Correlations of Salt Composition and Surface Insulation Resistance Results[a]

Nathan Pajunen[a], University of Waterloo, Waterloo, Ontario, Canada
Alexandre Romanov, Petro-Canada Lubricants, Mississauga, Ontario, Canada
Deepchand Ramjattan, Thalmic Labs, Kitchener, Ontario, Canada
Bev Christian[b] High Density Packaging User Group, Breslau, Ontario, Canada

Abstract

Sixteen simple inorganic salts were separately dissolved in water to a specific concentration, applied to separate Surface Insulation Resistance (SIR) coupons, dried and then the coupons were subjected to common SIR testing. A correlation between the final SIR resistance readings and the hydrated radii and ionic charges of the salts has been found. Squares of bare FR4 were immersed in more concentrated solutions of the same salts, rinsed, dried, ground up, leached and the concentrations of the liberated salts were obtained by ion chromatography and inductively coupled plasma optical emission spectroscopy. The results show some discernible correlations with the SIR results.

Introduction

SIR is a appropriate technique for checking for potential corrosion issues for laminate materials and bare boards[1,2]; flexible base dielectric[3]; fluxes; paste fluxes, core wire fluxes and underfills[4,5,6;] solder masks (both temporary and permanent)[7]; conformal coatings[8] and the efficacy of various cleaning protocols[9]. There are several corresponding test methods listed in documents of the International Electrotechnical Commission. They are not listed here in the references. The primary author has used SIR as one qualifying technique for any chemical that was going to temporarily or permanently come in contact with a printed circuit board assembly (PCBA).

The pass/fail criteria are set out in documents like IPC/EIA J-004[10], IPC J-005[11] and Telecordia (formerly Bellcore) GR-78[12]. The values listed have been determined and refined over the last half century of extensive testing by various research institutes, companies and universities. A review of this evolution will not be given here.

One of the main issues, besides chamber stabilization at a specific temperature and humidity, is the fact that for any liquid-based material that is going to be tested, surface tension can play havoc with any attempt at repeatability. This is especially the case with water-based materials because of the large surface tension of this excellent solvent. For example, over the years the primary author has had several students carry out SIR testing of the same salts under ostensibly the same concentration and conditions. See the results in **Figure 1**. Also what concentration should be used? From the results of previous work 7.5 x 10^{-4} Molar was chosen as this seems to be the range at which, at least for sodium chloride, the SIR results become more or less ”constant” (**Figure 2**).

Experimental Procedure

Materials

The required materials for the experiment were: a mixture of 25% de-ionized water and 75% isopropyl alcohol (ACS grade), bare FR4 (tetrabromobisphenol A (TBBPA) based) squares, SIR B-24 test boards, 18 megohm-cm water, sixteen (16) different simple salts, aluminum foil and SAC 305 wire solder (no-clean flux). The Surface Insulation Resistance (SIR) boards had 4 standard IPC-B-24 standard test patterns, interdigitated bare copper comb patterns. The comb patterns had trace widths of 0.4 mm and 0.5 mm of spacing between the traces. The 16 salt solutions, as noted in **Table 2**, were created and applied to the SIR

[a] Based on his co-op work term report as an undergraduate student, Department of Chemical Engineering, University of Waterloo
[b] Author to whom correspondence should be addressed

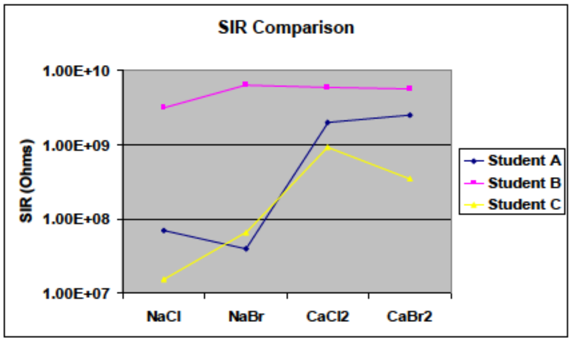


Figure 1 SIR Results for Four Salts Obtained by Three Different Students

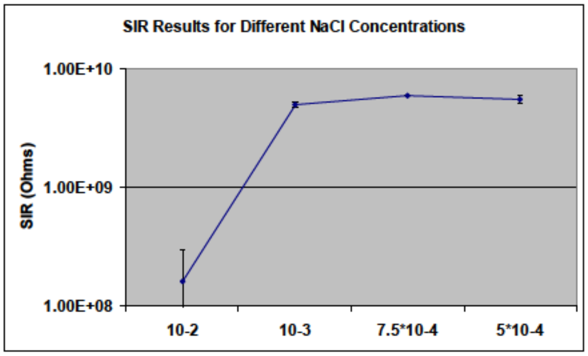


Figure 2 SIR Results for Different Concentrations of NaCl

boards for corrosion testing. All salts were ACS grade. The nominal concentration that was aimed for was 7.5E-4M, with the actual average concentration being 7.66E-4 +/- 0.23E-4M. For the immersion work where the bare FR4 squares were used the concentration of each salt was 1.000 +/- 0.002 M.

