

# Does Solder Particle Size Impact the Electrical Reliability of a No-Clean Solder Paste Flux Residue?

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## Abstract

No-clean soldering processes continue to dominate the electronics manufacturing world, especially amongst consumer-type electronics. For many years, type 3 was pretty much the “standard” solder paste particle size with a distribution of 25 – 45 microns (-325/+500 mesh). However, the ongoing trend toward ever-increasing miniaturization is putting pressure on solder paste manufacturers to produce solder pastes that can reliably print through smaller and smaller stencil apertures. While great advances have been made in solder paste flux technology to accommodate very small apertures, those advances alone cannot meet all the challenges of miniaturization. There is a point at which reducing the solder particle size becomes necessary in order to create solder pastes which can provide adequate transfer efficiency for small stencil apertures. As a result, many modern solder pastes are available on the market with particle sizes smaller than type 3 such as type 4 (20 – 38 microns), type 5 (15 – 25 microns), type 6 (5 – 15 microns), as well as non-IPC particle size distributions like type 4.5 (20 – 32 microns). Reducing the solder particle size for a given volume or mass of solder powder increases the total surface area. Therefore, reducing the particle size increases the surface area that the flux component of the solder paste needs to clean to cause good coalescence and wetting of the solder. The ingredients in the flux responsible for cleaning the surfaces are generically called activators. Because activators are corrosive in nature, they are one of the flux ingredients that can have a substantial impact on the electrical reliability of the flux residue. With decreasing solder particle size, resulting in increasing surface area, the flux has to work harder to clean these surfaces, consuming more of the activators while increasing the amount of activator/metal oxide interaction by-products in the flux residue. Therefore, everything else being equal, changing (reducing) the particle size is likely changing the contents of the flux residue. With this in mind, one must ask: Does reducing the particle size of a no-clean solder paste have a measurable impact on the electrical reliability of the flux residue? If so, is it substantial enough to be a cause for concern?

This paper uses IPC J-STD-004B SIR (Surface Insulation Resistance) testing to examine these questions. Two commercially available Pb-free no-clean solder pastes with varying particle sizes, one halogen- containing and the other halogen-free, were tested to see if and how different flux chemistries respond to reduced particle size. All of the solder pastes in this study were submitted to the same common air Pb-free reflow profile.

## Background

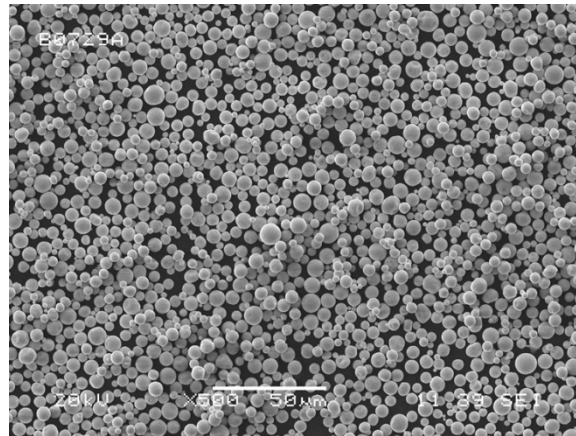
To understand the impact that reducing the particle size has on the surface area for a given mass of solder particles, the following table (Table 1) was created. This table uses type 3 powder as the point of reference and is assigned a value of 1. The other particle size surface areas are expressed as a ratio of the surface area of type 3 powder.

**Table 1 - Theoretical Mean Particle Size Surface Area Ratio for a Given Mass of Powder**

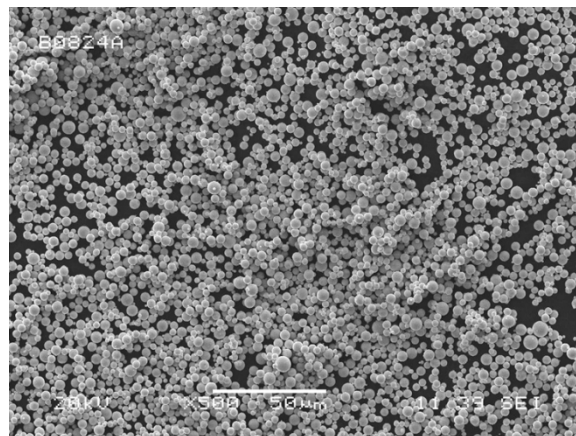
Type	Particle Size Distribution (microns)	Mean Particle Size (microns)	Mean Particle Size Surface Area Ratio
3	25 to 45	35	1.00
4	20 to 38	29	1.21
4.5	20 to 32	26	1.35
5	15 to 25	20	1.75
6	5 to 15	10	3.50

The purpose of Table 1 is to demonstrate that the surface area of the solder particles in the solder paste that the flux vehicle has to clean increases with decreasing particle size. Everything else being equal, switching from a type 3 to a type 4 powder results in a 21% increase in the surface area for a given mass of solder powder. Switching from a type 3 to a type 4.5 powder results in a 35% increase in the surface area, and so on. In the case of switching from a type 3 to a type 4.5 powder, the flux has to work 35% harder, potentially consuming 35% more activators and producing 35% more flux/metal oxide by-products. Decreasing the particle size increases the surface area of the solder powder, increases the amount of work that the flux needs to do, and potentially results in more flux/metal oxide by-products and less remaining activators in the solder paste flux residue.

Even slight changes in the particle size distribution can have a significant impact on the overall surface area. This is especially true in the case of ultrafine powders (e.g., Types 6, 7, and 8). Figure 2 shows a distribution of 2–11 microns and Figure 3 shows a distribution of 2–7 microns. It is very easy to see just from the SEM images that the Figure 3 powder (2–7 microns) would have significantly more surface area than Figure 2 (2–11 microns).



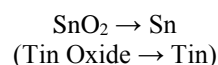
**Figure 2 - 2–11 micron powder**



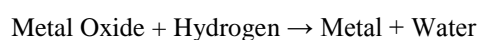
**Figure 3 - 2–7 micron powder**

It is important to note that the amount of oxidation present on the solder powder will be a function of the oxidation that occurs during the manufacture and handling of the solder powder and the oxidation that happens during the reflow process (heating), especially in an air reflow environment. While great care is taken to minimize the amount of oxidation that occurs during manufacture and handling of the solder powder, it is not possible to completely prevent it. For this work it is assumed that the solder powder for all solder pastes studied was exposed to the same oxidizing conditions.

Some people wrongly believe that the interaction of the flux vehicle and metal oxides is one of a reduction reaction. In other words, the flux activators reduce the metal oxides back to elemental metal. For example:



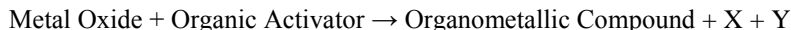
Or, more generically and completely:



However, this is not the case for a ROL0 or ROL1 solder paste. The flux vehicle does not reduce or convert the metal oxides back to pure metal. Rather, the flux activators react with the metal oxides and convert them into a compound that is soluble in the flux. Once the activators have converted the metal oxides into soluble metal compounds, the flux washes the soluble metal compounds away and allows metal-to-metal coalescence and wetting to occur. In the case of a solder paste with a halogenated flux vehicle, the generic reaction looks something like this:



In the case of a solder paste with a halogen-free flux vehicle, the generic reaction looks something like this:



It is important to keep in mind that the by-product(s) of the interaction between the flux activators and metal oxides, be they metal salts and/or organometallic compounds, become trapped and encapsulated in the solder paste flux residue. With decreasing particle size and increasing solder powder surface area, there is an increase in the amount of flux/metal oxide interaction by-products in the flux residue. How and if these by-products impact the electrical reliability of the flux residue is the focus of this study. As mentioned earlier, this study uses J-STD-004B SIR to examine this issue.

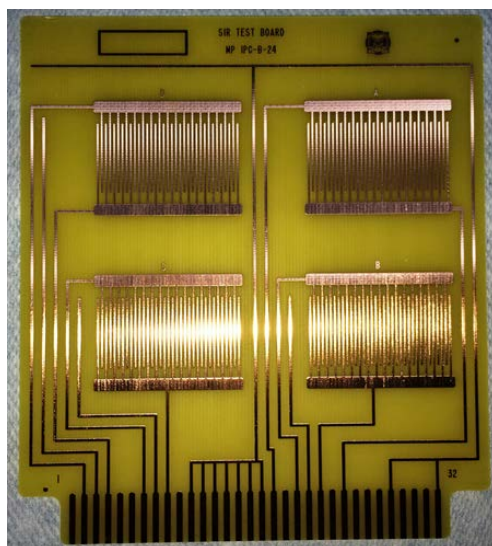
It should be noted that up to now the discussion has been from a theoretical point of view. However, it should be kept in mind that one of the parameters of a solder paste is its metal load. Even with the flux vehicle and solder alloy being the same, there is often a change in the metal load as the particle size changes, especially for solder pastes designed for stencil printing. While these changes are often small, it does present a variation from solder paste to solder paste, specifically, in the amount of solder powder, and, therefore, the solder powder surface area present in a given volume of solder paste. Typically, as the particle size gets smaller for a given solder paste, there will also be a reduction in the metal loading. The metal load is reduced to maintain an optimized viscosity for stencil printing. Table 2 shows the metal loads of the solder pastes used in this work. Furthermore, this work does not consider the amount of oxidation that may be present on a component lead and/or pad and/or a PCB pad where the solder paste flux would be responsible for cleaning (removing oxidation) as well as considering the surface of the solder powder. In this work it is assumed that the amount of oxidation present on the bare copper traces on the IPC-B-24 SIR test boards used is equivalent.

**Table 2 - Solder Paste Metal Loads**

	Metal Load Per Solder Paste Type	
Powder Type	Halogen-Free (ROL0)	Halogenated (ROL1)
Type 3	89%	88.75%
Type 4.5	88.50%	88.50%
Type 6	88%	88%

### Experimental Procedure

The IPC-B-24 SIR test board (Figure 3) was used as the vehicle for this study. Each board has 4 comb patterns, A through D. Each comb pattern has 0.4mm bare copper traces with 0.5mm spacing. The boards were prepared in accordance to IPC-TM-650 2.6.3.3 and tested per IPC-TM-650 2.6.3.7 for 168 hours (7 days). The environment inside the test chamber was 40°C with 90% RH. An electrical bias was applied to the comb patterns creating a field strength of 25V/mm. A resistivity measurement was made of each comb pattern at least once every 20 minutes.



**Figure 3 - IPC-B-24 SIR Test Board**

Table 3 shows the test matrix used for this experiment. Four SIR test boards were prepared for each solder paste type (halogenated and halogen-free) and powder type (Types 3, 4.5, and 6). Two control boards were also prepared.

Table 3 - Test Matrix

			Solder Paste Type			
Powder Type	Particle Size Distribution	Mesh Size	Halogen-Free (ROL0)	Halogenated (ROL1)		
Type 3	25 – 45 microns	325/500	4 boards	4 boards		
Type 4.5	20 – 32 microns	450/635	4 boards	4 boards		
Type 6	5 – 15 microns	N/A	4 boards	4 boards		
IPC-B-24 SIR Test Board						
					2 boards	Controls

The SIR test boards were cleaned prior to use in accordance with IPC-TM-650 2.6.3.3.

The solder pastes were stored under refrigeration until the time of use. Before use, they were removed from refrigeration and allowed to come up to room temperature in the closed jar. A 150-micron thick (~.006") stencil was used to print the solder pastes onto the SIR test boards as prescribed by IPC-TM-650 2.6.3.3. After the solder paste was applied to the SIR test boards, the boards were sent through the same reflow profile (Figure 4). As can be seen in Figure 4, a linear Ramp-to-Peak profile with a peak temperature of ~239°C was used with 64 to 66 seconds over 217°C.. A previous work by the author [1] has shown that the shape of the profile, Ramp-to-Peak versus Ramp-Soak-Spike, and the peak temperature also appear to have an impact on SIR performance of no-clean solder paste flux residues.

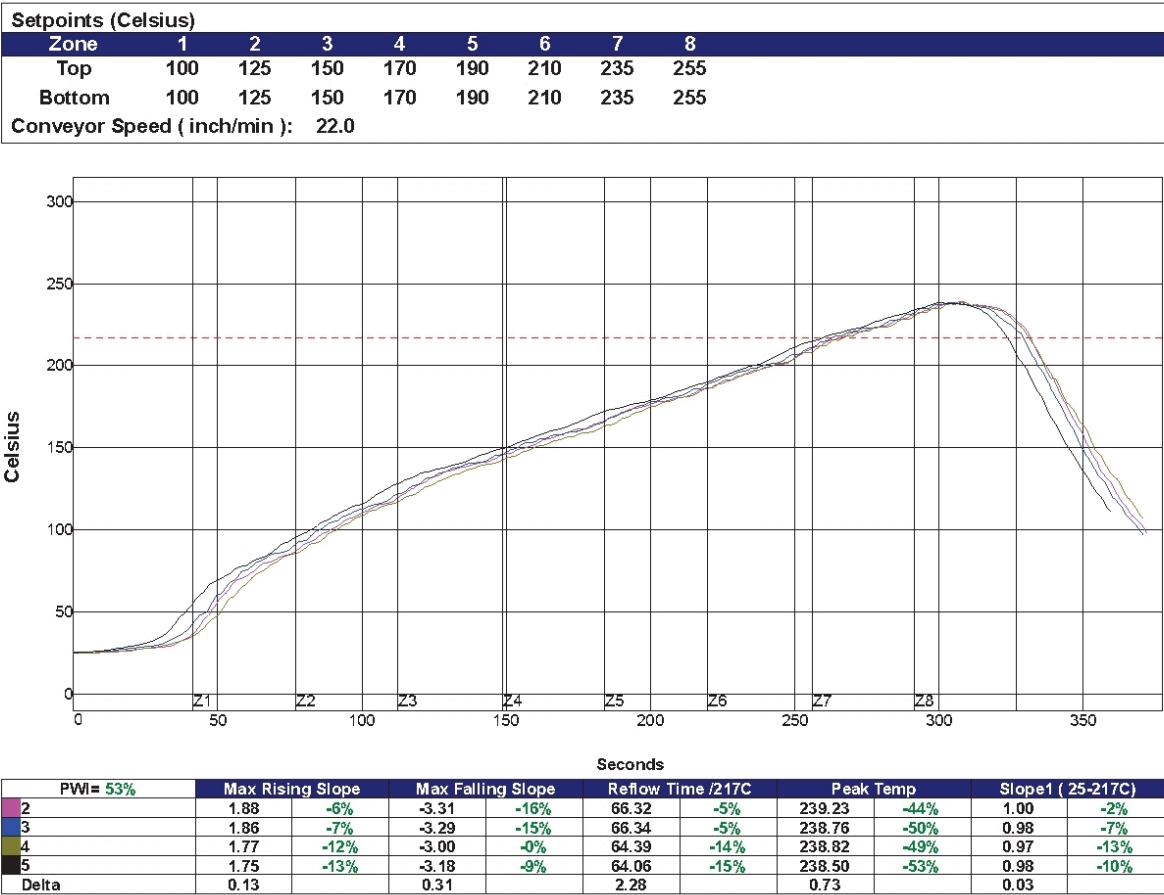
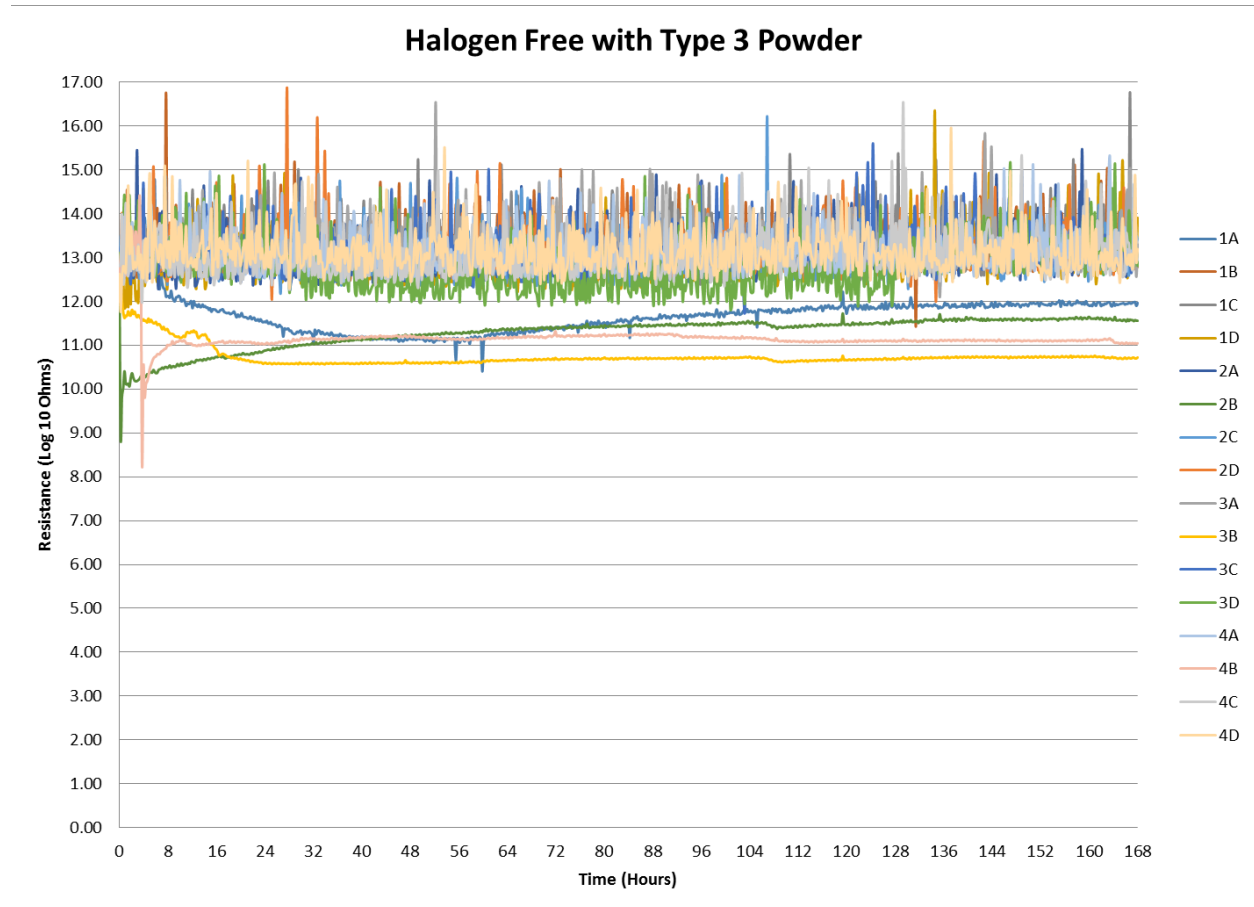


