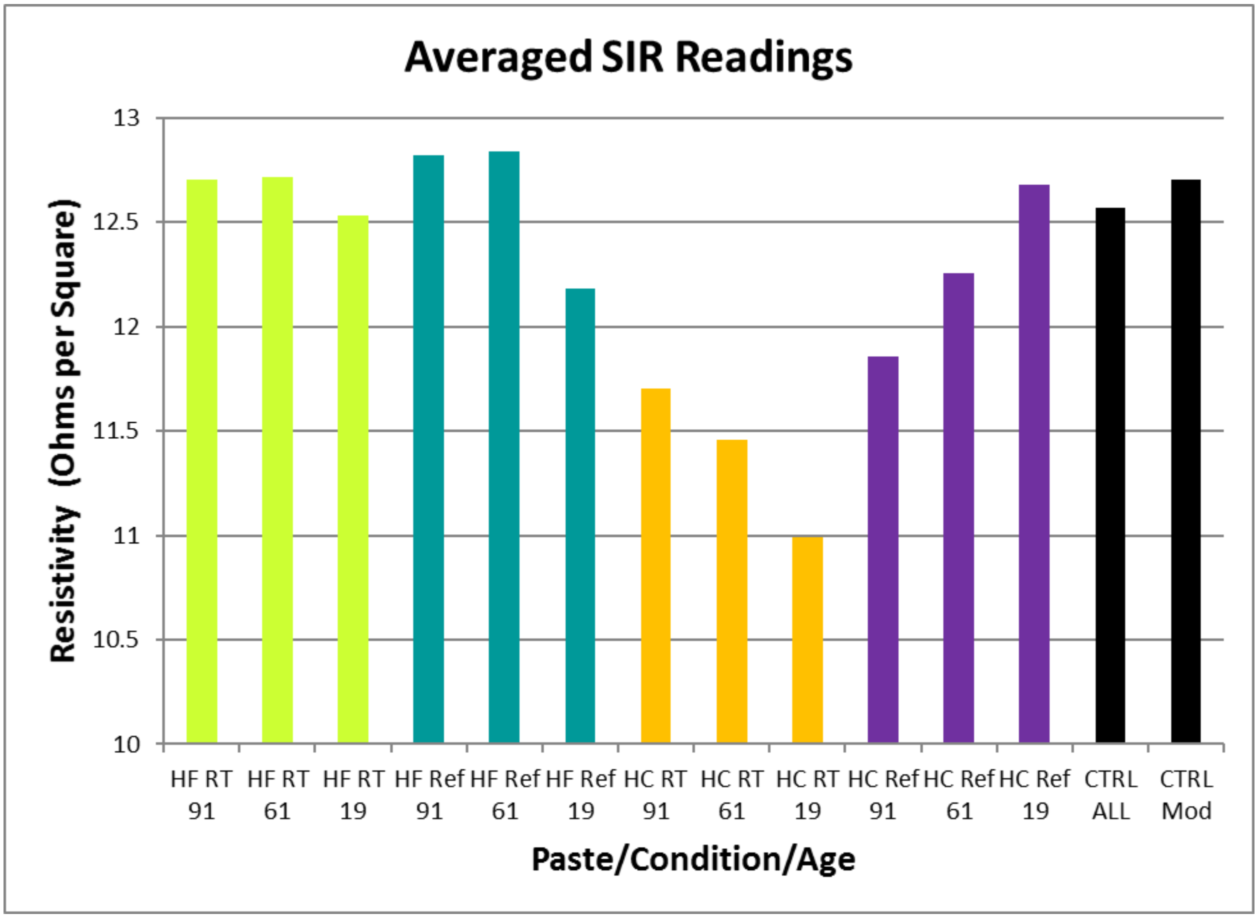


Figure 20: Averaged SIR Readings

First and foremost, it should be mentioned that regardless of age or storage conditions, all scenarios “pass” per the IPC J-STD-004B SIR requirements of a minimum SIR value of 1 x 10^8 ohms per square.

The averaged SIR values, as seen in Figure 20, suggest that the halogen-free solder paste is somewhat immune to the storage conditions, as both the room temperature and refrigerated scenarios produce similar SIR values. It would also seem to indicate that this halogen-free formulation may actually benefit (produce higher SIR values) when aged, as the 91- and 61-day-old pastes produced higher SIR values than the 19-day-old paste.

The halogen-containing solder paste appears to be sensitive to the storage temperature. The refrigerated samples produced consistently higher SIR values than those stored at room temperature. However, the SIR performance of the room temperature samples seems to improve with age, whereas- the refrigerated samples show lower SIR values with age.