It was noted that the bottle of calcium bromide was labelled $CaBr_2xH_2O$, denoting an indeterminate amount of hydrated water in the crystal structure of the salt. To solve for x, a titration was performed using production complexometric Black T as an indicator to calculate the moles of calcium in a known mass of calcium bromide. Stoichiometric calculations for x provided a value of 1.126.

Equipment and Procedure

A production ionic contamination test system was used to eliminate ionic contamination by putting the SIR boards and the FR4 squares into its mixture of 25% de-ionized water and 75% isopropyl alcohol. The SIR boards and FR4 squares were washed for 30 minutes using the test system to eliminate any surface ionic contamination. After cleaning all B-24 coupons and FR 4 squares were handled with clean tongs or clean nitrile gloves. The FR-4 squares were then placed in a holder, covered with aluminum foiled and placed in a fumehood until they became fully dry. Drying of the B-24 coupons is explained below. The SIR coupons were wired to a horizontal humidity chamber metal rack. Soldering of wires to the SIR boards was completed with the aid of a soldering iron, lead free low activity cored solder wire and aluminum foil to avoid flux splatter on the SIR traces. The cabling was carefully arranged to not drape across adjacent coupons. The cables exited through the cable port on the side of the chamber. The rack was placed in the chamber such that the coupons lay flat and were parallel to the air movement.

The salt solutions for the SIR testing were prepared at a nominal concentration of 7.5×10^{-4} M using a 4-figure production precision scale. Due to the low concentrations, a volume of 1 L for each individual master salt solution was chosen to ensure an easily workable mass of each salt for weighing. Using eight SIR boards (32 comb patterns), each individual salt solution was applied on two separate comb patterns. For each salt solution 150µL were carefully placed on each comb pattern using an auto pipette. A full size production humidity chamber was used to create the conditions for the SIR testing. The humidity chamber was set to 85°C and low humidity to dry the applied salts and operated for 1 hour. Using the 150µL volume, careful placement, keeping the coupons level and undisturbed are believed to be some of the factors that lead to reasonably consistent results achieved. After visual confirmation that the salt solutions had dried, the SIR test was started by raising the humidity to 85% relative humidity and activating the Sirometer software to apply a constant DC voltage of 100V across the boards through a 1 megohm resistor for each circuit and take resistance measurements every hour for 168 hours. All SIR measurements were taken at 85°C/85%RH. The IC and OES-ICP conditions used are shown in **Table 1**. A production digital microscope was utilized at the end of the test to take images of the SIR boards for the purpose of examining the extent of corrosion and to view dendrite formations.

For the second set of experiments, the same salts as mentioned previously were again used, but made up as 1M solutions. The salts were weighed to the required amount, added to 500 mL volumetric flasks and then 18 megohm-cm water was added to the 500 mL mark on each volumetric flask. Initially, three FR4 squares were placed in production bags along with an individual salt solution and the bag heat sealed. There were also blanks processed (no salt in the water). The bags were then placed in a chamber for 1 hour at a temperature of 70°C. Afterwards, the boards were taken out of their bags with clean tongs and rinsed with 18 megohm-cm water to remove any salt on the surface of the boards. When the boards were dry they were then ground into a powder using two previously cleaned grinders, a production equipment grinder(for the initial grind) and another production equipment grinder (for the fine grind), sequentially. The grinders were cleaned for every different test iteration using production lab grade cleaning soap and rinsed thoroughly with 18 megohm-cm water. The dry powder was weighed and placed into the bags along with 40 mL of 18 megohm-cm water. The bags were then submerged into a production water bath at a temperature of 70°C for a duration of 1 hour to leach salt ions out of the powder. Following the leaching, appropriate pipettes, 15 mL and 50 mL centrifuge vials, a centrifuge and volumetric flasks were used to prepare the salt solutions for chemical analysis. Nitric acid was added into the extracted solutions (to a concentration of 2%) before the ICP-OES analytical runs. The ion concentrations were determined by the use of a production Inductively Coupled Plasma Optical Emission Spectrometer (OES-ICP) (for cations) and a production Ion Chromatograph (IC) (for anions). Appropriate standards were prepared and tested for both analytical instruments. All measurements were determined in triplicate.