Figure 4 - Reflow Profile

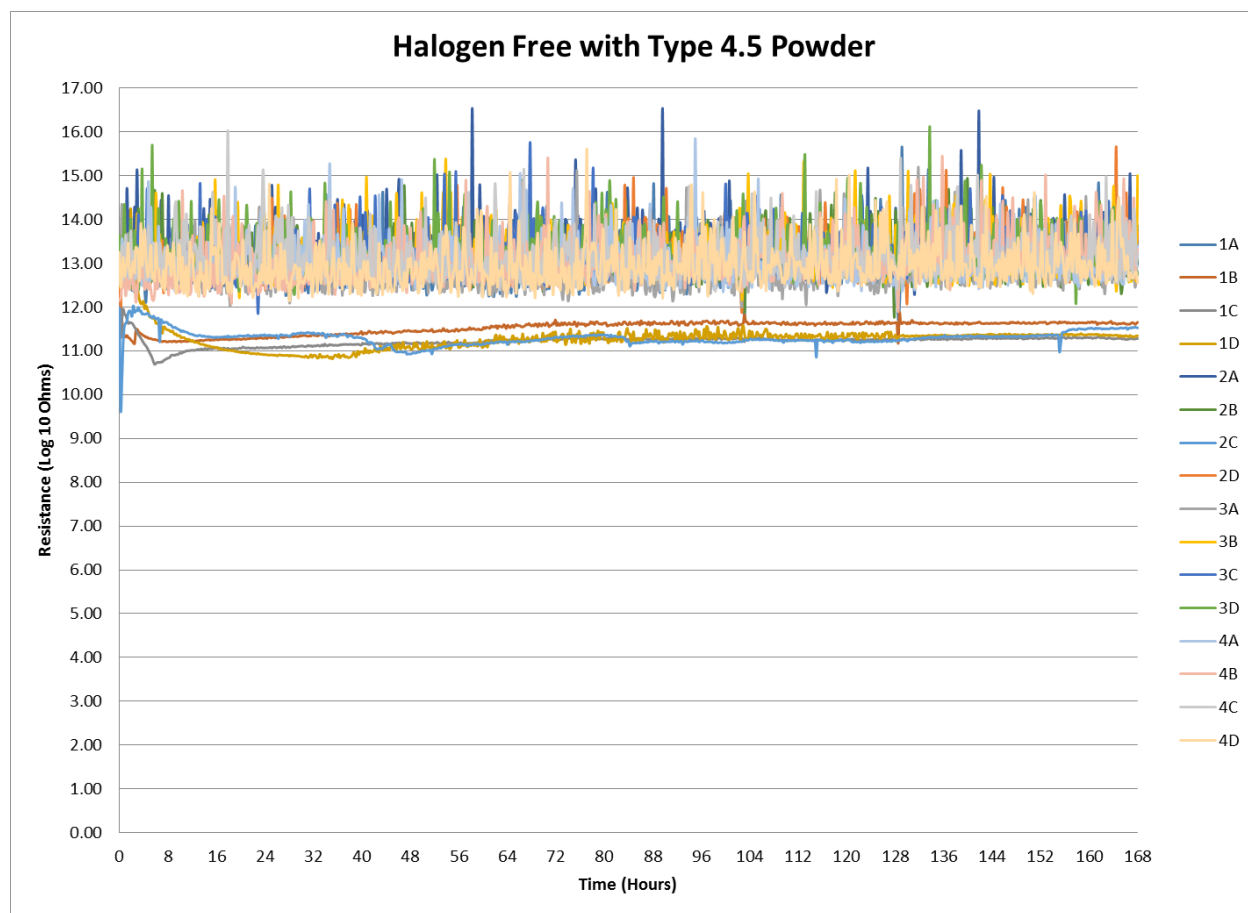
After being reflowed, the SIR test boards were tested per IPC-TM-650 2.6.3.7 for a period of 168 hours as prescribed by J-STD-004B standard.

## Results and Discussion

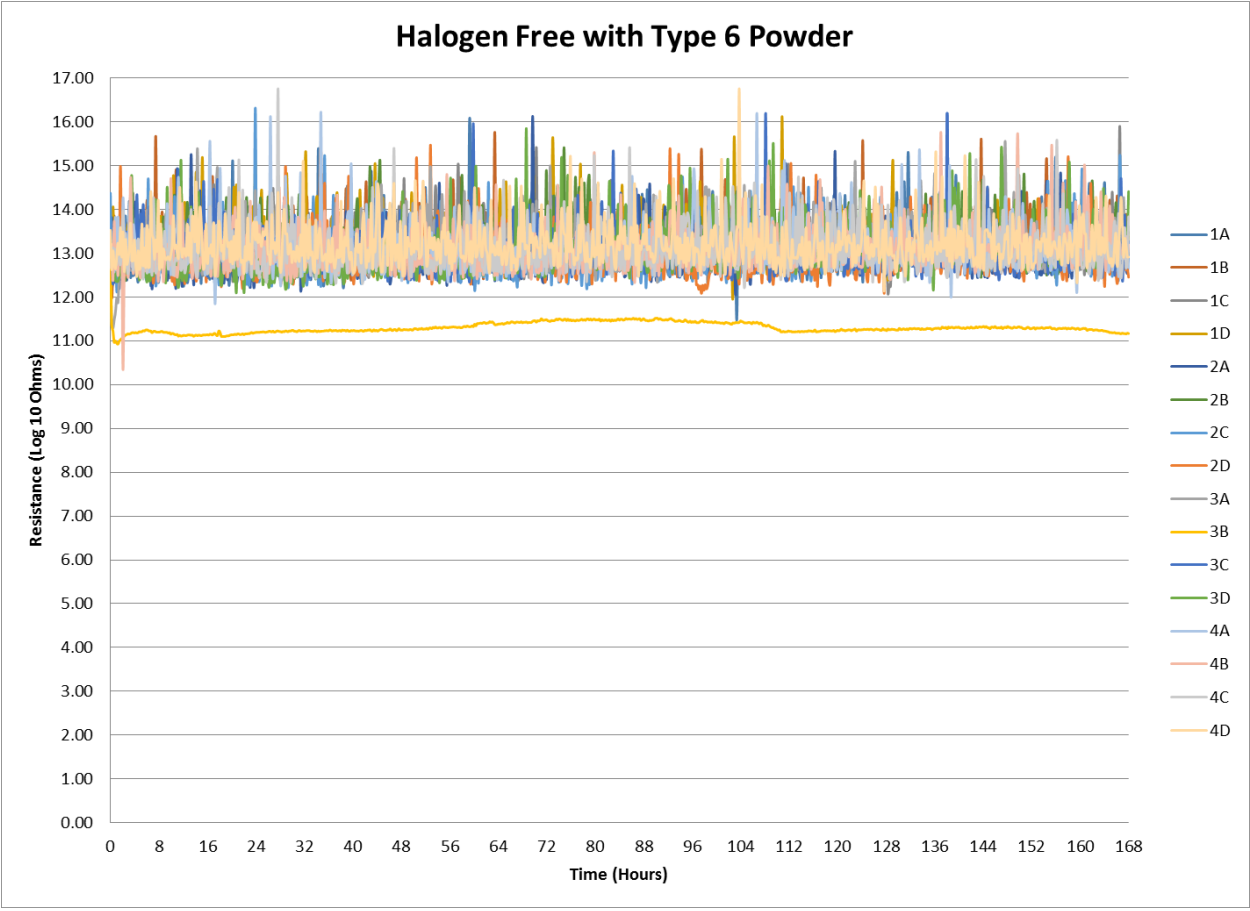
The following figures (Figures 5 through 11) show the results of the SIR testing for the different powder size and solder paste type.



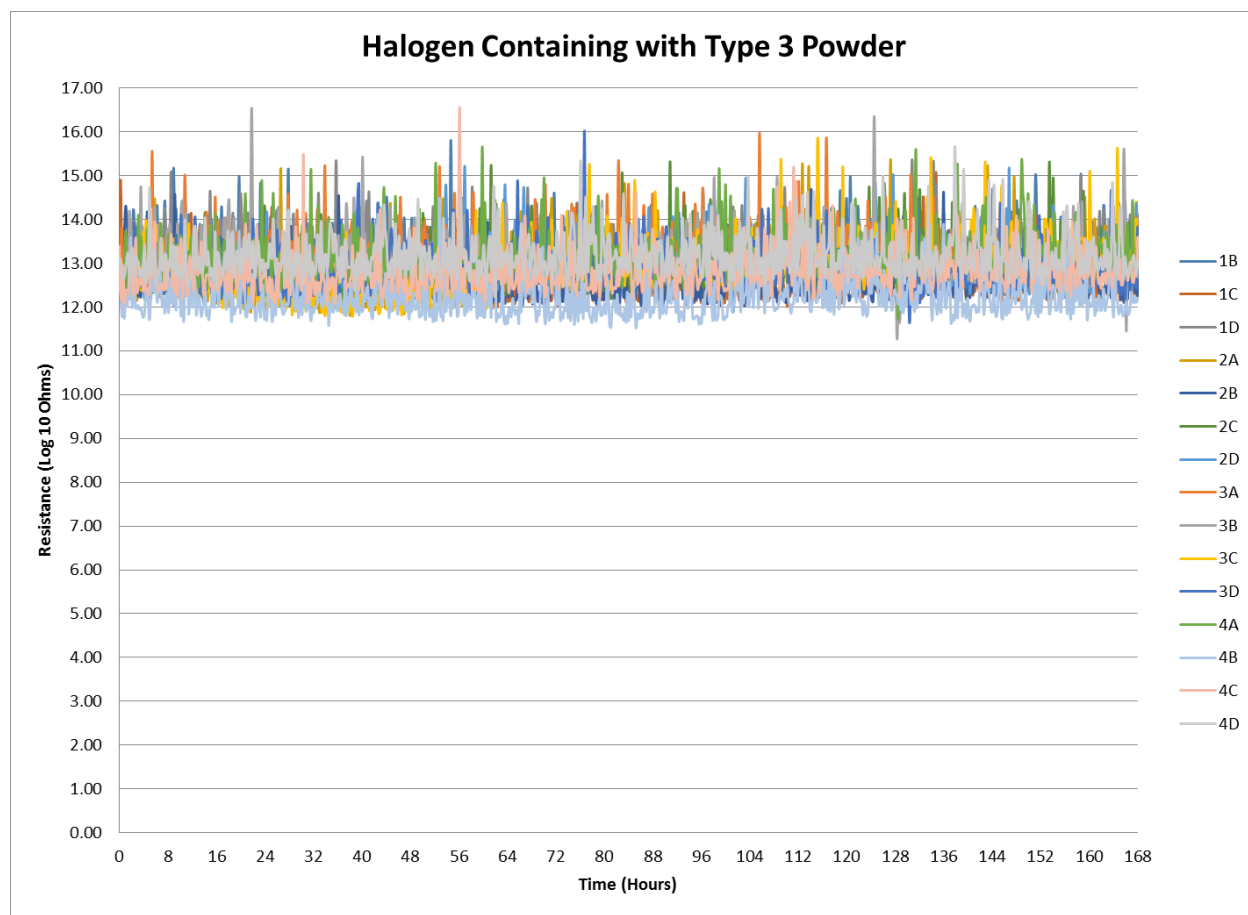
**Figure 5 - Halogen-Free with Type 3 Powder**