Interestingly, sometimes a pattern or patterns, in a given scenario, produce noticeably lower SIR results than the other patterns. For the sake of this paper, these patterns shall be referred to as "divergent patterns." Figure 21 shows an example of what is being defined as a divergent pattern; see the two pattern plots (blue and purple) indicated by arrows at the left of the graph.

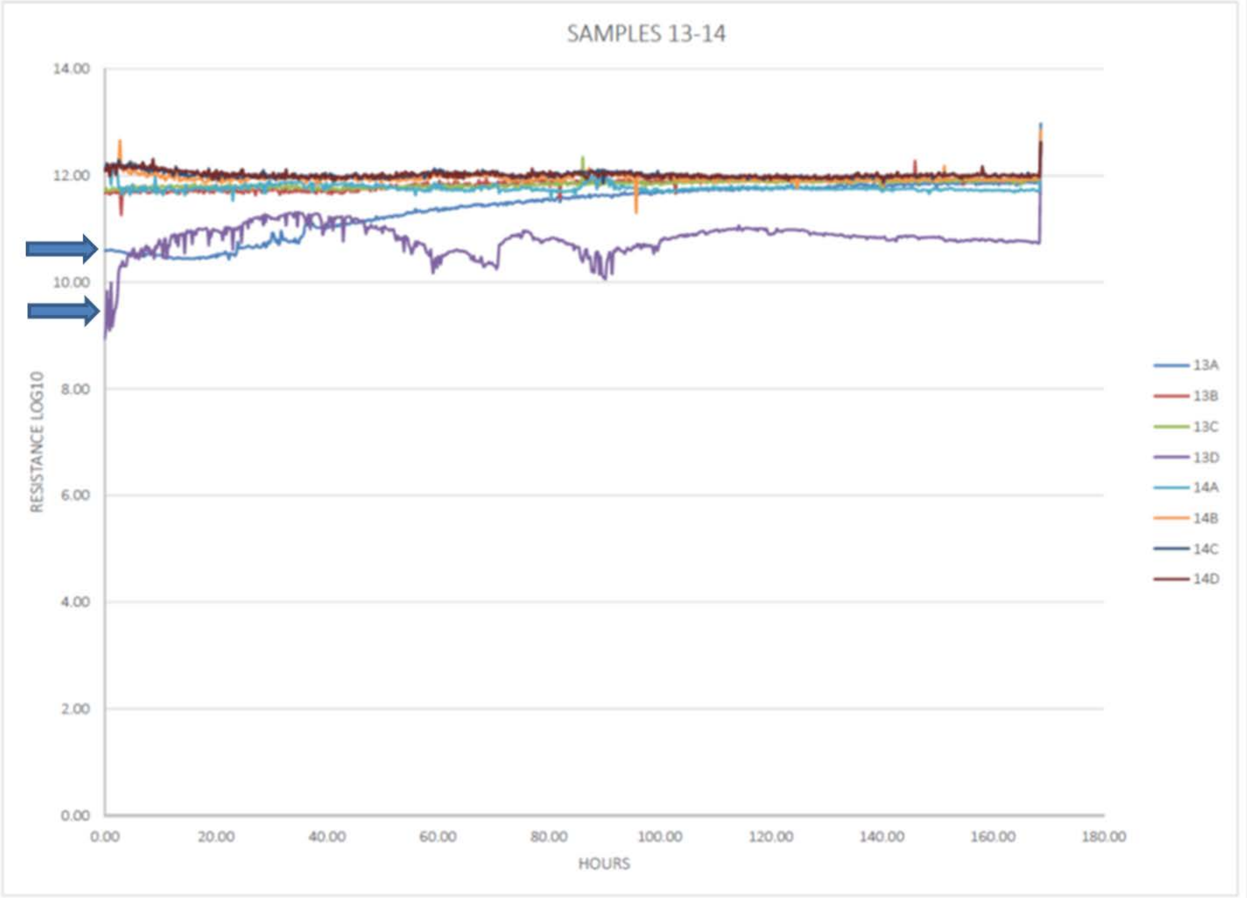


Figure 21: Example of Divergent Patterns

There seems to be a correlation or trend between the number of divergent patterns and the scenario (Paste/Condition/Age) as shown in Figure 22. With the exception of the halogen-containing refrigerated paste, newer pastes exhibit more divergent patterns than older pastes. Is there something that is present in newer pastes that breaks down into something innocuous as the paste ages?