Table 1 IC and OES-ICP Parameters

Ion Chromatography Parameters	**OES-ICP Parameters**
Guard column -4mm	Plasma gas flow: 15mL/min
4mm column	Auxiliary gas flow: 0.2 mL/min
(4mm) suppressor	Nebulizer gas flow: 0.5 mL/min
Injection loop 20um	RF: 1450 Watts
Eluent: 8.0mM Na2CO3/1mM NaHCO3	Pump speed: 2mL/min
Eluent flow rate 1mL/min	

Table 2: Salt solutions with concentrations of 7.5E-4M

Salt	MW (g/mol)	Volume (L)	Target Concentration (mol/L)	Target Mass (g)	Measured Mass (g)	Actual Concentration (mol/L)
NaCl	58.44	1	7.50E-04	0.0438	0.0456	7.80E-04
NaBr	102.89	1	7.50E-04	0.0772	0.0776	7.54E-04
Na_2SO_4	142.04	1	7.50E-04	0.1065	0.1061	7.47E-04
$NaNO_3$	84.99	1	7.50E-04	0.0637	0.061	7.18E-04
KCl	74.55	1	7.50E-04	0.0559	0.0588	7.89E-04
KBr	119	1	7.50E-04	0.0892	0.0951	7.99E-04
K_2SO_4	174.26	1	7.50E-04	0.1307	0.134	7.69E-04
KNO_3	101.1	1	7.50E-04	0.0758	0.0783	7.74E-04
$CaCl_2{\bullet}2H_2O$	147.01	1	7.50E-04	0.1103	0.1186	8.07E-04
$CaBr_2{\bullet}xH_2O$, where x = 1.126	220.18	1	7.50E-04	0.1651	0.1688	7.67E-04
$CaSO_4{\bullet}2H_2O$	172.17	1	7.50E-04	0.1291	0.1276	7.41E-04
$Ca(NO_3)_2{\bullet}4H_2O$	236.15	1	7.50E-04	0.1771	0.1761	7.46E-04
$MgCl_2{\bullet}6H_2O$	203.3	1	7.50E-04	0.1525	0.1579	7.77E-04
$MgBr_2{\bullet}6H_2O$	292.2	1	7.50E-04	0.2192	0.2158	7.39E-04
$MgSO_4$	120.37	1	7.50E-04	0.0903	0.0934	7.76E-04
$Mg(NO_3)_2{\bullet}6H_2O$	256.41	1	7.50E-04	0.1923	0.1997	7.79E-04

Results and Discussion

The NaCl and NaBr SIR test patterns exhibited numerous shorts/recovery cycles. See **Figure 3**. The sodium sulfate test patterns started out the same but one failed to provide data to the end of the test. The sodium nitrate test patterns did not provide viable data and have therefore been ignored for the rest of the discussion. There was not time to repeat the experiment for any salts. All the sodium salt test patterns had final SIR reading in the range of 8E7 ohms.

The potassium salts produced fewer shorts in the 168 hours of testing. As well, the resistance values at the end of the testing were also higher, in the range of 2-8E8 ohms. One potassium test pattern flat lined before the end of the test. The SIR summary charts are shown in **Figure 4**.

All eight of the magnesium salt test patterns provided data until the end of the 168 hour test. Of the four sets of salts (Na^+, K^+, Mg^{+2} and Ca^{+2}), the magnesium salts produced the most consistent results, test pattern to test pattern. The final SIR values were in the range of 2-5E9 ohms. See **Figure 5**.

The results for the calcium salts are shown in **Figure 6**. One of the calcium bromide test patterns did not make it to the end of the testing. For a good portion of the second half of the testing, one of the calcium sulfate test patterns showed unusually high SIR values while at the same time showing the usual formation and extinction of dendrites seen for the other salts. This test pattern showed more usual SIR readings during the last few hours of the testing. The final results are in the range of 2-4E9 ohms.

The last five readings of the data sets that were collected at the end of the experiment (still at 85°C/85%RH) were averaged. If a particular salt had two complete measurement sets, then there were ten points to average. **Figures 7 and 8** show the results of this exercise. Note that the value plotted for sodium nitrate came from the data set of an earlier student's work. These summary charts clearly show that the order of increasing corrosiveness of the cations is highest for sodium and follows the order $Na^+ > K^+ > Ca^{+2} \geq Mg^{+2}$. The single charged cations are worse than the double charged cations. It is not as clear cut for the anions, but is generally in the order of $Br^- > Cl^- > NO_3^- \geq SO_4^{-2}$ from most to least corrosive.