**Figure 6 - Halogen-Free with Type 4.5 Powder**



**Figure 7 - Halogen-Free with Type 6 Powder**



**Figure 8 - Halogen-Containing with Type 3 Powder**



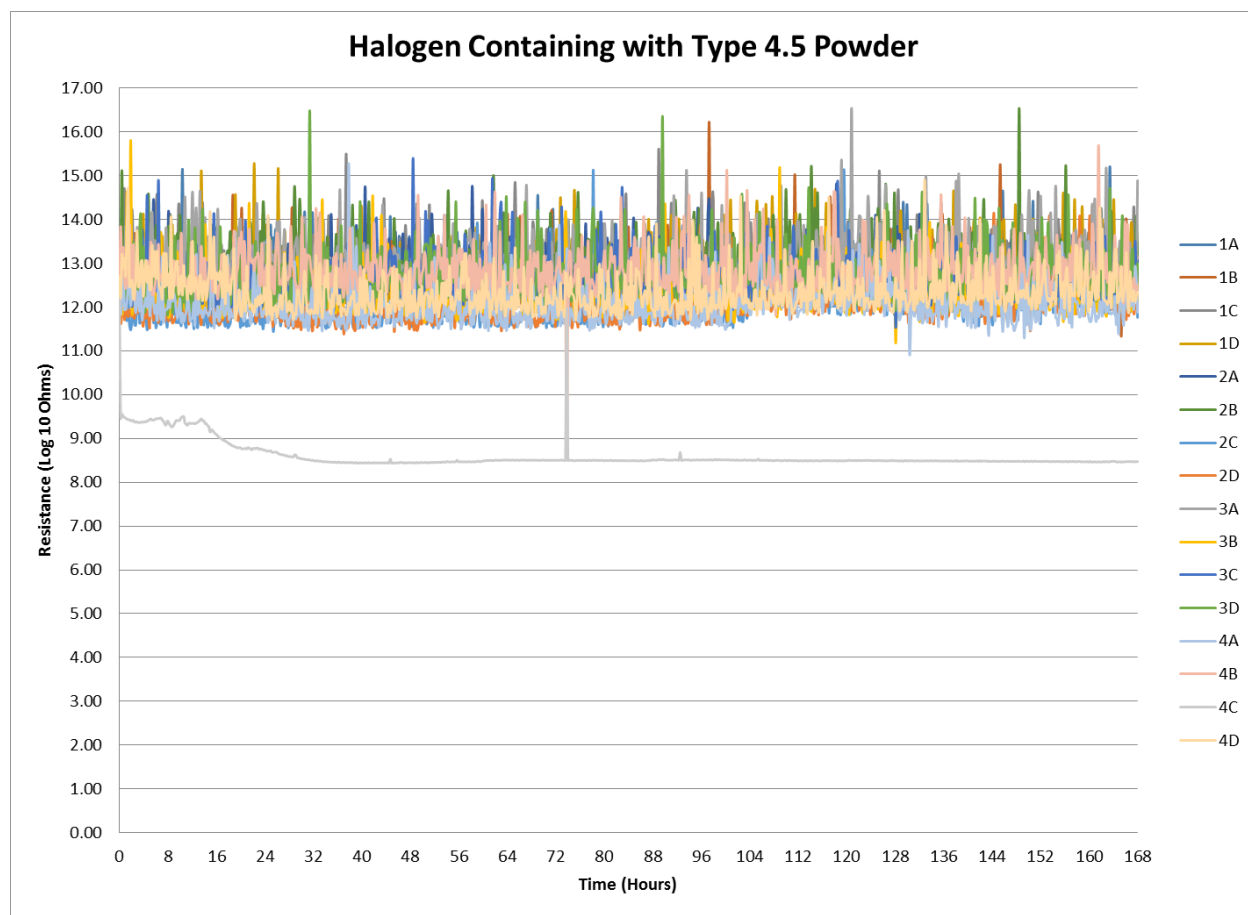


Figure 9 - Halogen-Containing with Type 4.5 Powder

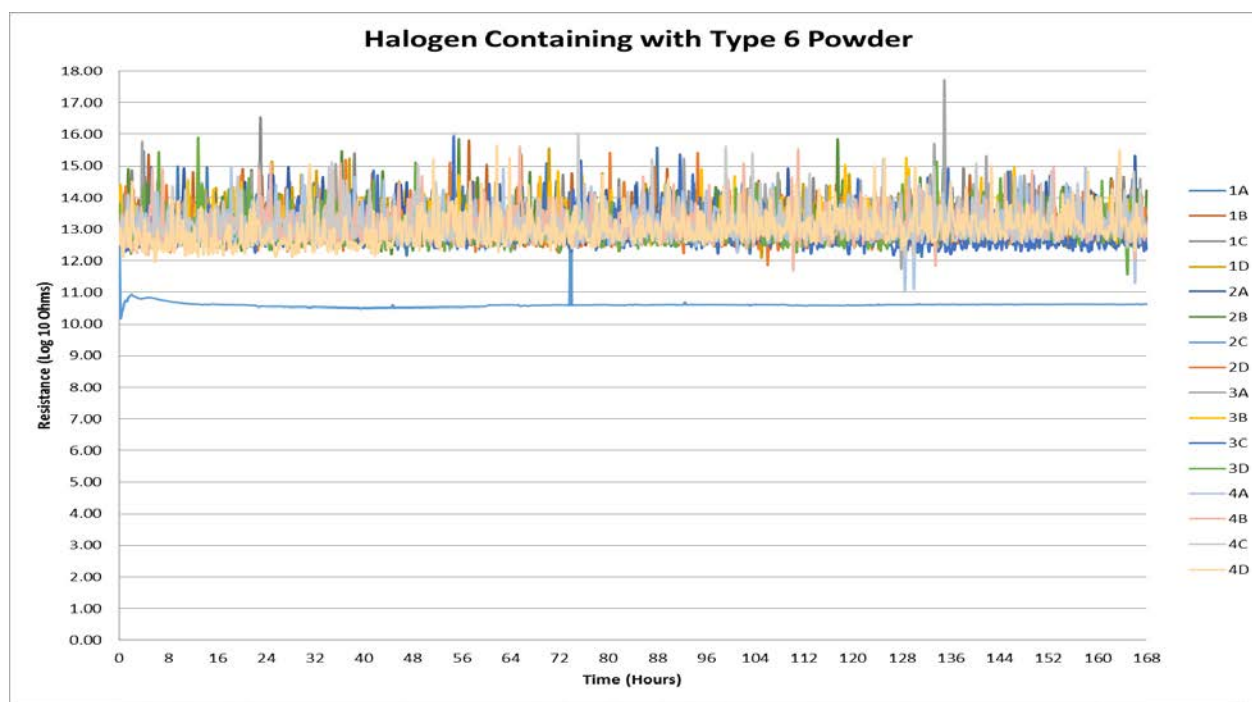
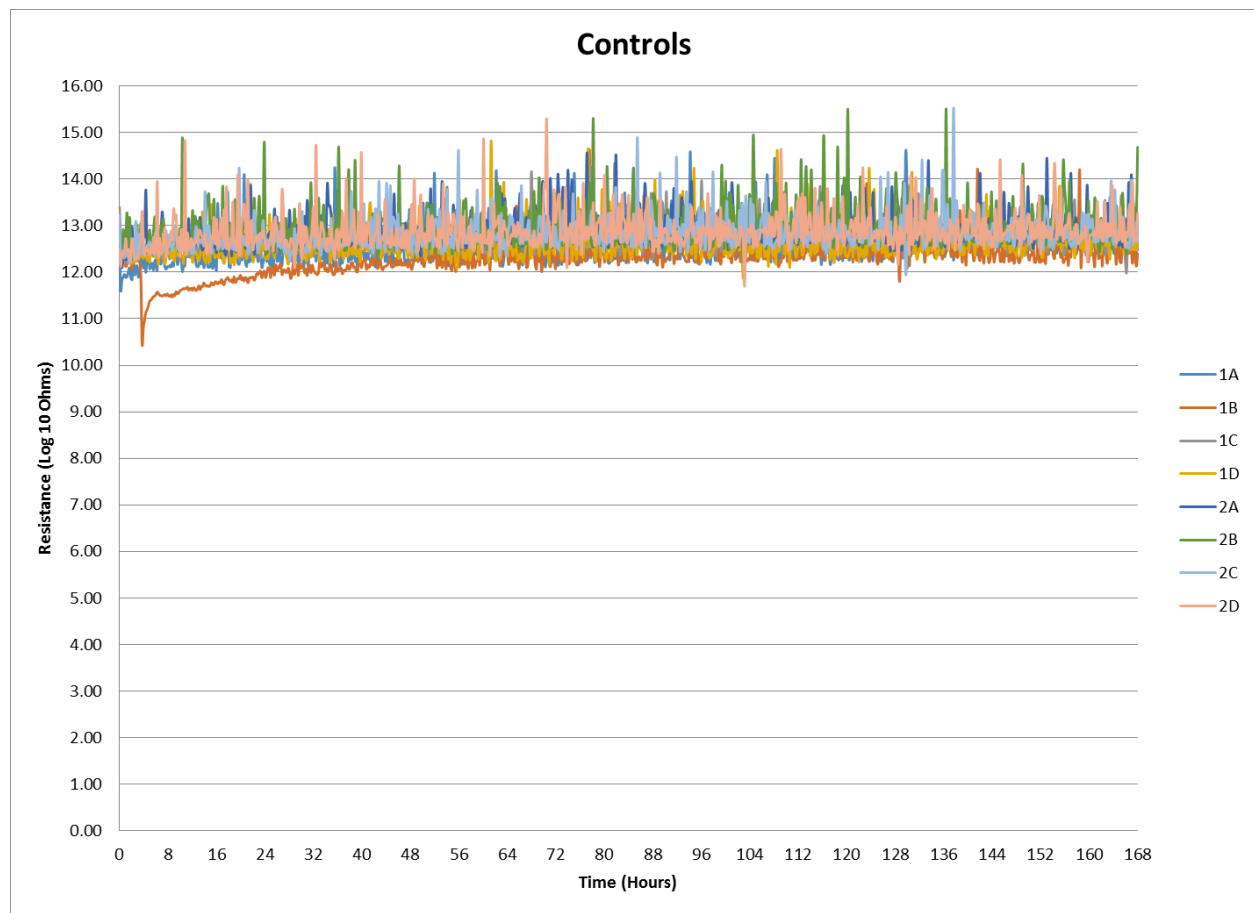
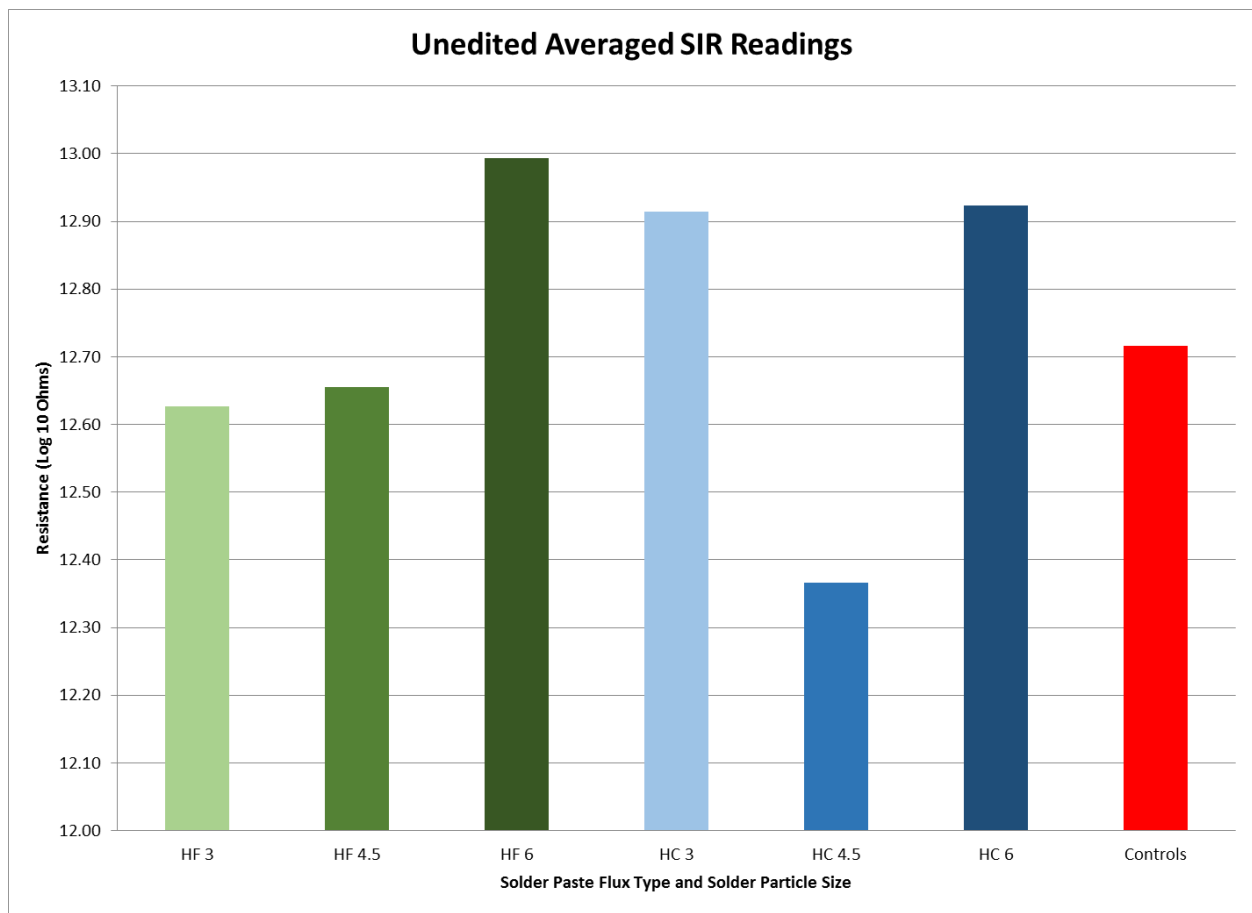


Figure 10 - Halogen-Containing with Type 6 Powder



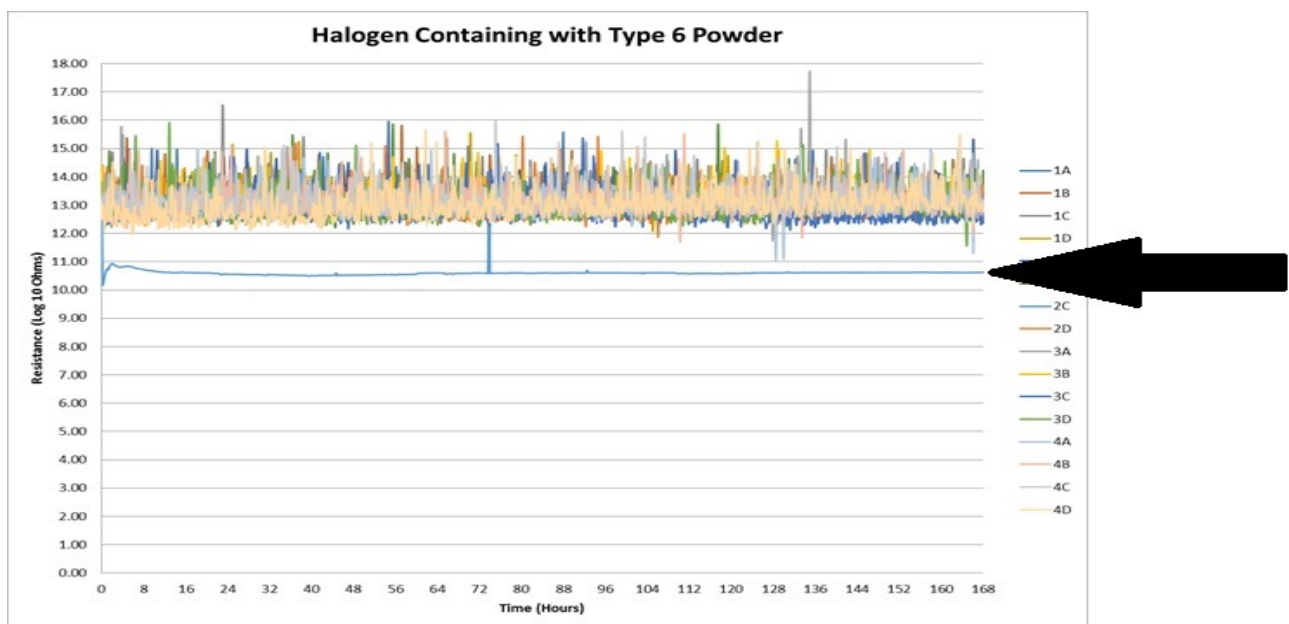
**Figure 11 - Controls**

The intent of this work was to determine if and how much the particle size impacted SIR performance of a given solder paste. To this end, the author was looking to see if there was any notable trend. In order to better identify any trend, all the SIR values for a given scenario were averaged together as shown in Figure 12. This figure is identified as “unedited” because there was no manipulation or omission of the raw data.



**Figure 12 - Unedited Averaged SIR Readings**

In the process of reviewing the SIR graph of each scenario, it was noticed that all but one scenario, halogen-containing with Type 3 powder (Figure 8), exhibited between 1 and 4 divergent patterns. A divergent pattern is a single comb pattern on a SIR test board that yields values significantly and consistently different (lower) than the other patterns. The black arrow in Figure 13 indicates a divergent pattern. The only pattern with an obvious assignable cause for a divergent pattern was 4C of the halogen-containing with Type 4.5 powder, where a piece of lint was observed (Figure 14).



**Figure 13 - Divergent Pattern**



Halogen Containing with Type 4.5 Powder      PATTERN 4C LINT

**Figure 14 - Lint on pattern 4C of Halogen-Containing with Type 4.5 Powder**

Because these divergent patterns depress the average SIR value for a given scenario, it makes it difficult to recognize if and to what extent a trend exists. For this reason, the data from the divergent patterns was removed and the graphs and averages redone (Figures 15 through 21). Removing data is not ideal. However, it is important to keep in mind that IPC requires that 3 SIR test boards (12 SIR patterns) be used per scenario. Because 4 SIR test boards (16 SIR patterns) were analyzed for each scenario, even with removing the data of up to 4 divergent patterns, the quantity of readings is still as numerous as or greater than that obtained from 3 SIR test boards.

Note that the halogen-containing with Type 3 powder graph (Figure 18) is shown again in this group of figures, even though it did not contain any divergent patterns and was, therefore, not edited.