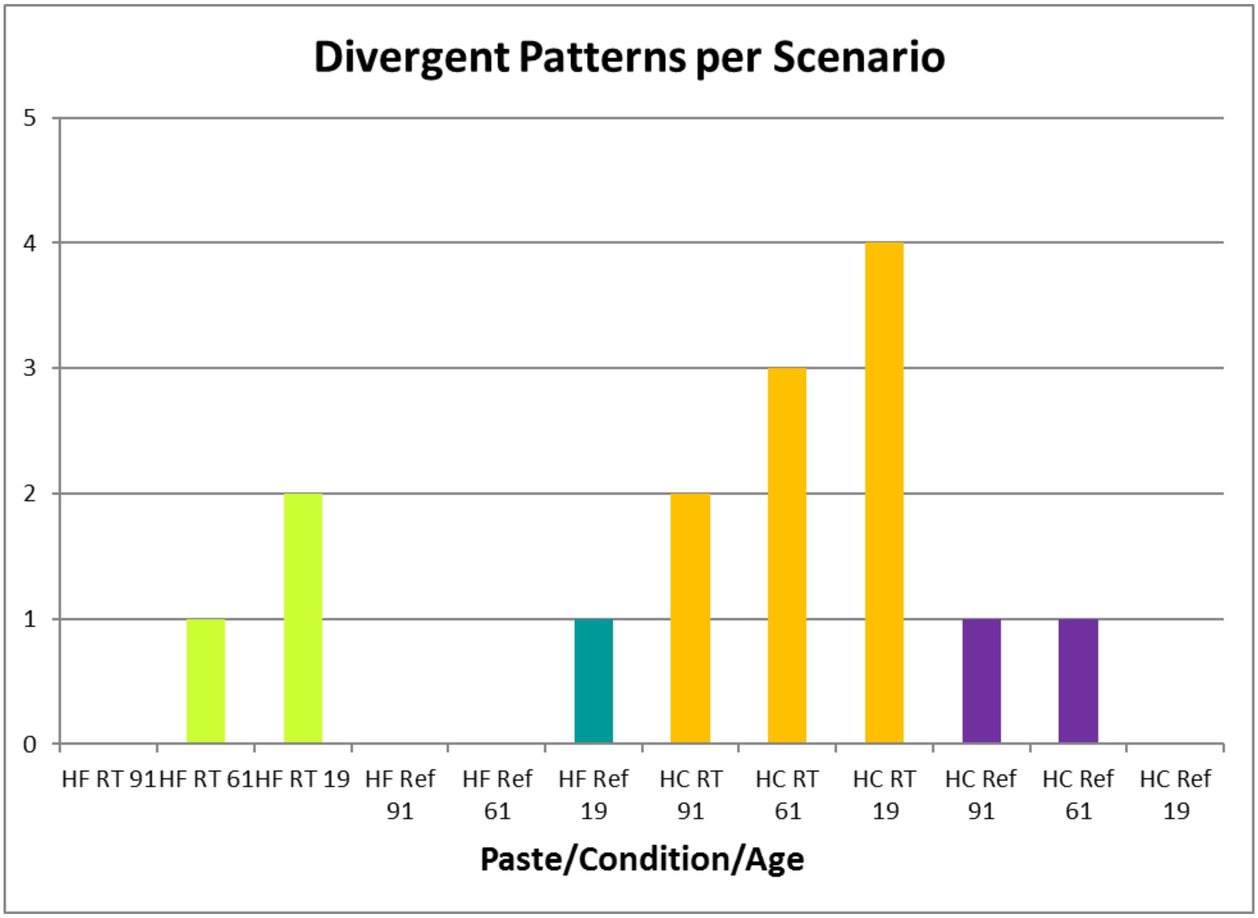


Figure 22: Number of Divergent Patterns per Scenario

To examine the possible relationship between scenario, average SIR values, and number of divergent patterns, all three were plotted on the same graph (see Figure 23).