Attempts to find a relationship between the SIR results and ionic charges, sizes, limiting ionic conductivity values and other common terms related to aqueous ionic conductivity, even the Debye Hückel equation proved fruitless. However, a reasonable relationship between the SIR results as a function of both ion charges and aqueous radii did show some correlation, as shown in **Figure 9**. The size of the hydrated sulfate ion was estimated to be 6 Angstroms from the graph in reference 13, while others were taken form reference 14. The 6 Å value may be on the high side.

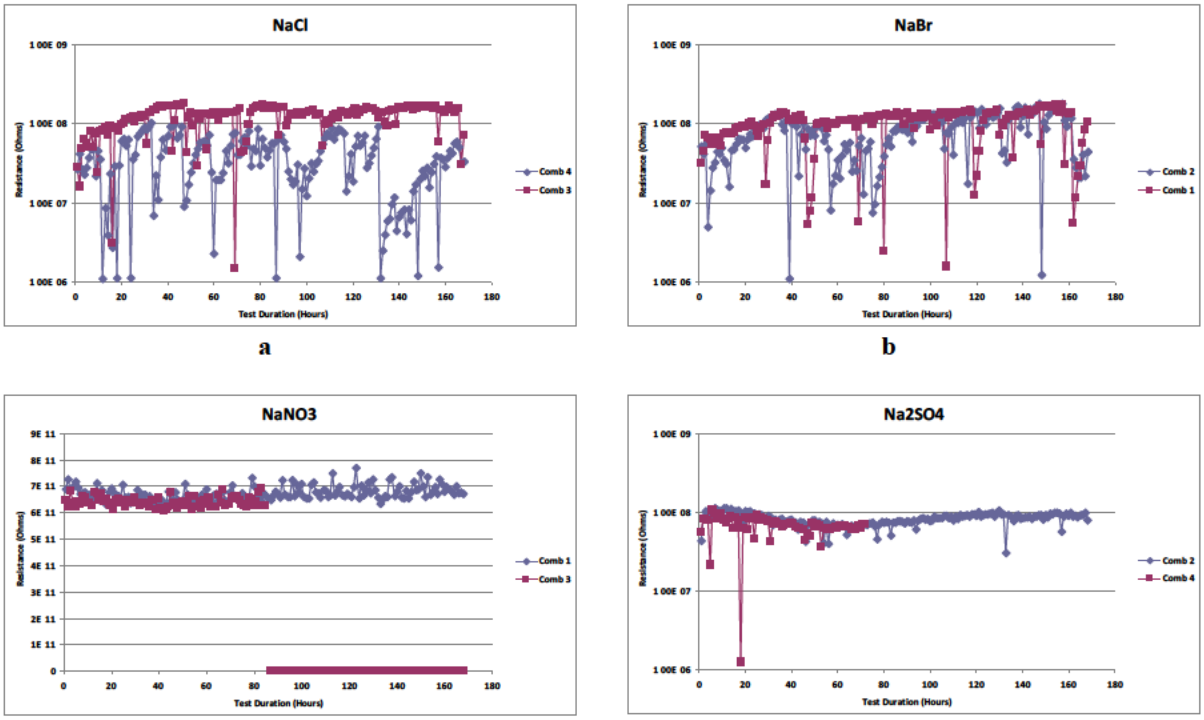
A summary of the results for the immersion portion of the project are shown in **Tables 3** & **4.** **Figures 10 & 11** show the results for individual ions. The concentration values are expressed as µgrams of leached ion per gram of FR4 material. Values in red underline are less than the blank value for that ion by more than the sum of the one standard deviation of the blank and the ion in question, while values in blue italics are more than the blank value for that ion by more than the sum of the one standard deviation of the blank and the ion in question.

For the particular TBBPA-based FR4 used in this study the following observations were made:

1. Subtracting out the blank values, the overall order of decreasing anion leaching is $Br^- > Cl^- > SO_4^{-2} > NO_3^-$.
2. Subtracting out the blank values, the overall order of decreasing cation leaching is $Na^+ > K^+ > Ca^{+2} > Mg^{+2}$.
3. Generally speaking, single charged ions are more likely to leach out than double charged ions.
4. The presence of any added salt decreases the release of magnesium to less than that of the blank.
5. There is not much more calcium ions leached from the boards, over and above that of the blank.
6. Bromide readily leaches out of the particular FR4 used in this study and this is generally enhanced by adding other salts.
7. For chloride release, this is enhanced in decreasing order by bromide>chloride>sulfate> nitrate, irrespective of the cation.
8. For bromide release, the same order is more or less seen, although it is not as evident because of the large standard deviation for the magnesium chloride data.
9. For sulfate release, more is liberated for boards soaked in sulfate salts of potassium, magnesium and calcium, but it appears to not be the case for the sodium sulfate but this is only because of the large standard deviation for the sodium bromide data.
10) There is little nitrate in FR4 and the presence of other salts decreases the release of the little that is there.
11) There are not definitive trends for the nitrate leaching data.
12) From the blank samples 1-15 micrograms of each ion per gram of board material is leached, except in the case of calcium ions where 50 micrograms/gram of FR4 are leached.