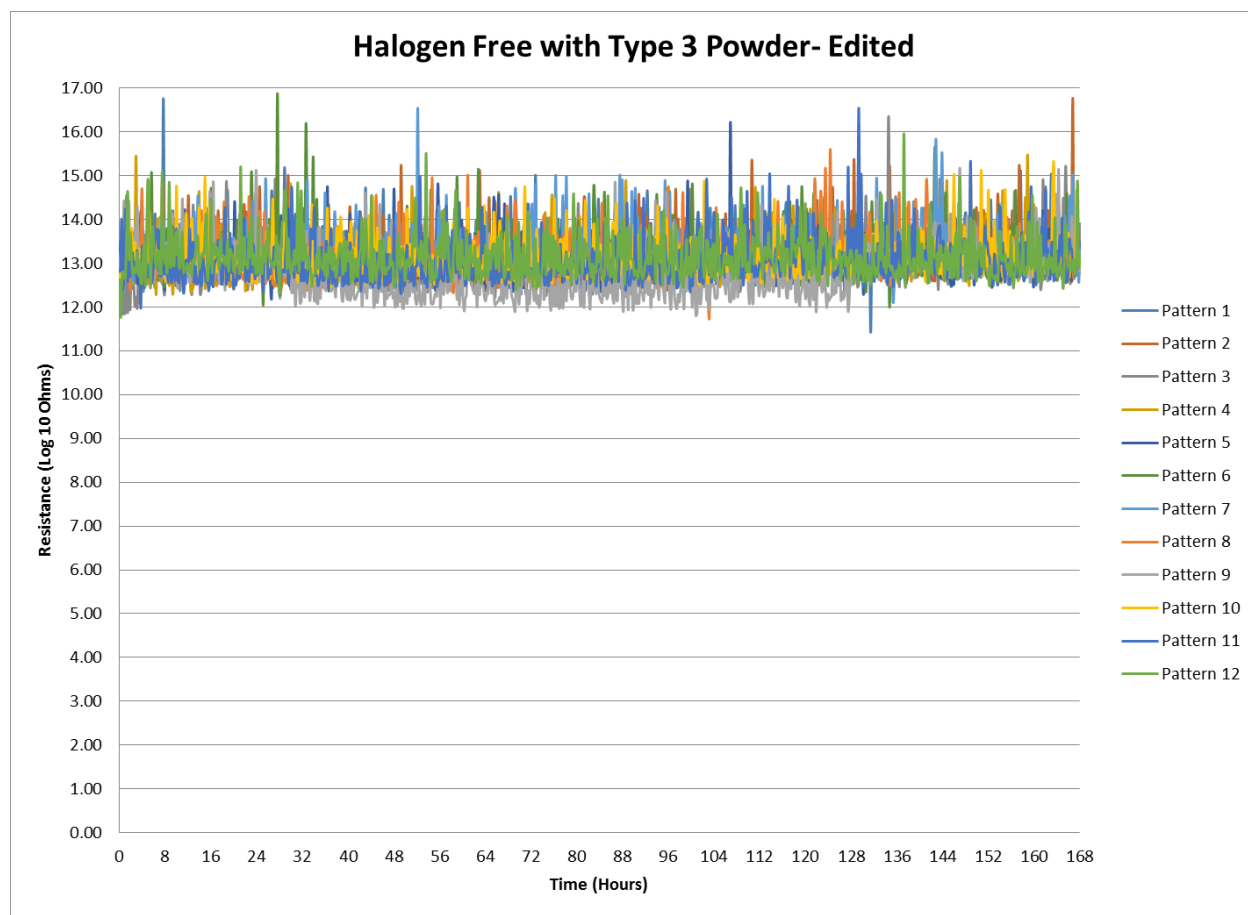


Figure 15 - Halogen-Free with Type 3 Powder – Edited

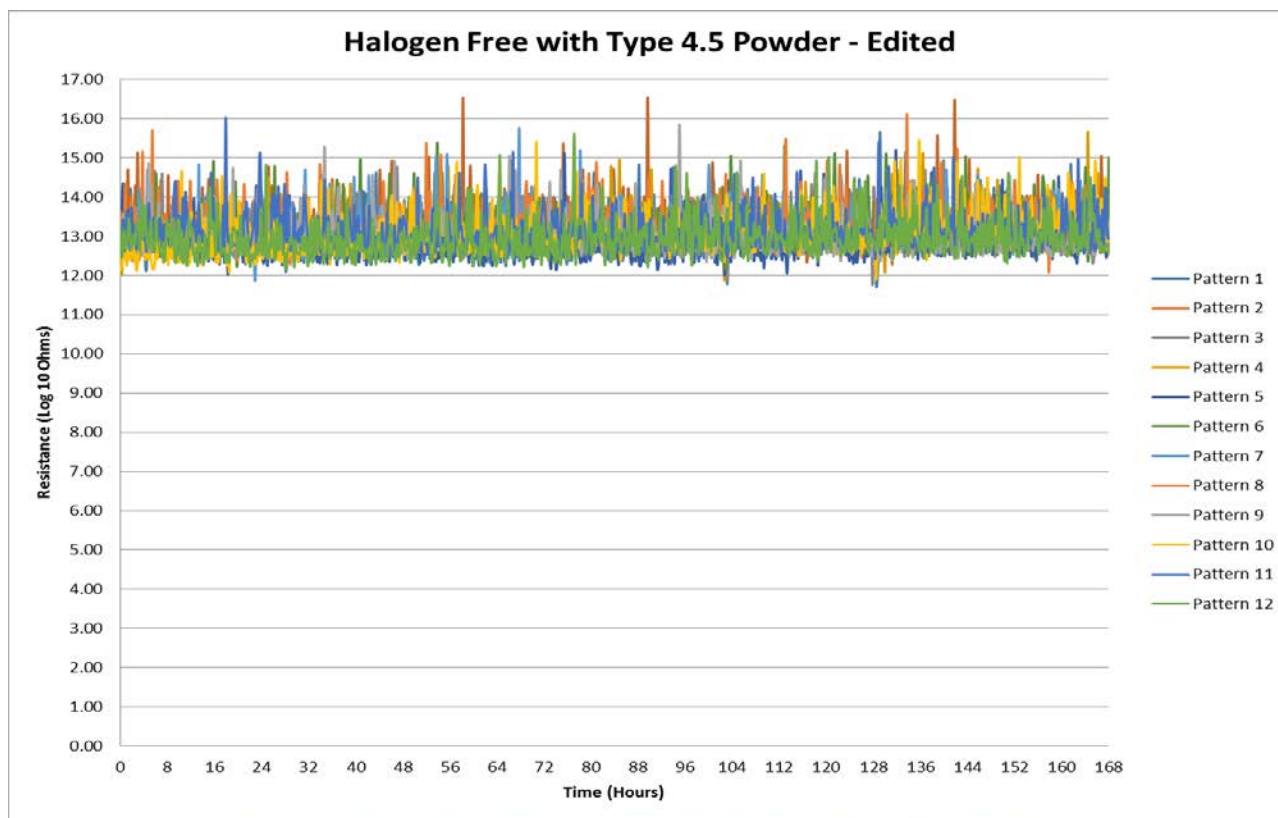


Figure 16 - Halogen-Free with Type 4.5 Powder - Edited

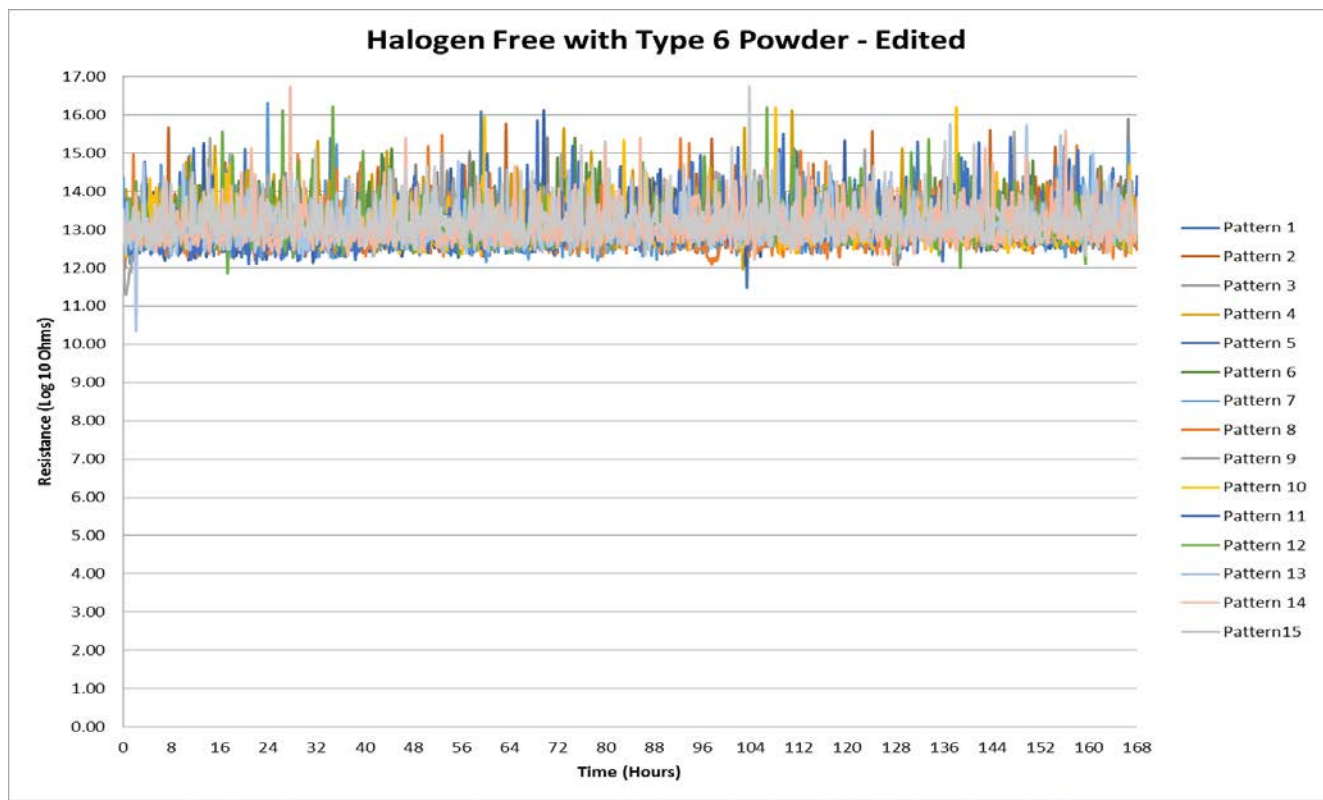


Figure 17 - Halogen-Free with Type 6 Powder – Edited

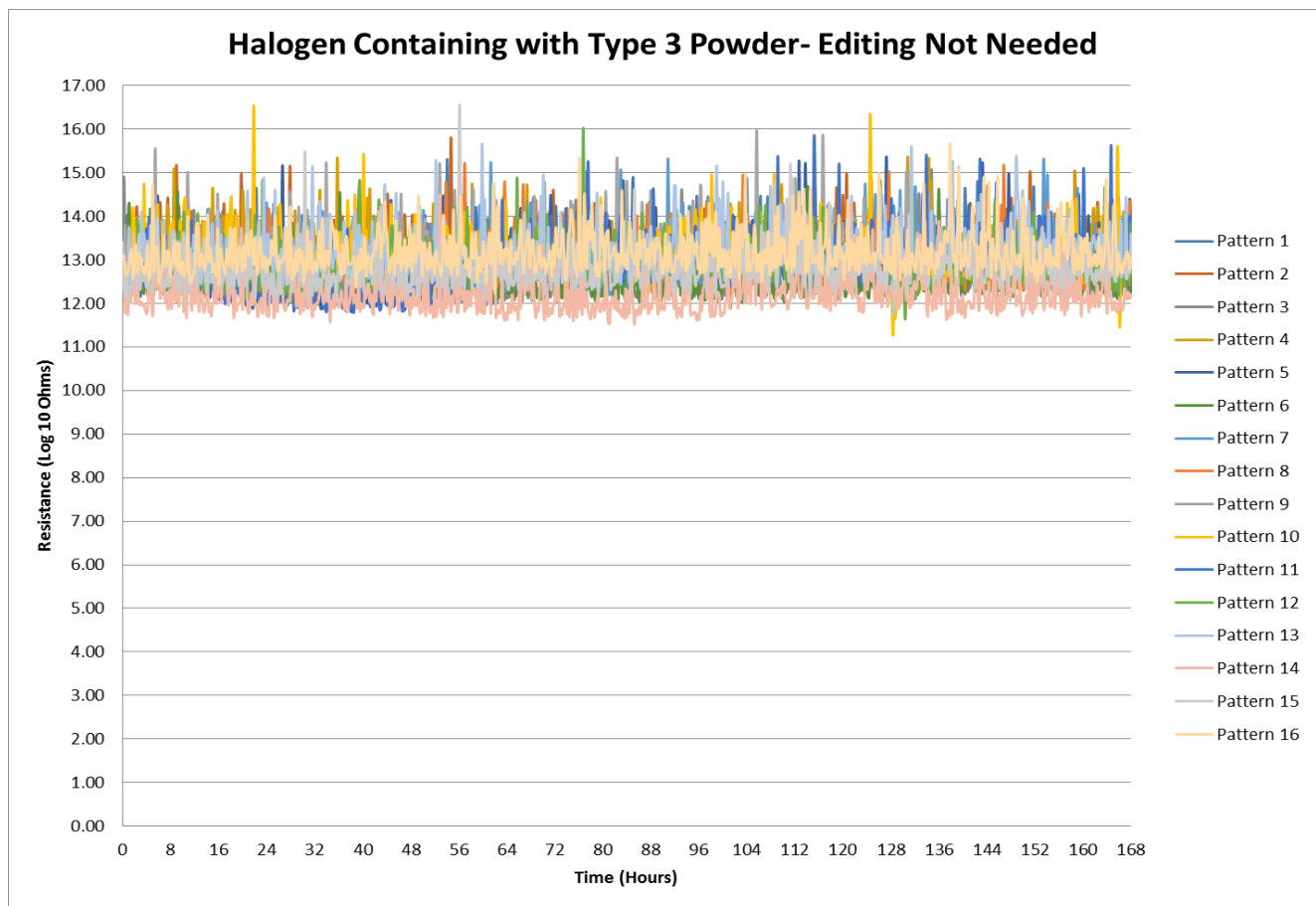


Figure 18 - Halogen-Containing with Type 3 Powder – Editing Not Needed



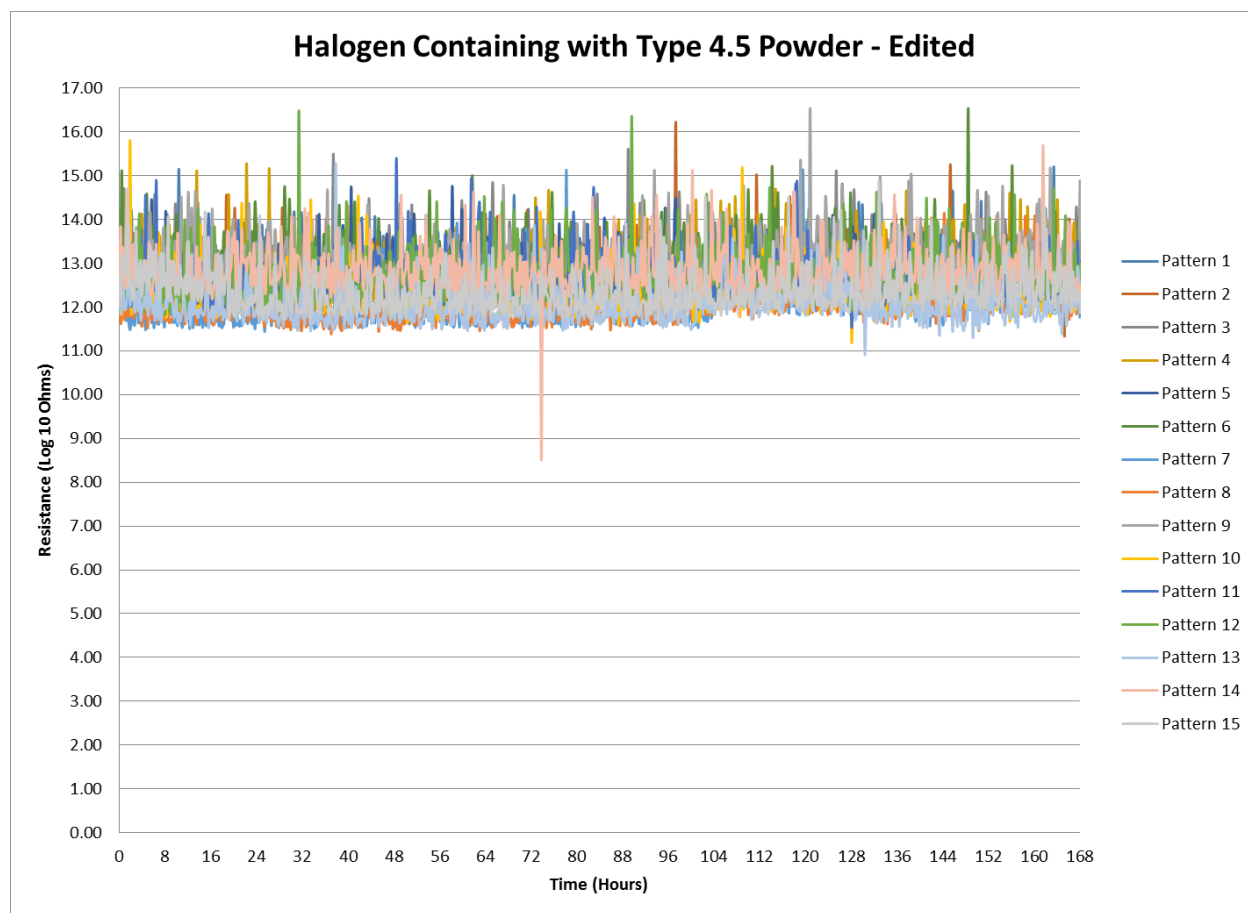


Figure 19 - Halogen-Containing with Type 4.5 Powder – Edited

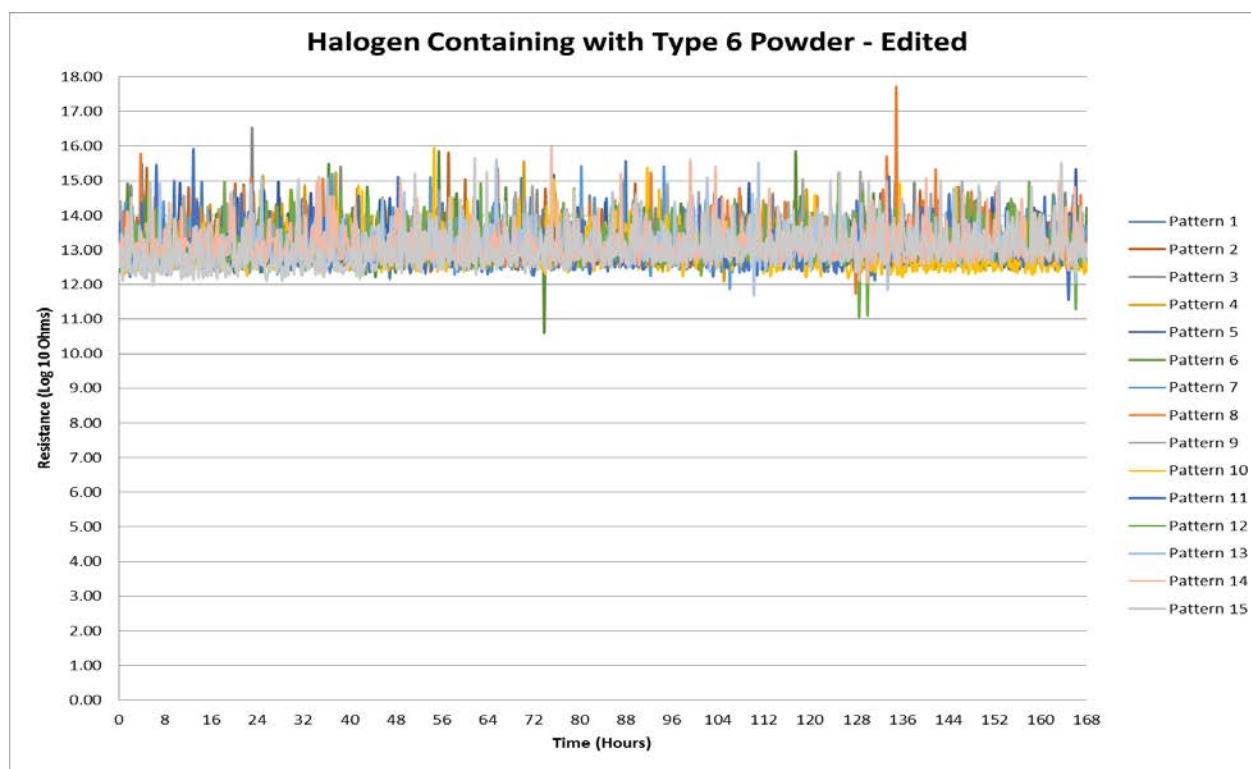
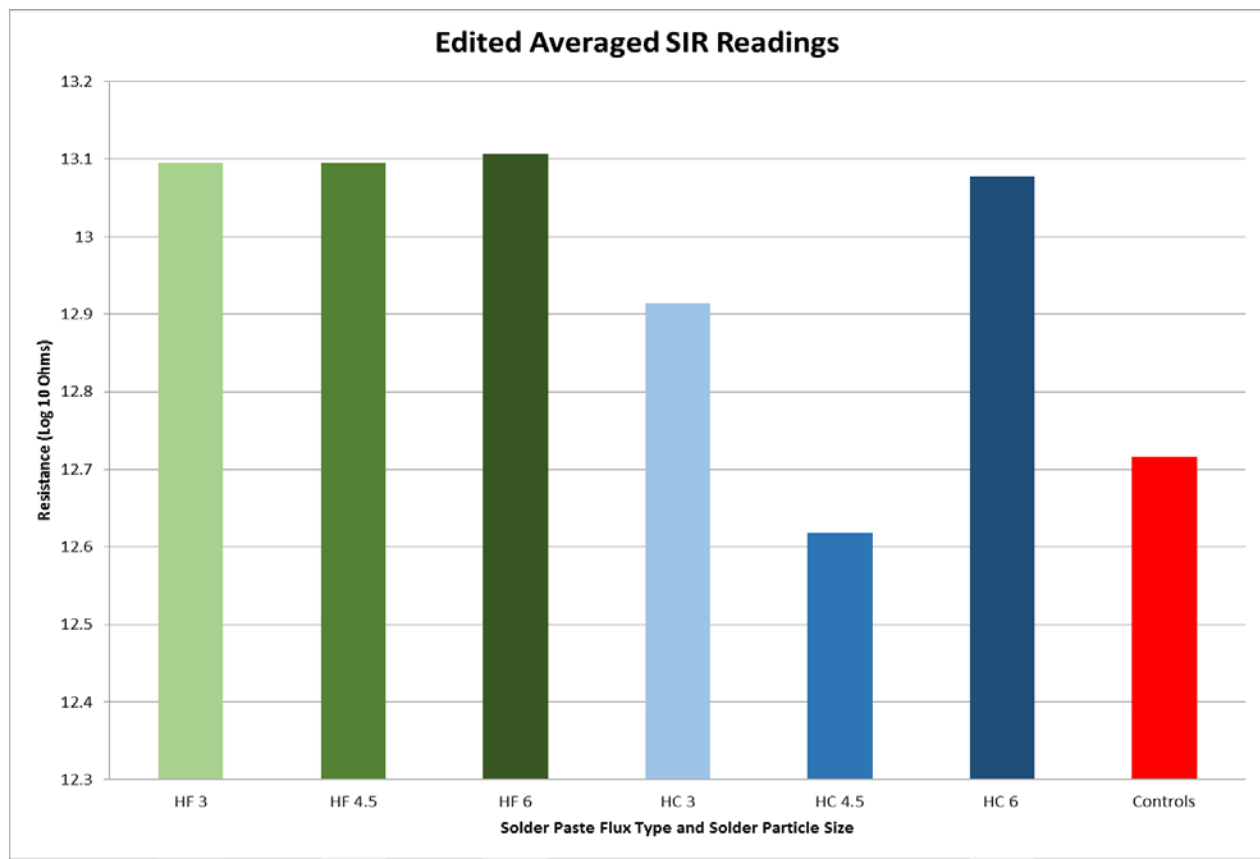


Figure 20 - Halogen-Containing with Type 6 Powder - Edited



**Figure 21 - Edited Averaged SIR Readings**

The averaged SIR readings that the edited data yields seems to indicate the halogen-free solder paste flux vehicle is quite immune to the solder powder particle size. The averaged readings for halogen-free Type 3, 4.5, and 6 are virtually identical. There is greater differentiation in the averaged SIR readings obtained from the halogen-containing solder paste scenarios. Nevertheless, considering the scale used for this graph, the values are still very similar. Furthermore, all scenarios, except for the halogen-containing with Type 4.5 powder, produced averaged SIR readings higher than that of the controls, and all scenarios produced averaged SIR values well above the 100MΩ minimum resistance value as stipulated by IPC J-STD-004B. Even with the piece of lint on the one pattern and the unedited data, all SIR readings for all scenarios stayed above the 100MΩ minimum resistance value.