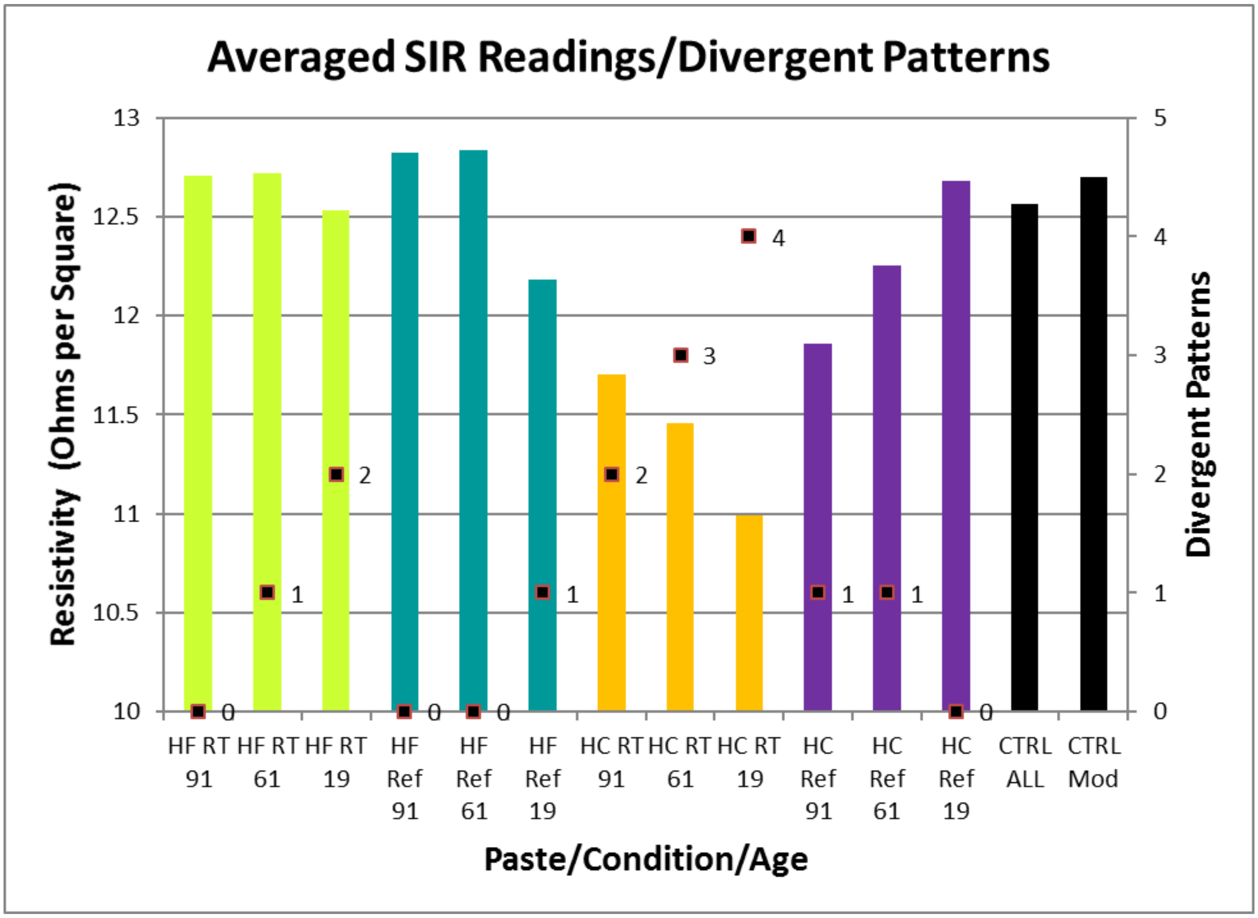


Figure 23: Scenario/Average SIR Values/Number of Divergent Patterns

It is obvious that a lower average SIR value for an individual scenario is often the result of one or more divergent patterns. And, the greater the number of divergent patterns for a scenario, the lower the average SIR value and vice versa.

Another observation that can be made by examining the SIR plots for each scenario is that the non-divergent patterns, or those patterns whose SIR values are closely grouped, are very good (high), often in the 1 x 10^12 ohms per square range. In the absence of dendrites or other obvious causes of the divergent patterns, it is difficult to explain why they occur when the other patterns maintain very good SIR values.

If a solder paste changes as a function of storage conditions and age, it must not be a change that occurs throughout the entire volume of solder paste within a given container. Rather, one is led to assume that it occurs at isolated locations within the bulk solder paste. There may be scenario-induced reactions or interactions that occur only at certain locations that have the right combination of factors which are not evenly distributed throughout the entire volume of solder paste, e.g., nucleation sights, that allow for crystal growth, etc. (The author is not chemically savvy enough or knowledgeable enough about the flux chemistries to be able to comment or speculate with any level of certainty.) Because these locations are not found throughout the entire container of paste, they get deposited only on some of the patterns during the printing process. This may explain the presence or occurrence of the divergent patterns and accompanying lower SIR values. If so, the frequency of divergent patterns would likely be linked to the number of locations within the bulk solder paste.

The author does not presume to be in a position of knowledge or authority such that comments can be made with certainty about how and why, or even if, these subtle differences really occur or matter. But there seems to be some meaningful ideas that can be gleaned by careful study of the data.

In closing, we can see that solder paste flux chemistry, storage temperature, and age seem to have a subtle impact on SIR performance of the flux residue. The performance differences that may exist are not enough to threaten an SIR failure as defined by IPC J-STD-004. Nevertheless, those applications that are especially sensitive to current leakage may want to take these factors into consideration when selecting and storing a no-clean solder paste.