Figure 12 is a summary chart for the cations while **Figure 13** is a similar display for the anions. In work by Munson et al.[15] they applied various ionic salt solutions of individual salts to specialized simple circuit boards with varying spacing between two electrodes. They measured the SIR values over the course of the test and the concentration of the ions on the board before and after the SIR testing. The SIR results cannot be directly compared because of so many differences (board type, coupon pattern, electrode spacing, temperature and voltage). However, the relative results for the salt concentrations of their study and the current one are interesting. There were four salts common to both studies. The results for those four salts for the different spacings from the Munson work (constant spacing in the current study) are shown in **Figure 14**. Note, two of the KBr concentrations were different from the other salts (6 μg/in^2 and 12 μg/in^2 rather than the usual 5 & 10 μg/in^2), but have been plotted as 5 and 10 μg/in^2. Sodium and potassium were the dominant cations used in the referenced work, so there is not enough data for a similar comparison to the four cations of the present work. It is immediately evident that although the units of measurement are different (μ/g FR4 vs. μg/in^2 FR4) that the same order of concentrations was found in both studies. That is, bromide is released from FR4 during the SIR testing with decreasing amounts of chloride and with sulfate and nitrate generally absorbed/released in about equal amounts.

Some initial work by the present research group was done looking at the differences in leaching FR4 boards at 2 different temperatures. As expected, there was more leaching at 80°C than 70°C but the changes were not dramatic. See **Fig. 15**.



a

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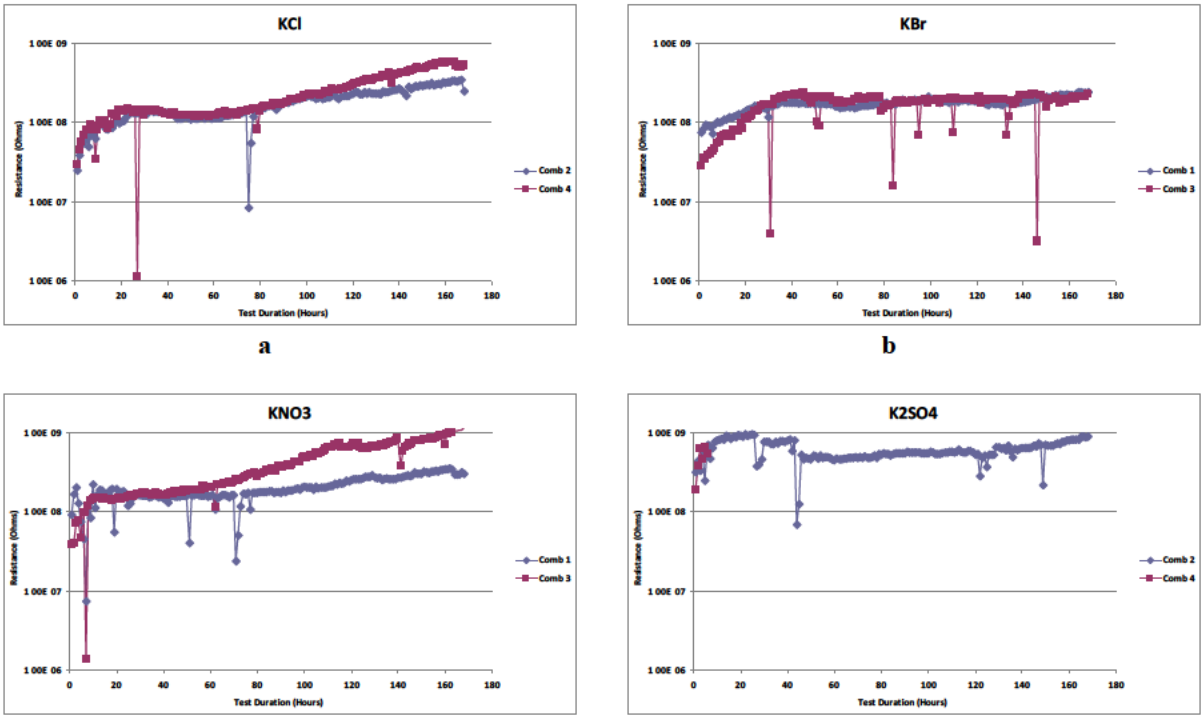
Figure 3 SIR Results a) NaCl, b) NaBr, c) $NaNO_3$ and d) Na_2SO_4