## Conclusions

There is nothing in this data that would suggest that solder particle size has any significant measurable effect on the SIR performance of a no-clean solder paste. Also, there is nothing substantial in the data to suggest that one flux chemistry type is pointedly worse or better than another. The minor differences that may be present are not anything noteworthy enough to produce SIR values low enough to be of any concern. Five of the six scenarios produced better average SIR values than the controls.

Unfortunately, there are a number of possible confounding factors that may overpower any subtle differences that may truly be present. They include, but may not be limited to, possible variation in the amount of oxidation present on the various solder powders and copper traces on the SIR boards, and intentional variation in the metal loading.

## Acknowledgements

The author would like to thank the following people for their assistance in preparing the SIR test boards:

Sarah Bjornland – Summer intern from the University of Rochester, Rochester, NY

Mark Reece – Summer intern from SUNY Polytechnic Institute, Utica, NY

## References

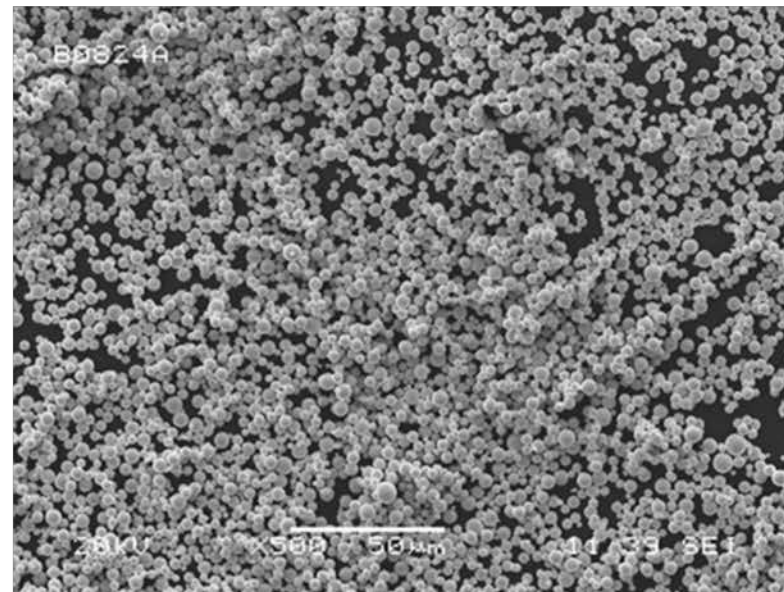
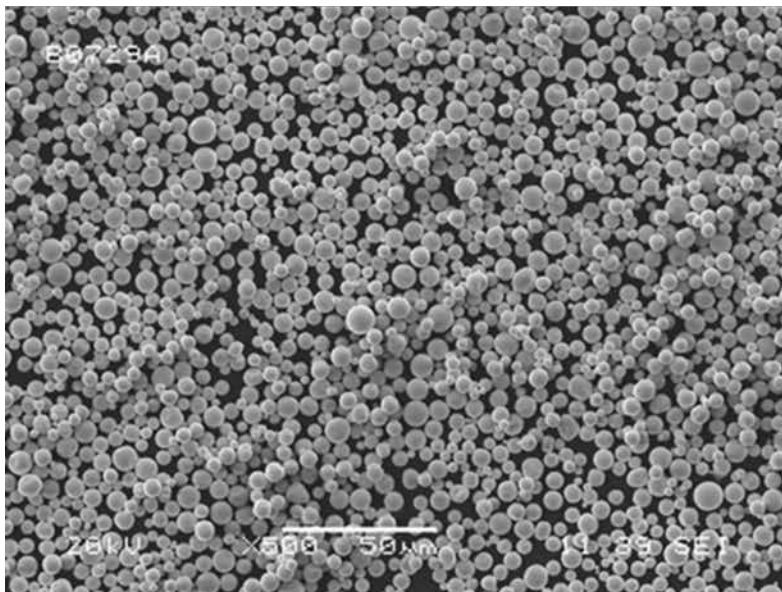
1. E. Bastow, "The Effect of Reflow Profiling on the Electrical Reliability of No-Clean Solder Paste Flux Residues", IPC APEX 2014 conference.

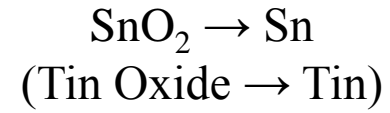


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Type	Particle Size Distribution (microns)	Mean Particle Size (microns)	Mean Particle Size Surface Area Ratio
3	25 to 45	35	1.00
4	20 to 38	29	1.21
4.5	20 to 32	26	1.35
5	15 to 25	20	1.75
6	5 to 15	10	3.50





Larger Particle Size

Reduces Surface Area

Consumes Less  
Activator

Less Act/MO By-  
product in Residue

More Leftover Act in  
Residue

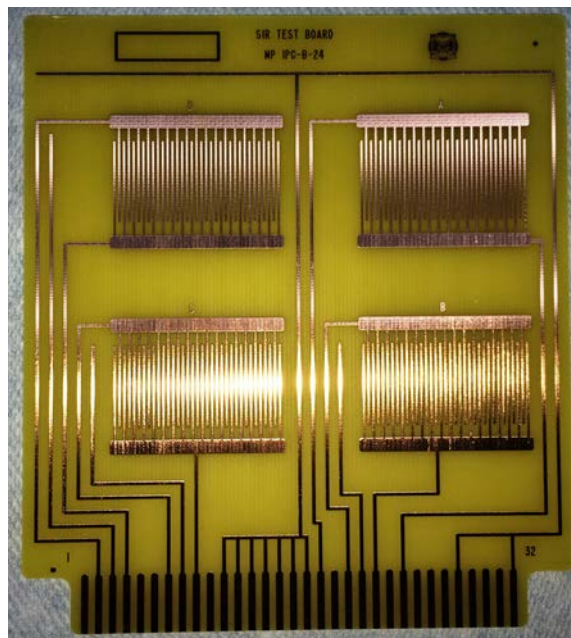
Smaller Particle Size

Increases Surface  
Area

Consumes More  
Activator

More Act/MO By-  
product in Residue

Less Leftover Act in  
Residue



			Solder Paste Type		
Powder Type	Particle Size Distribution	Mesh Size	Halogen Free (ROL0)	Halogenated (ROL1)	
Type 3	25 - 45 microns	325/500	4 Boards	4 Boards	
Type 4.5	20 - 32 microns	450/635	4 Boards	4 Boards	
Type 6	5 - 15 microns	N/A	4 Boards	4 Boards	
IPC-B-24 SIR Test Board					2 Boards Controls

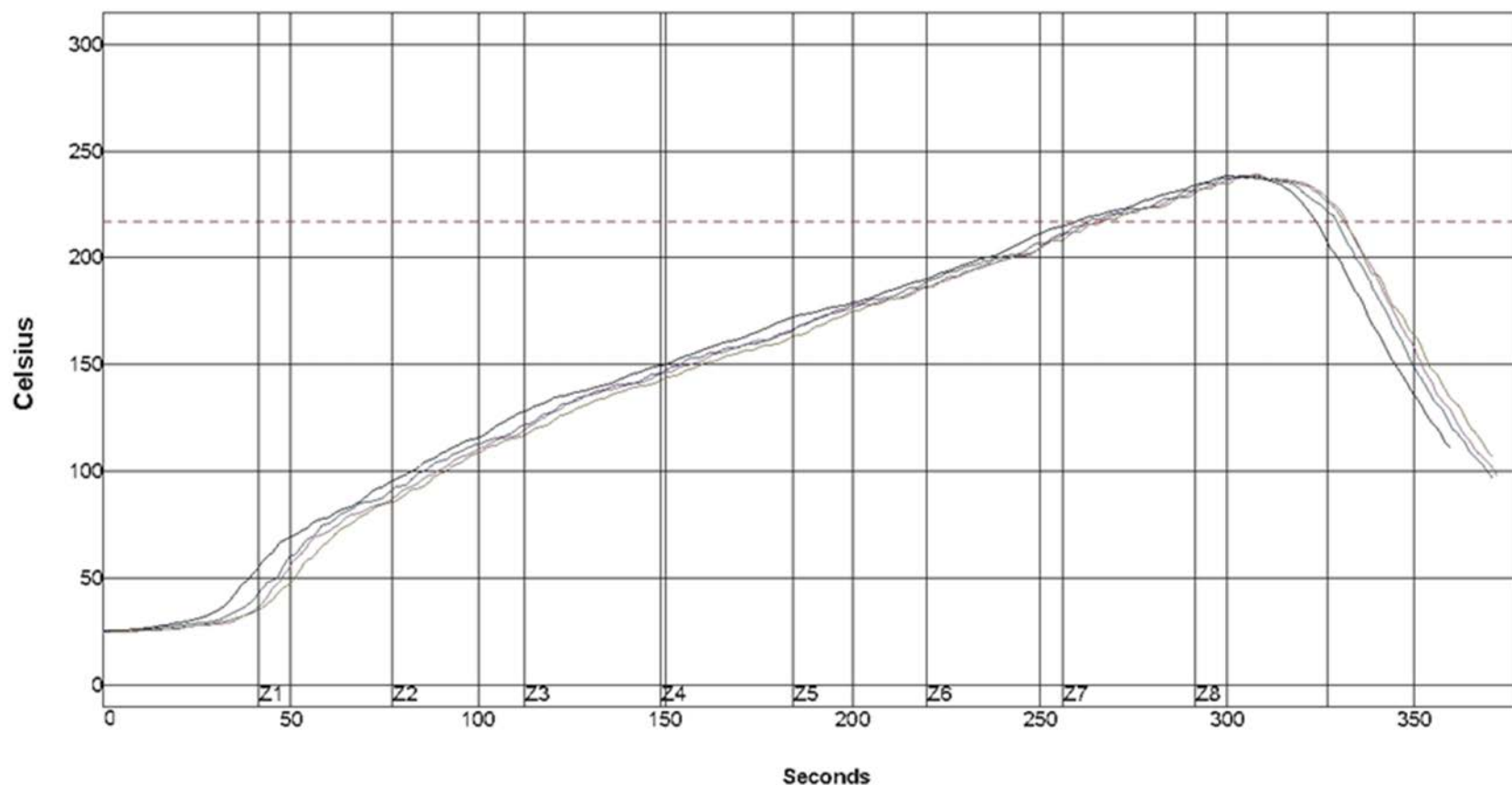
Powder Type	Metal Load per Solder Paste Type	
	Halogen Free (ROL0)	Halogenated (ROL1)
Type 3	89%	88.75%
Type 4.5	88.50%	88.50%
Type 6	88%	88%



**Setpoints (Celsius)**

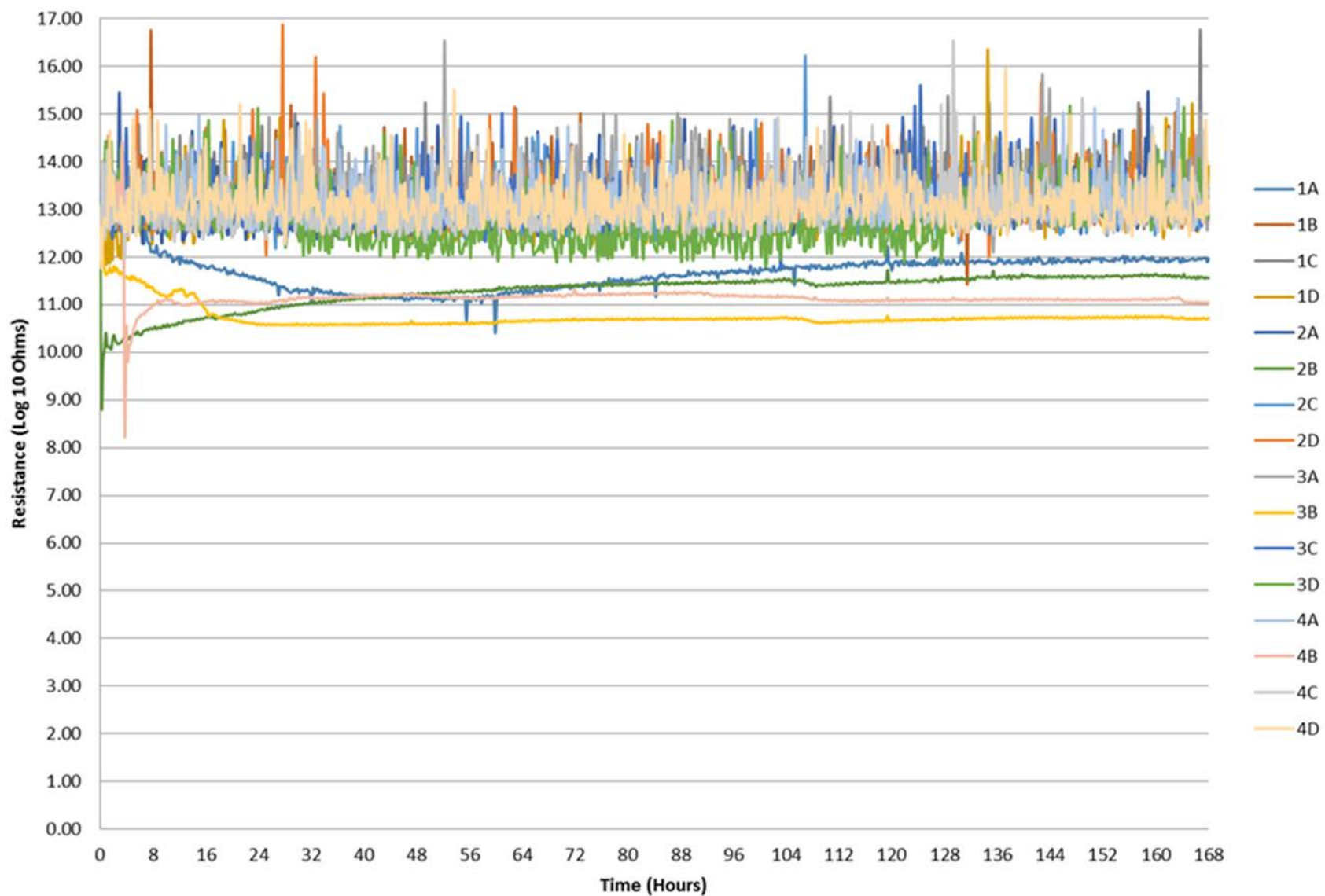
Zone	1	2	3	4	5	6	7	8
Top	100	125	150	170	190	210	235	255
Bottom	100	125	150	170	190	210	235	255

Conveyor Speed ( inch/min ): 22.0



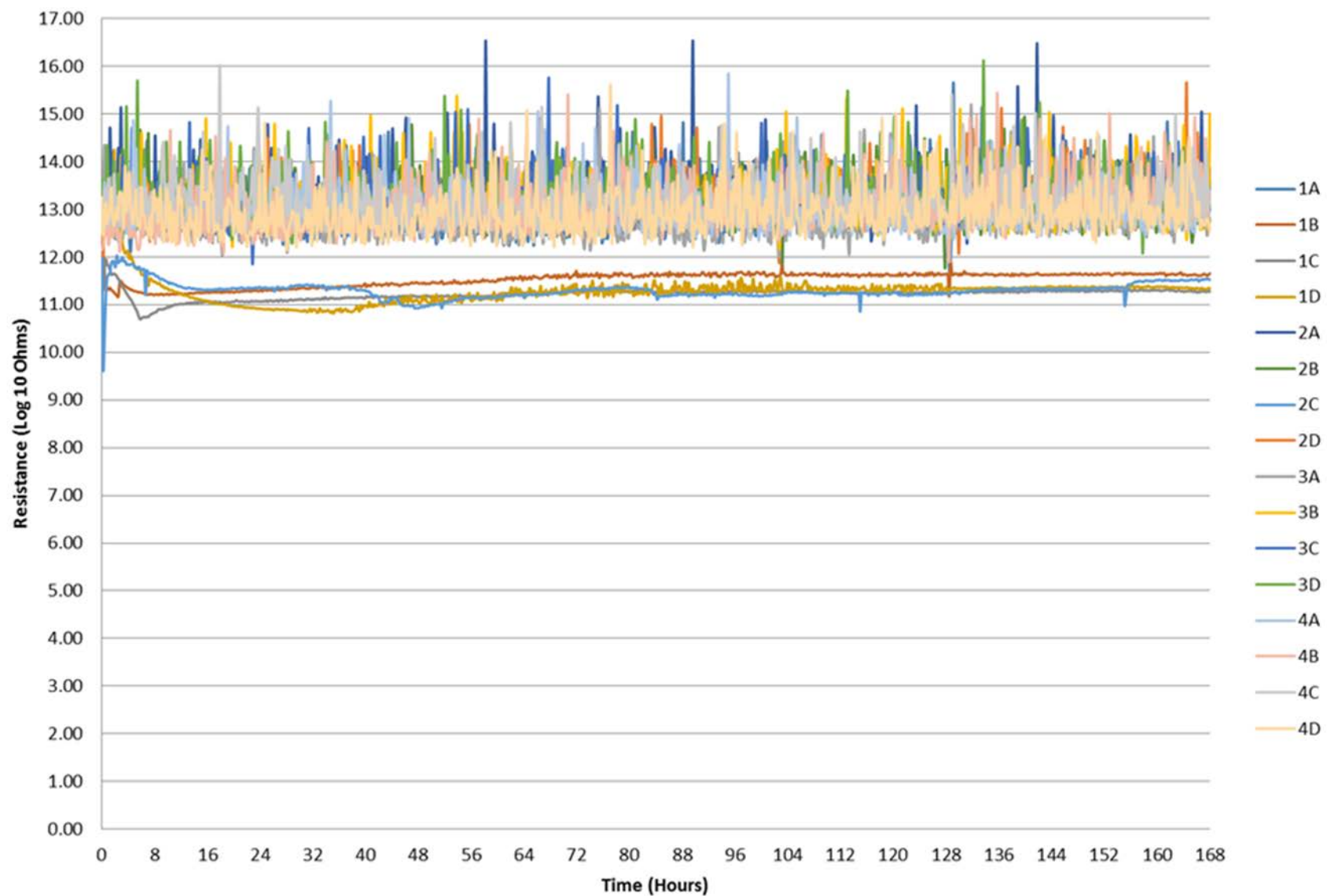
PWt= 53%	Max Rising Slope		Max Falling Slope		Reflow Time /217C		Peak Temp		Slope1 ( 25-217C)	
2	1.88	-6%	-3.31	-16%	66.32	-5%	239.23	-44%	1.00	-2%
3	1.86	-7%	-3.29	-15%	66.34	-5%	238.76	-50%	0.98	-7%
4	1.77	-12%	-3.00	-0%	64.39	-14%	238.82	-49%	0.97	-13%
5	1.75	-13%	-3.18	-9%	64.06	-15%	238.50	-53%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