a b

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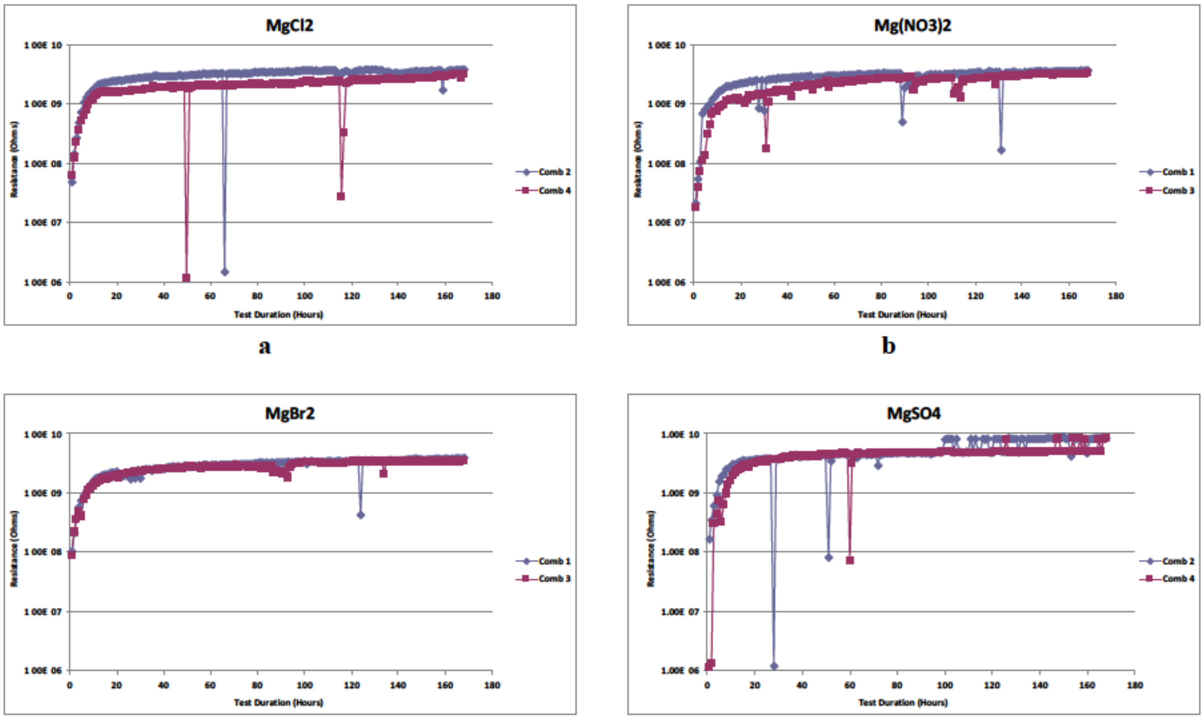
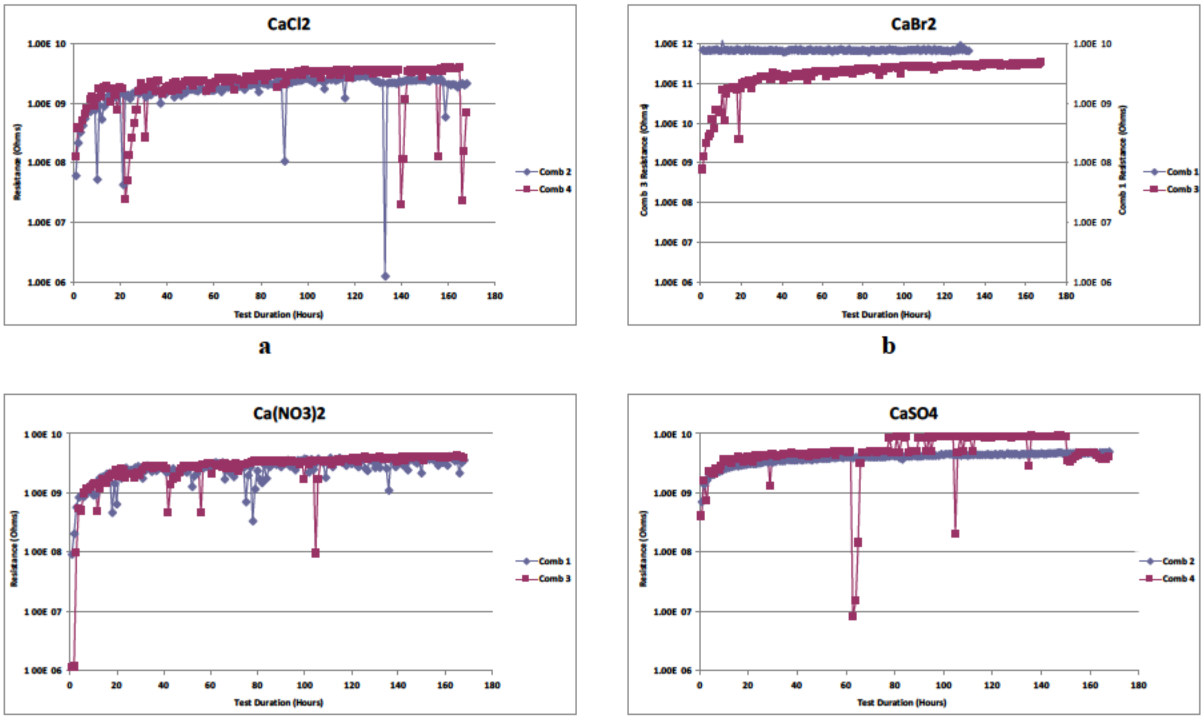
Figure 4 SIR Results a) KCl, b) KBr, c) KNO_3 and d) K_2SO_4