## Halogen Free with Type 3 Powder

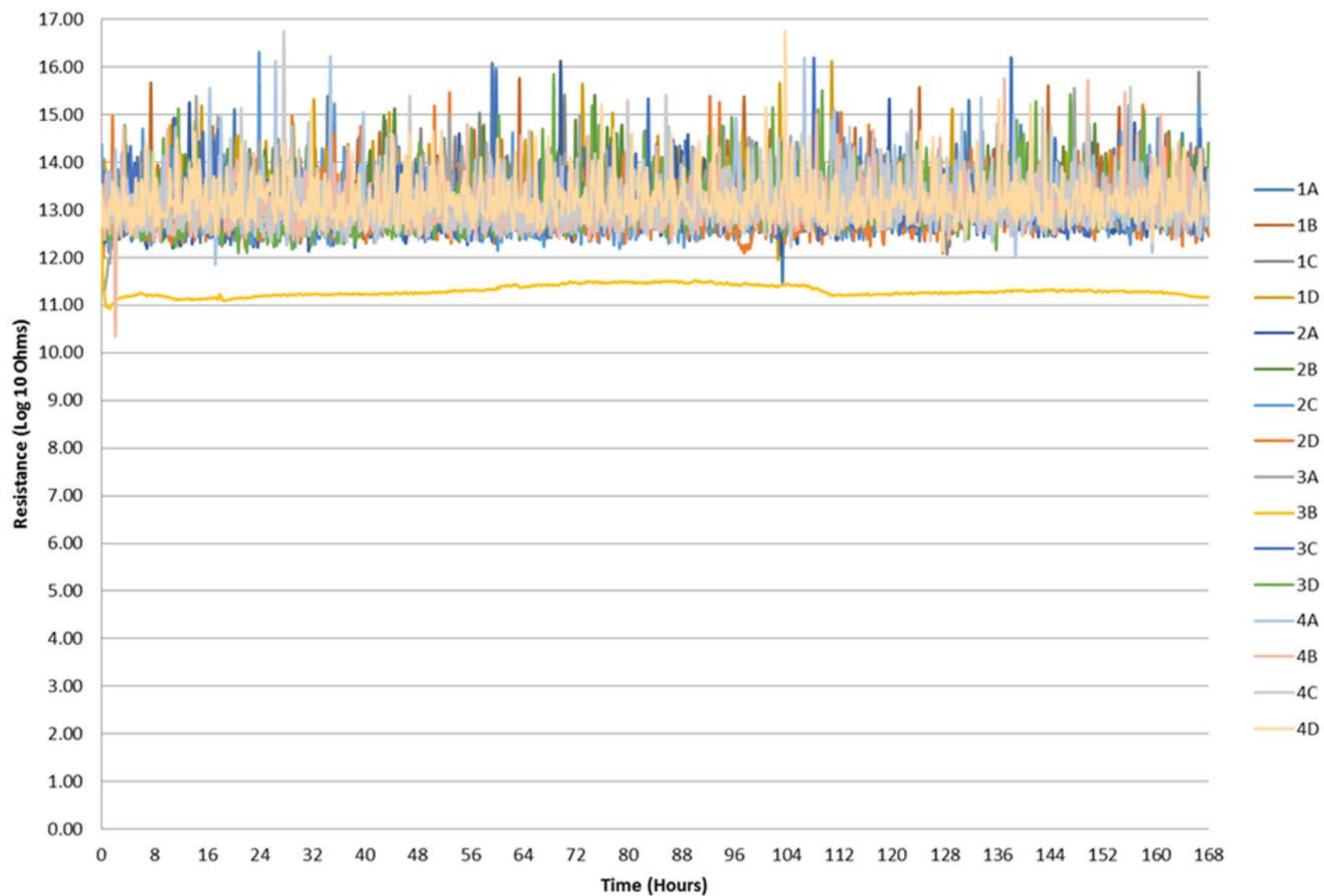




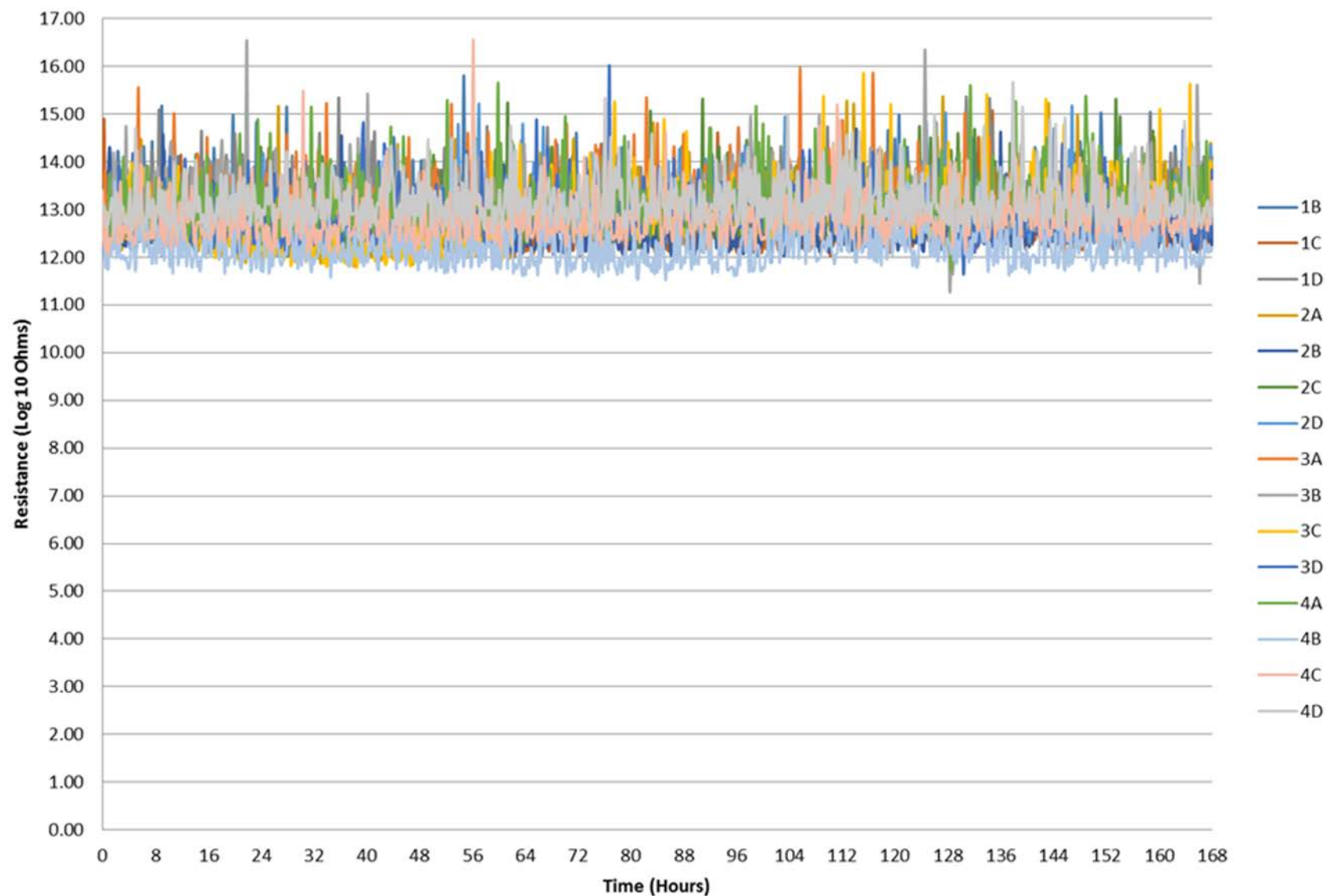
## Halogen Free with Type 4.5 Powder



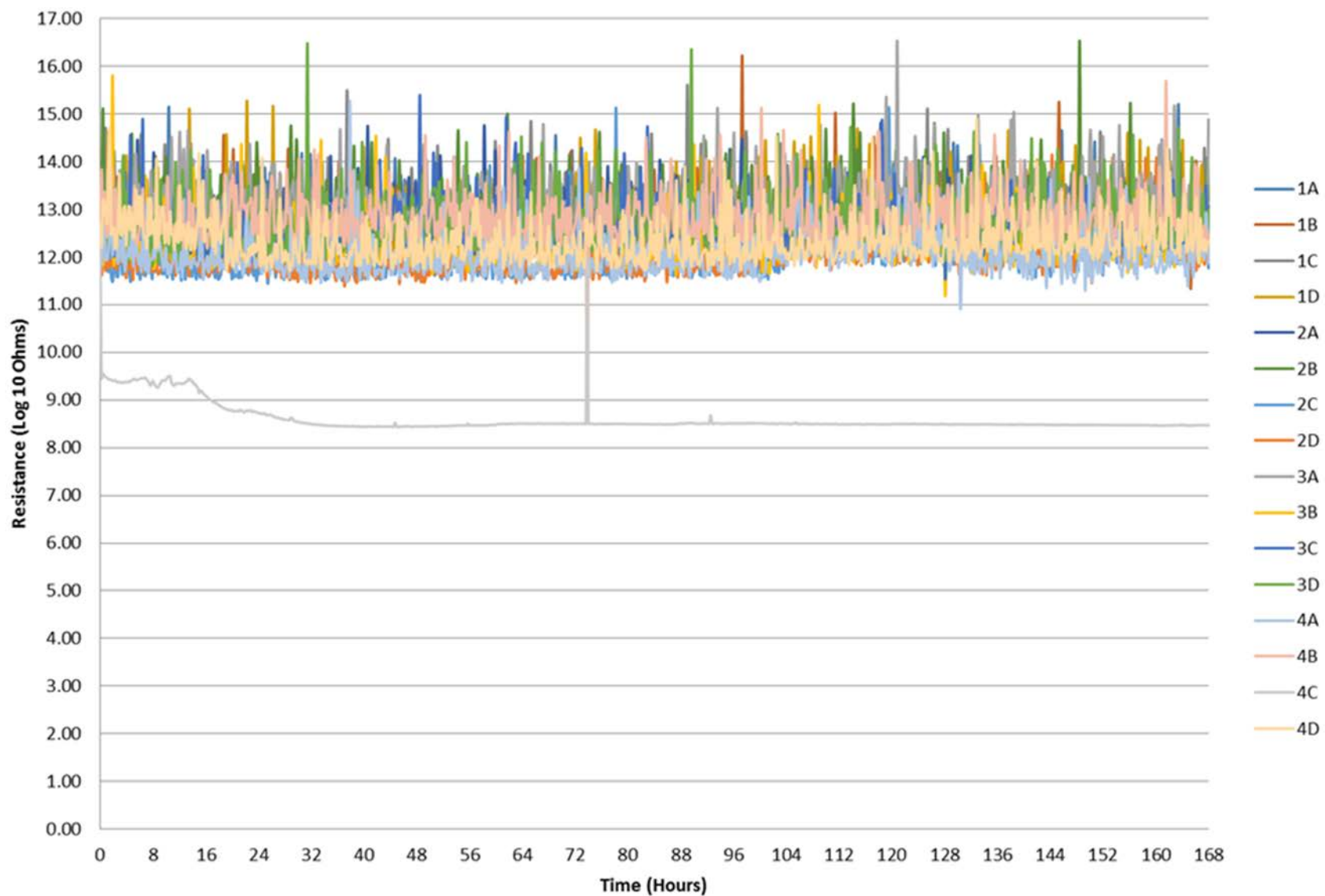
## Halogen Free with Type 6 Powder



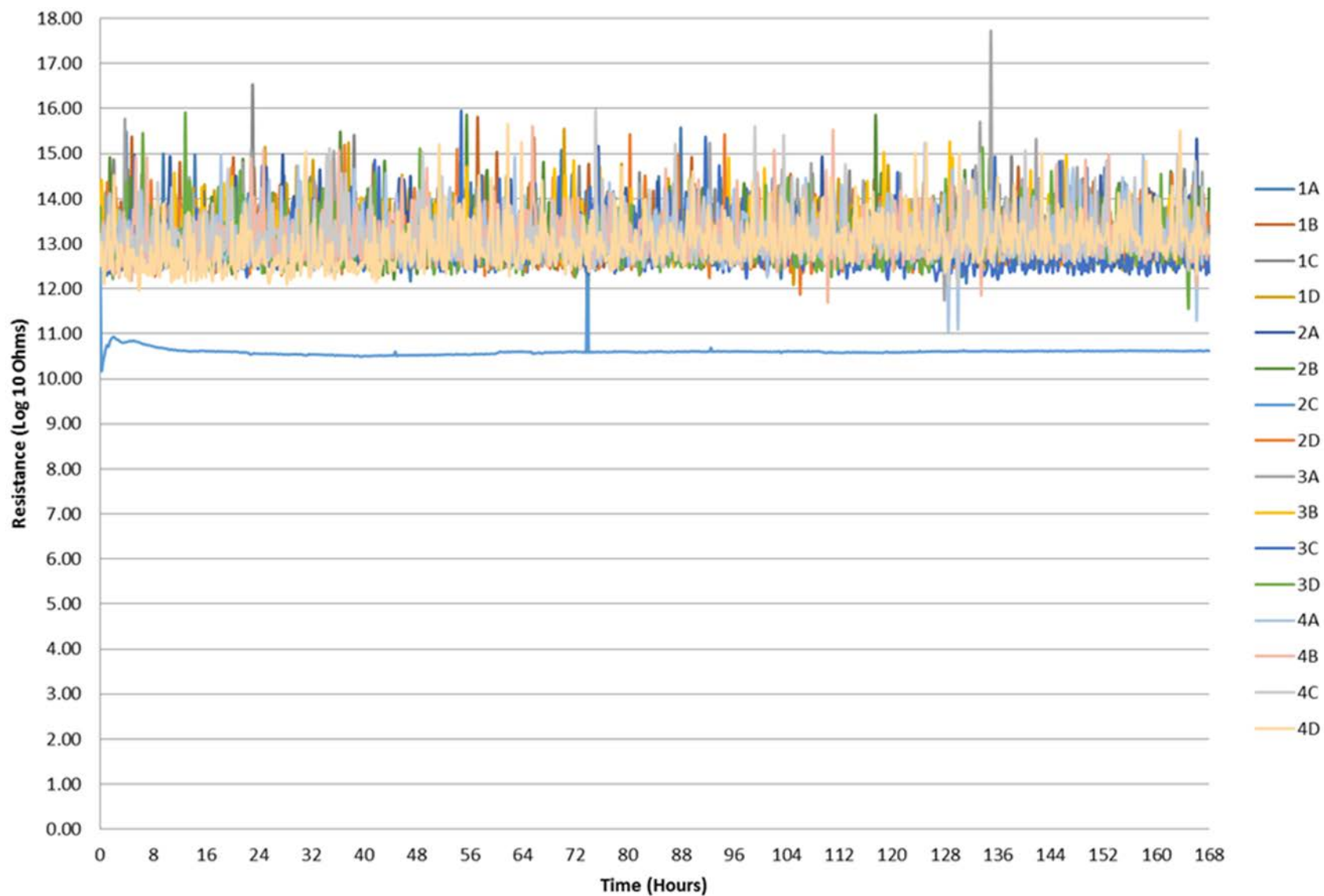
### Halogen Containing with Type 3 Powder