a b

c d

Figure 5 SIR Results a) $MgCl_2$, b) $MgBr_2$, c) $Mg(NO_3)_2$ and d) $MgSO_4$



a b

c d

Figure 6 SIR Results a) CaCl2, b) CaBr2, c) $Ca(NO_3)_2$ and d) $CaSO_4$

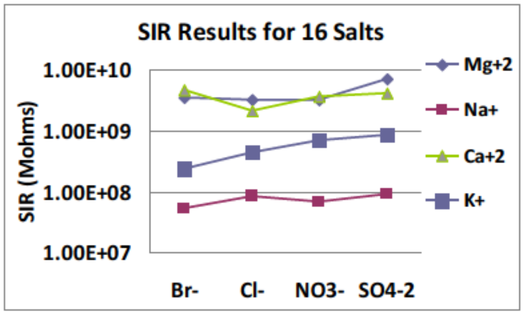


Figure 7 SIR Results Plotted as a Function of the Anions

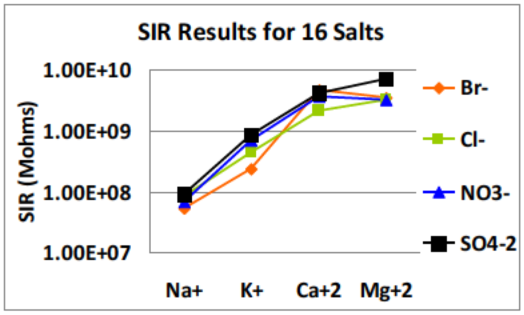


Figure 8 SIR Results Plotted as a Function of the Cations

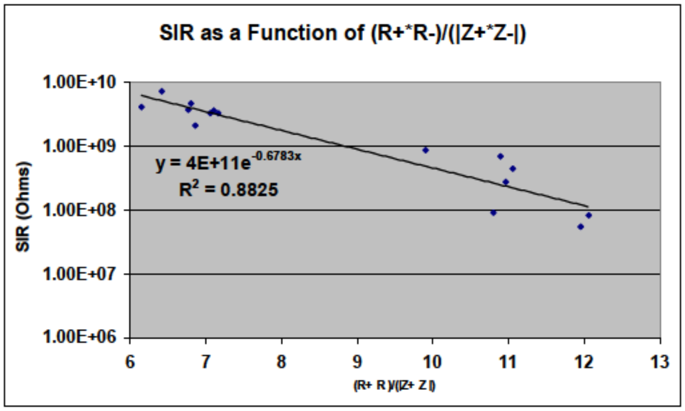
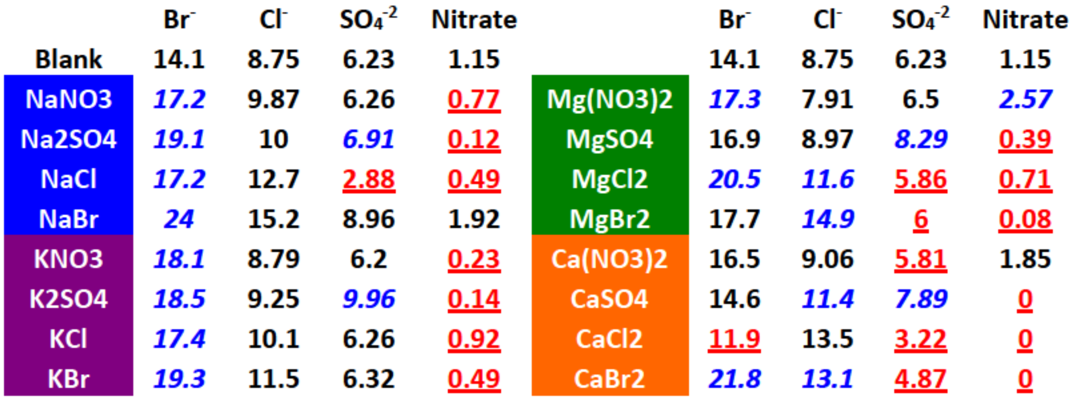