## Halogen Containing with Type 4.5 Powder

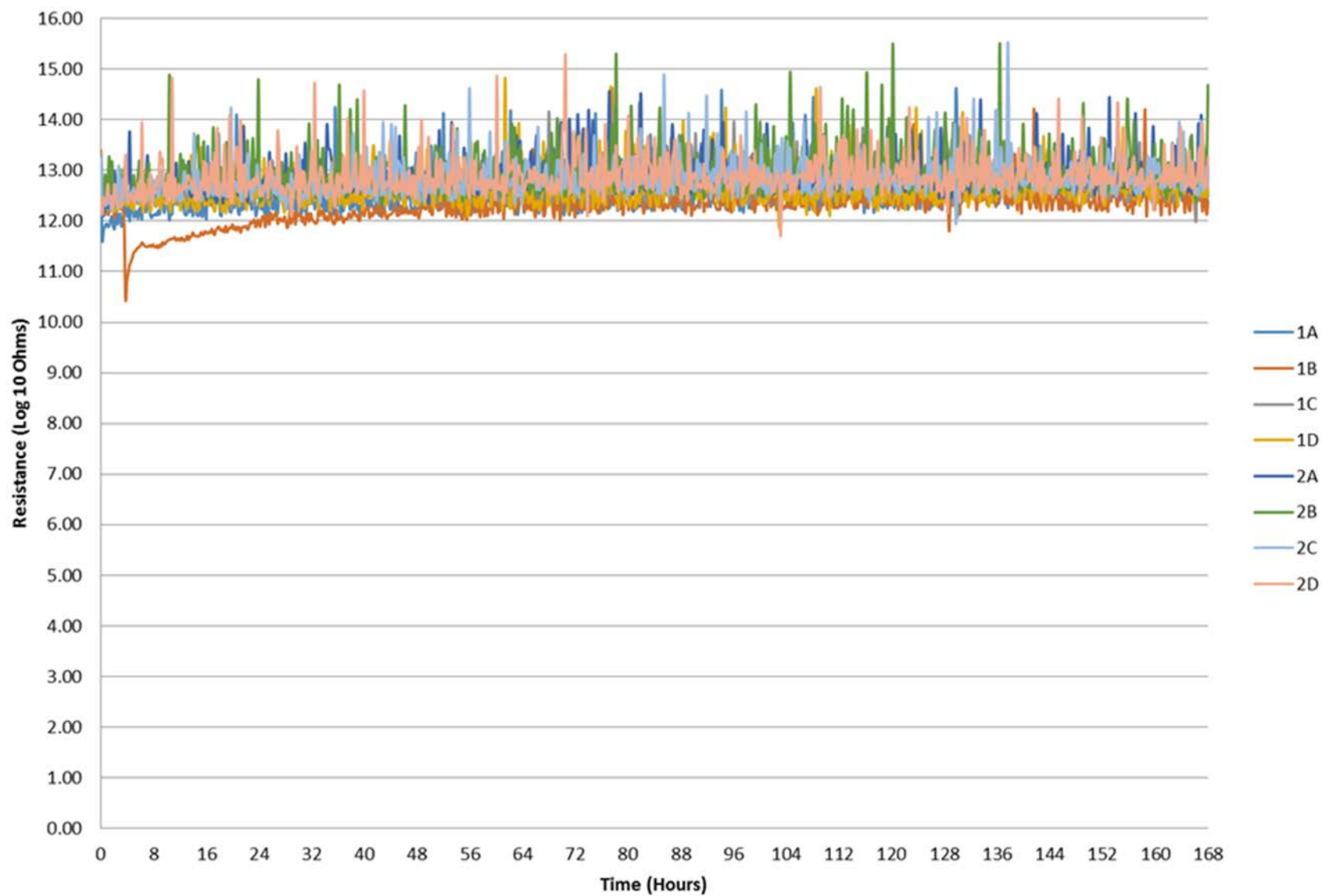


## Halogen Containing with Type 6 Powder

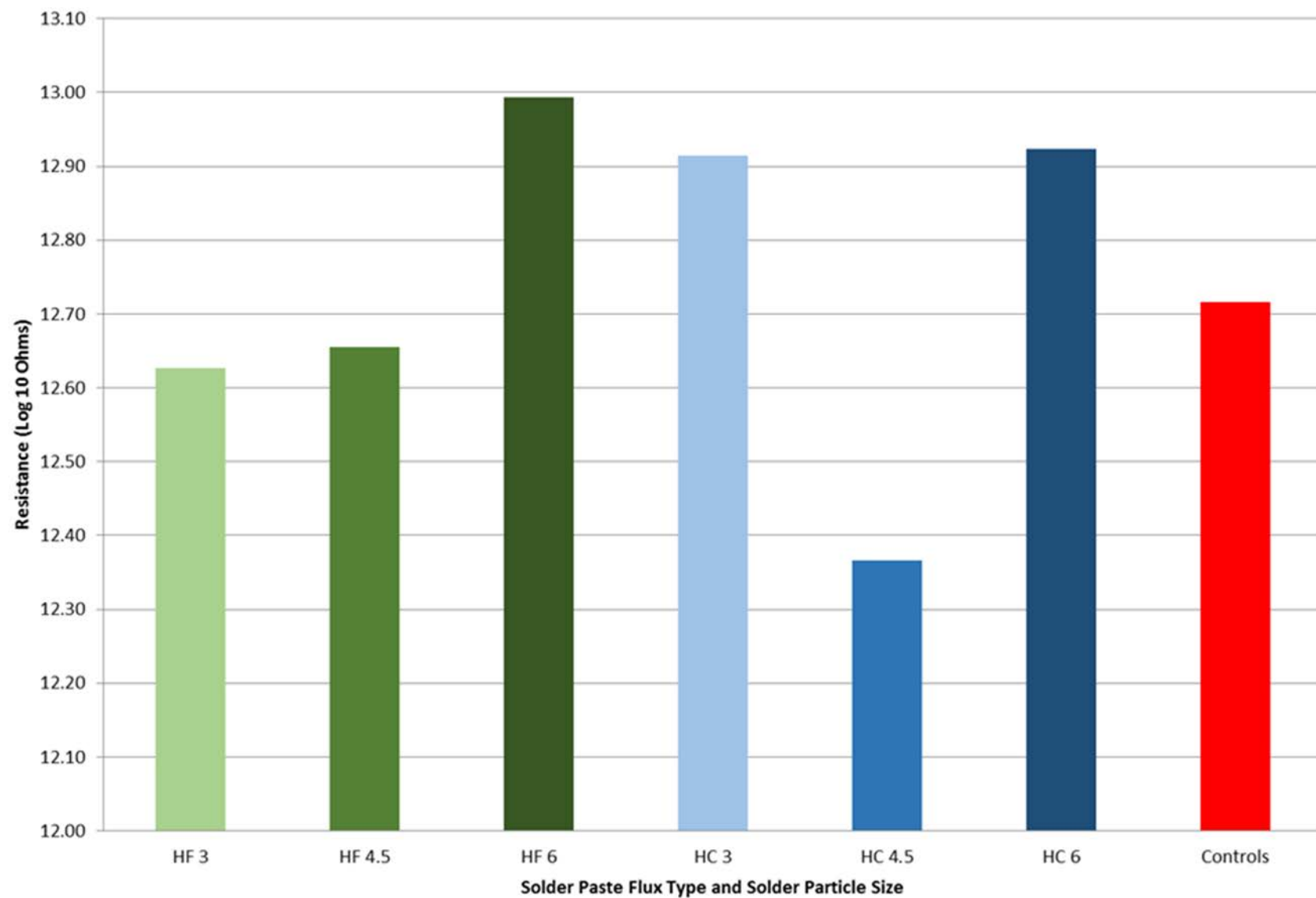


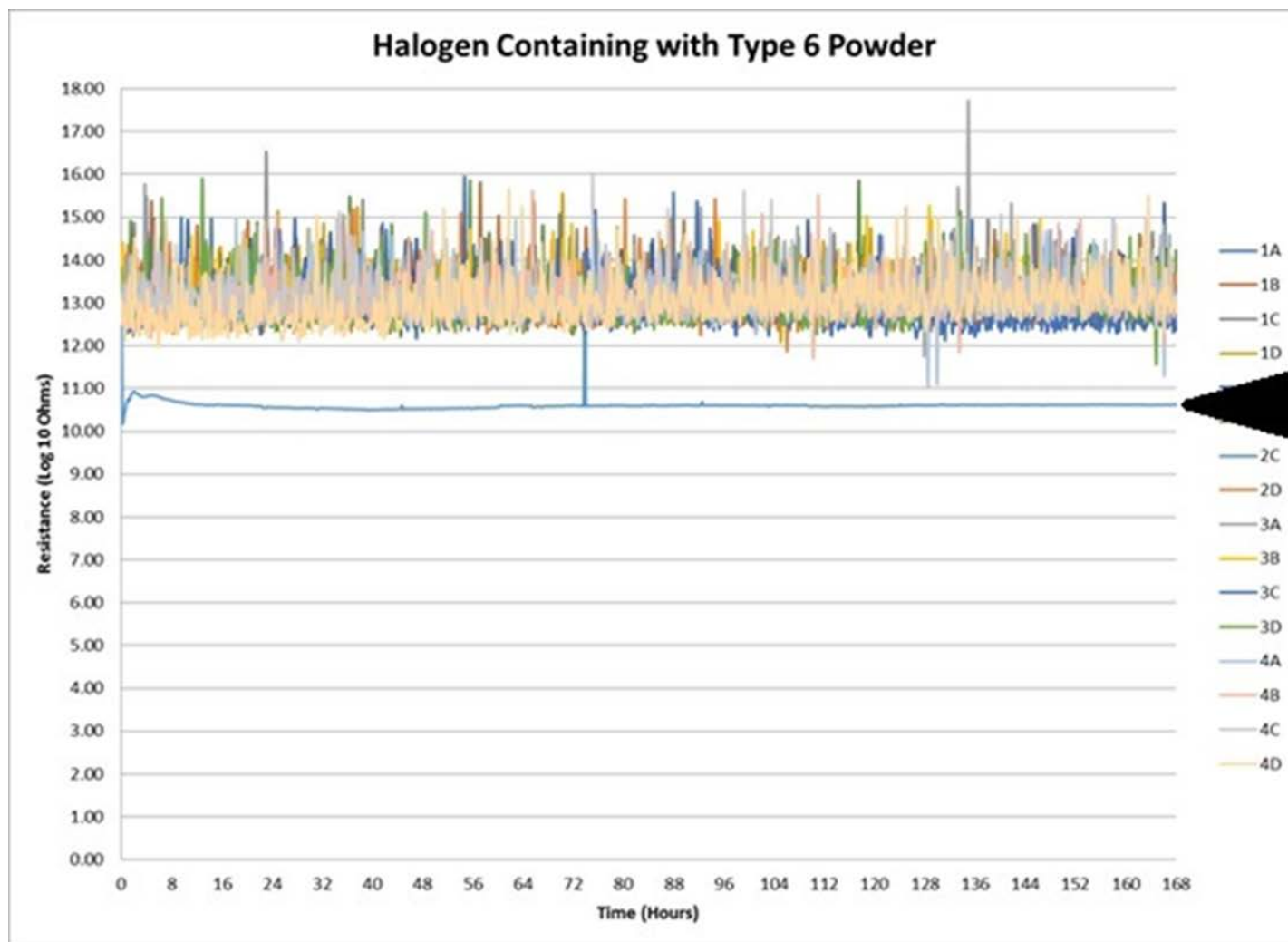


## Controls



### Unedited Averaged SIR Readings





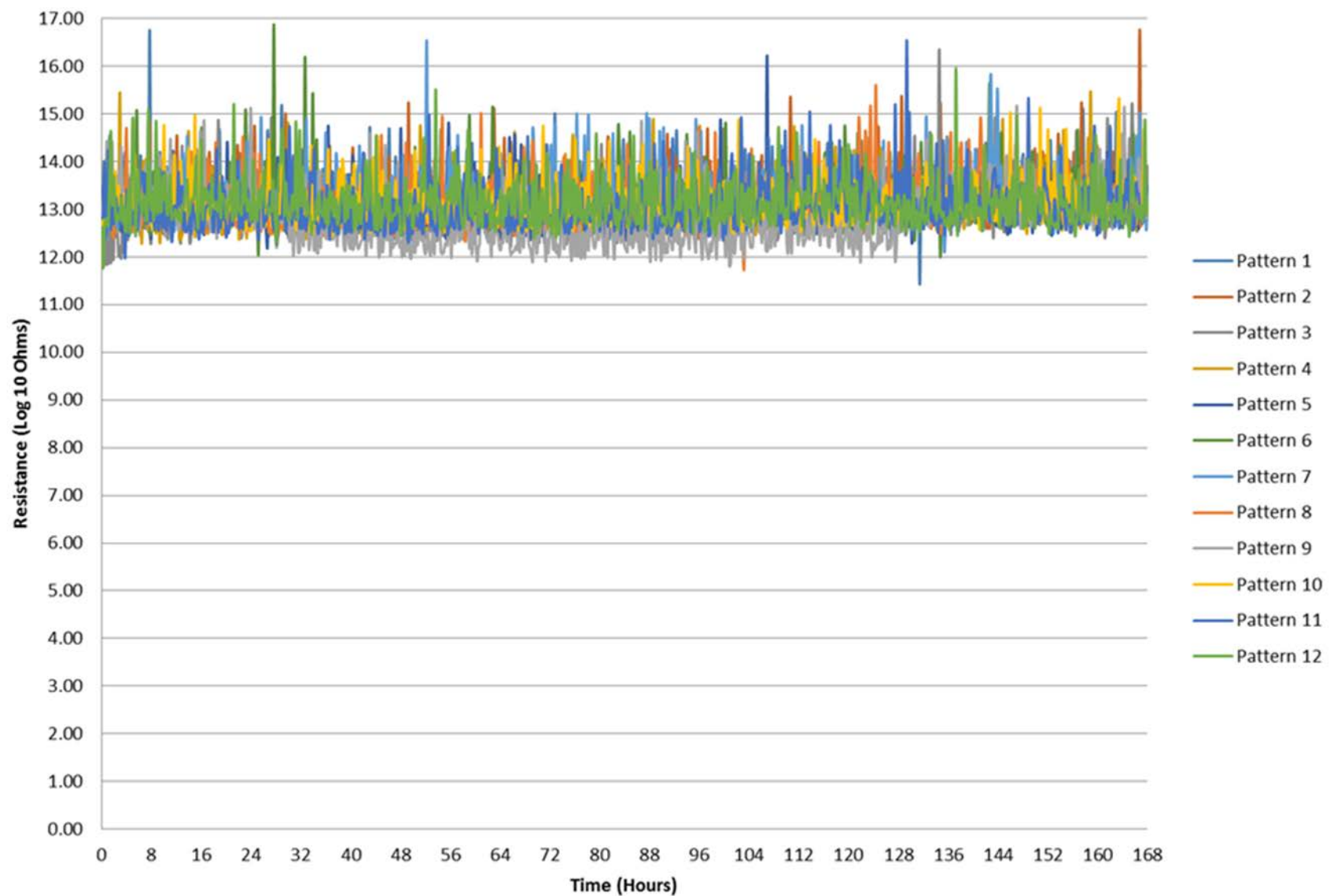




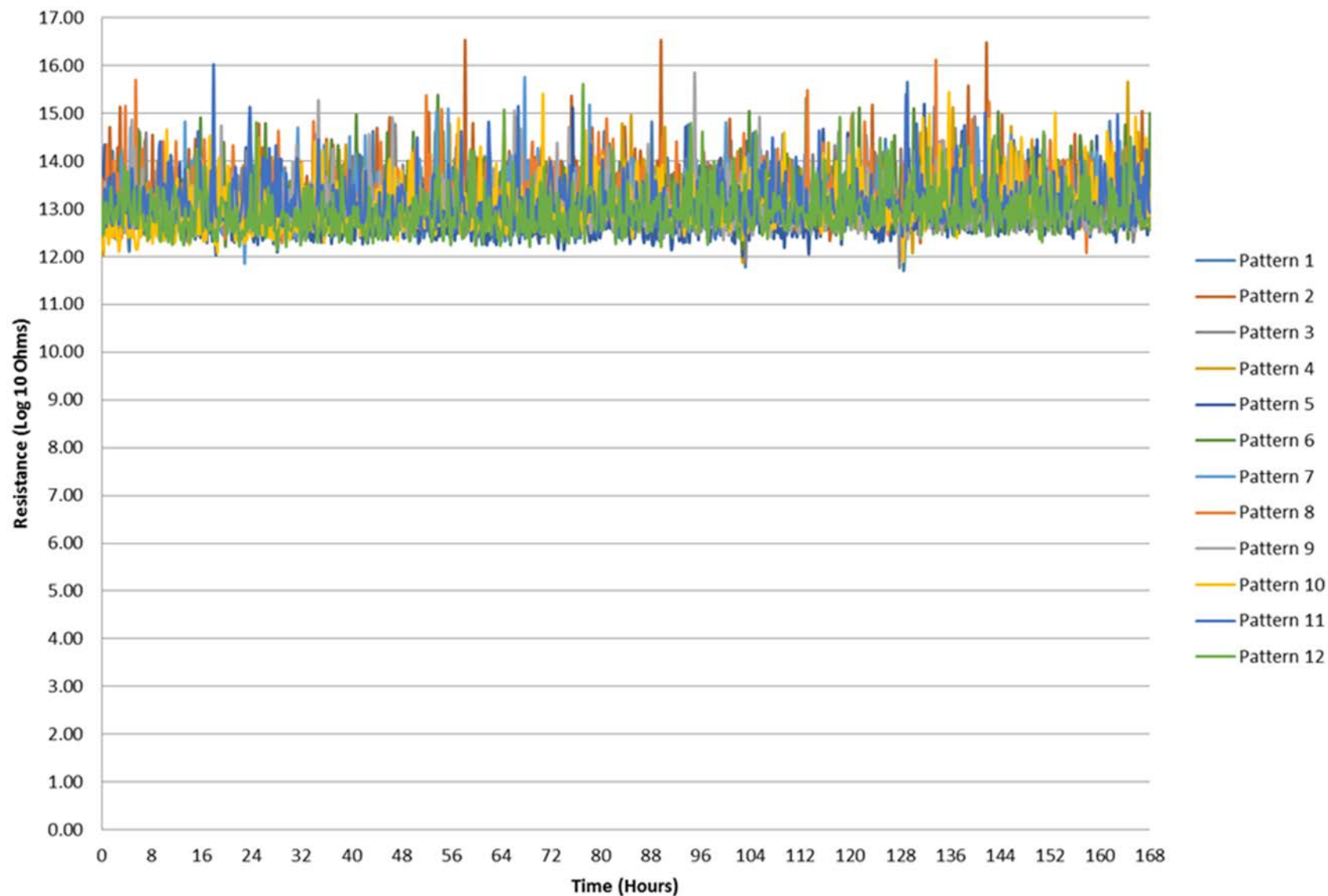
Halogen Containing with Type 4.5 Powder

PATTERN 4C LINT

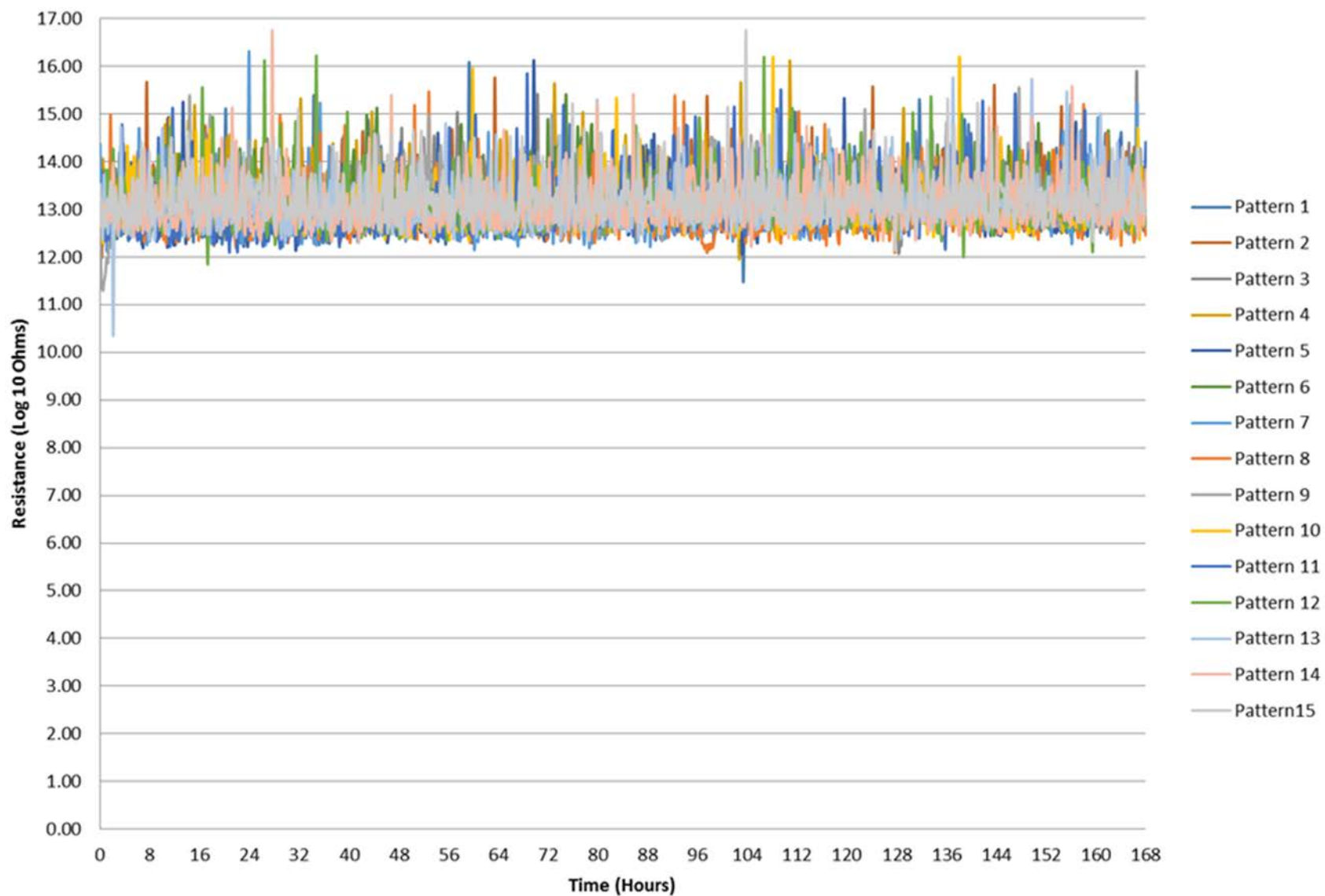
### Halogen Free with Type 3 Powder- Edited



### Halogen Free with Type 4.5 Powder - Edited

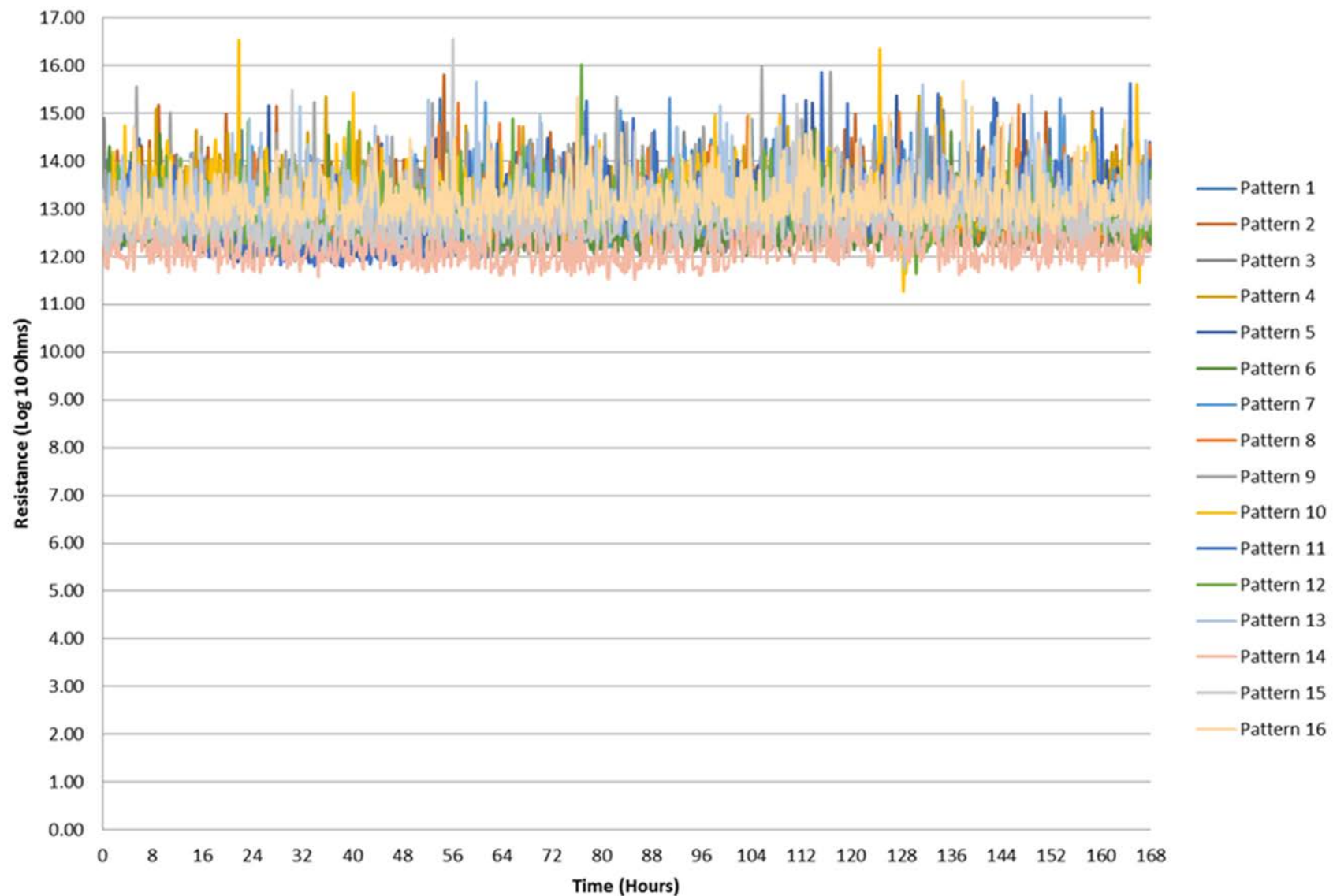


### Halogen Free with Type 6 Powder - Edited

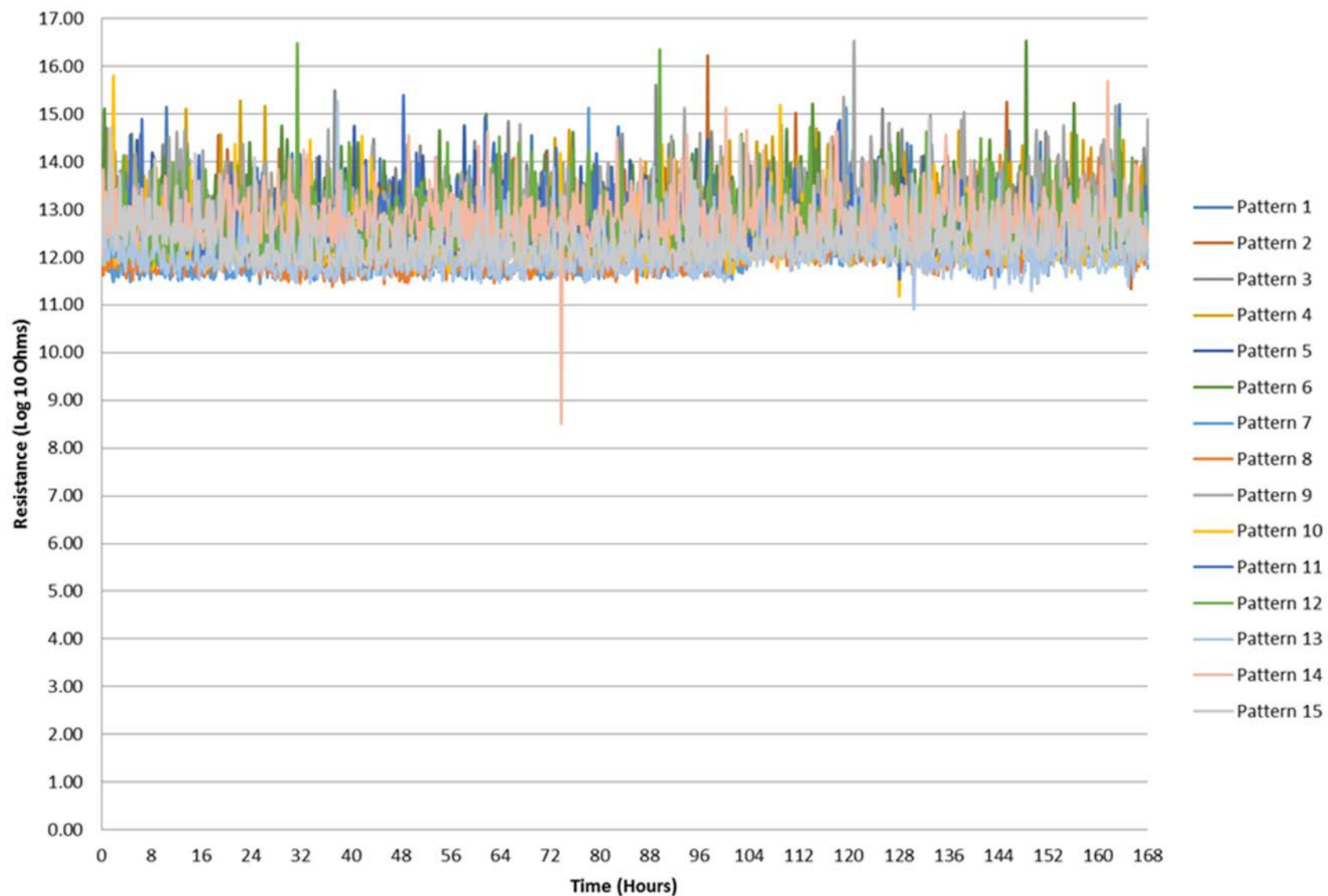




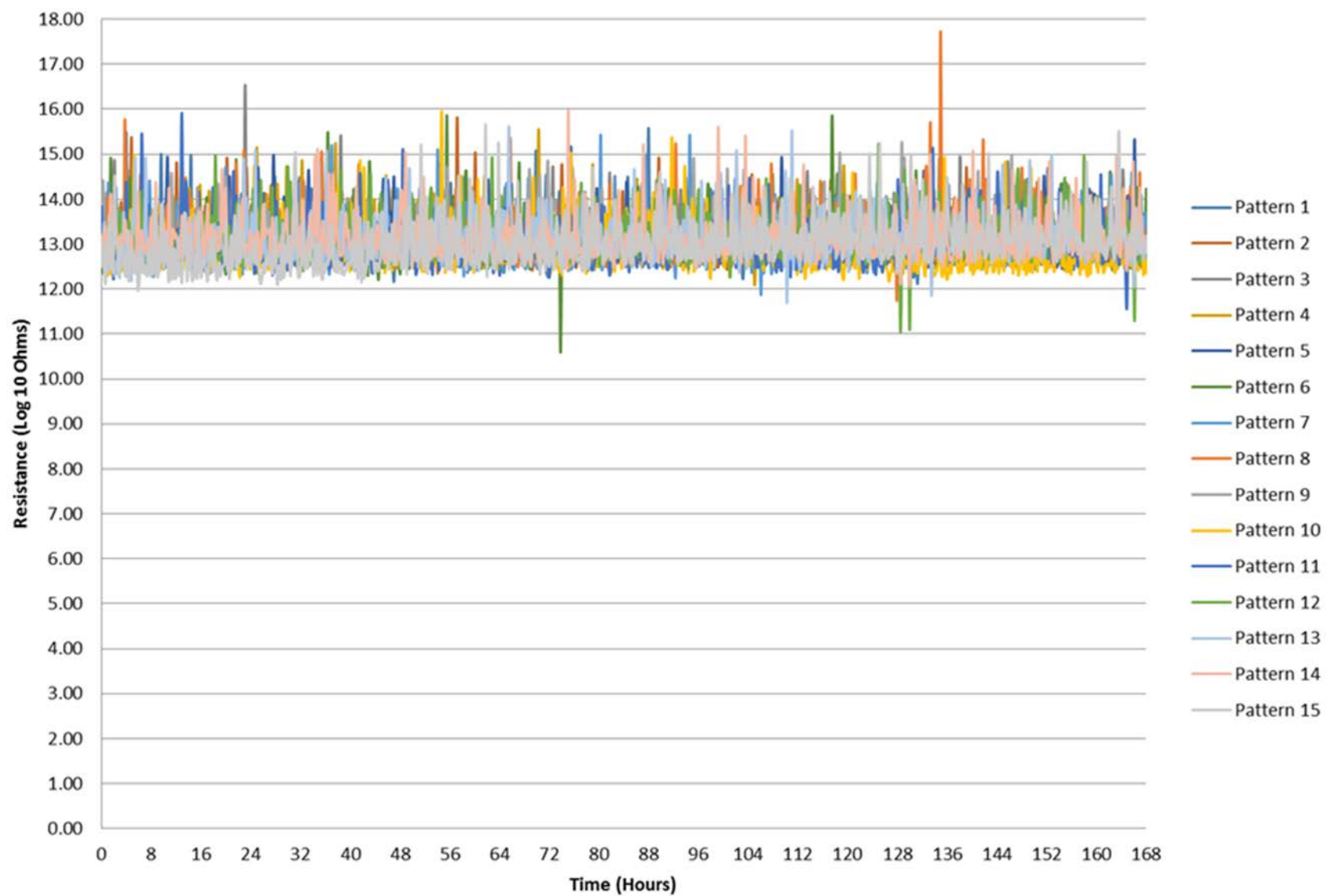
## Halogen Containing with Type 3 Powder- Editing Not Needed

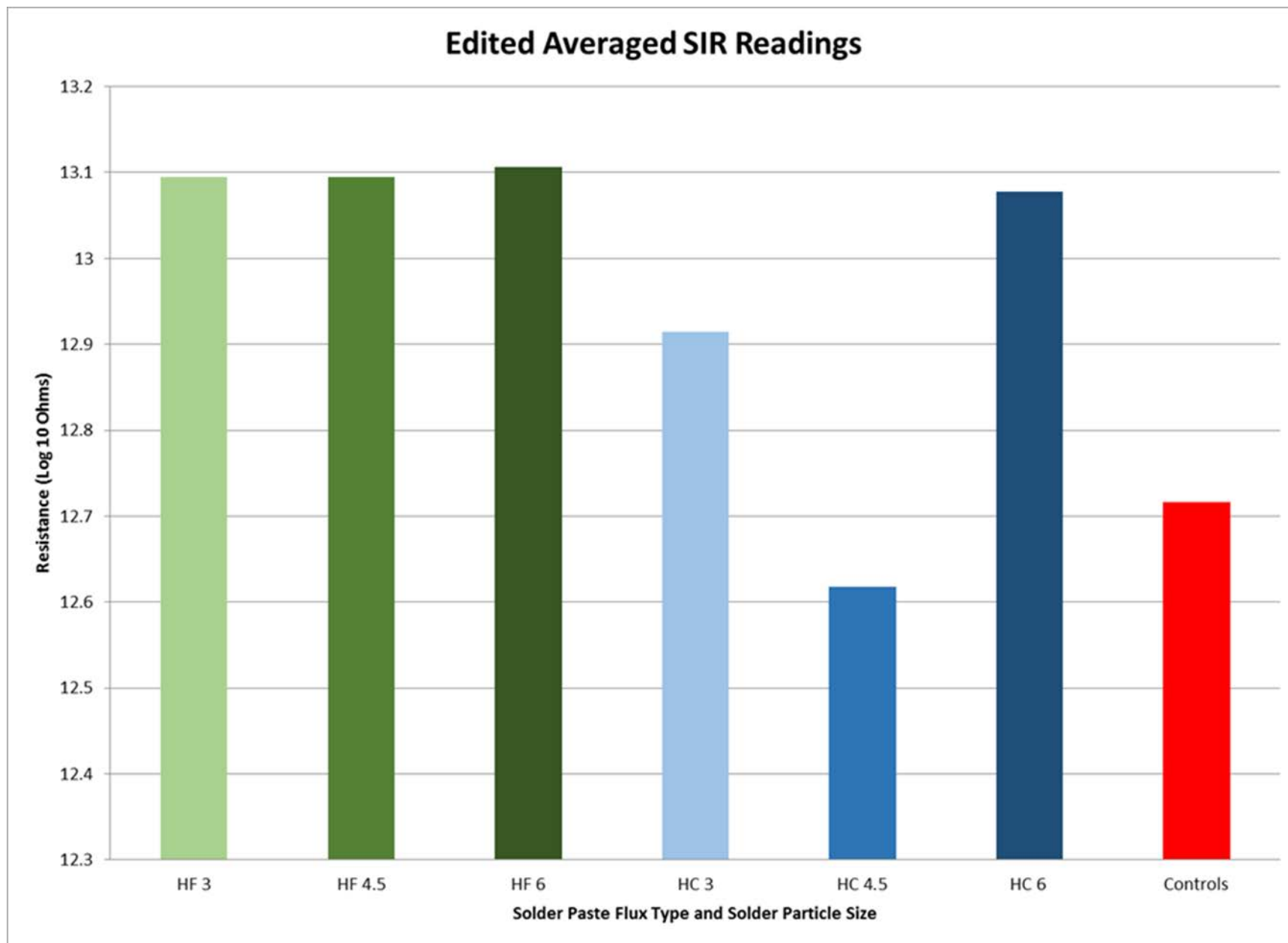


### Halogen Containing with Type 4.5 Powder - Edited



### Halogen Containing with Type 6 Powder - Edited







- All Averaged Readings
  - Very Similar to Each Other
  - Very Similar to and Higher than Controls
  - Very High
    - No Failure ( $>100\text{M}\Omega$ )
- Halogen Free – Immune to Particle Size
  - Virtually Identical
  - Based on Edited Averaged SIR Data
- Halogen Containing – Greater Variation
  - Based on Edited Averaged SIR Data
- Too Subtle or Confounded
  - Metal Load Variation
  - Oxidation Variation
    - Solder Powder
    - Copper Traces

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