Figure 9 Final SIR Values as a Function of Ion Charges and Hydrated Radii

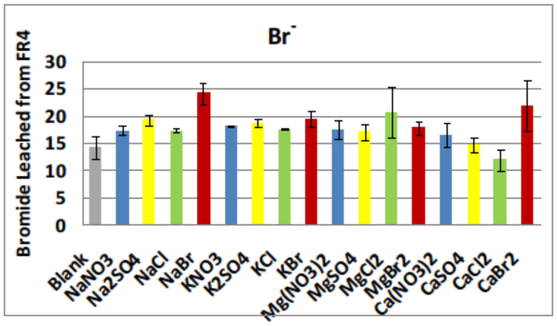
Table 3 µg/g of Cations Leached from the FR4

	Na^+	K^+	Mg^{+2}	Ca^{+2}		Na^+	K^+	Mg^{+2}	Ca^{+2}
Blank	11.6	0.82	6.29	50.5		11.6	0.82	6.29	50.5
NaCl	*39.1*	*1.41*	1.61	43.4	MgCl2	23.6	1.03	2.65	47.3
NaBr	13.2	0.89	2.34	46.4	MgBr2	14.7	0.78	2.33	52.6
NaNO3	11.3	0.43	1.47	41.7	Mg(NO3)2	18	0.96	2.18	48.6
Na2SO4	11.5	0.59	1.5	43	MgSO4	20.5	0.87	2.45	48.6
KCl	11.9	0.87	1.89	51.4	CaCl2	11.6	0.89	1.58	44.8
KBr	16.7	1.02	2.07	53.5	CaBr2	11.2	0.52	2.1	45.1
KNO3	10.6	0.69	1.76	49.9	Ca(NO3)2	9.2	0.49	1.73	50
K2SO4	12.7	*1.15*	1.85	50.1	CaSO4	14.5	0.56	2.16	55

Table 4 µg/g of Anions Leached from the FR4



	Br^-	Cl^-	SO_4^{-2}	Nitrate		Br^-	Cl^-	SO_4^{-2}	Nitrate
Blank	14.1	8.75	6.23	1.15		14.1	8.75	6.23	1.15
NaNO3	*17.2*	9.87	6.26	0.77	Mg(NO3)2	*17.3*	7.91	6.5	*2.57*
Na2SO4	*19.1*	10	*6.91*	0.12	MgSO4	16.9	8.97	*8.29*	0.39
NaCl	*17.2*	12.7	2.88	0.49	MgCl2	*20.5*	*11.6*	5.86	0.71
NaBr	*24*	15.2	8.96	1.92	MgBr2	17.7	*14.9*	6	0.08
KNO3	*18.1*	8.79	6.2	0.23	Ca(NO3)2	16.5	9.06	5.81	1.85
K2SO4	*18.5*	9.25	*9.96*	0.14	CaSO4	14.6	*11.4*	*7.89*	0
KCl	*17.4*	10.1	6.26	0.92	CaCl2	11.9	13.5	3.22	0
KBr	*19.3*	11.5	6.32	0.49	CaBr2	*21.8*	*13.1*	4.87	0



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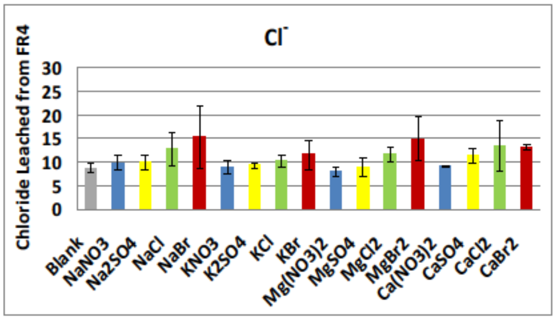

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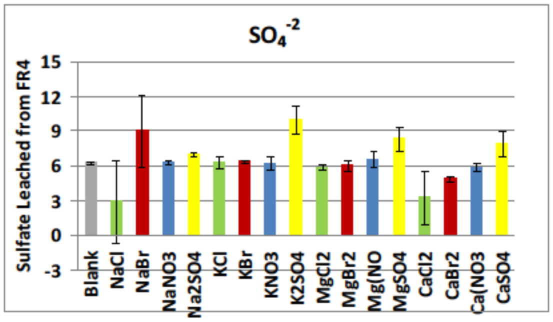

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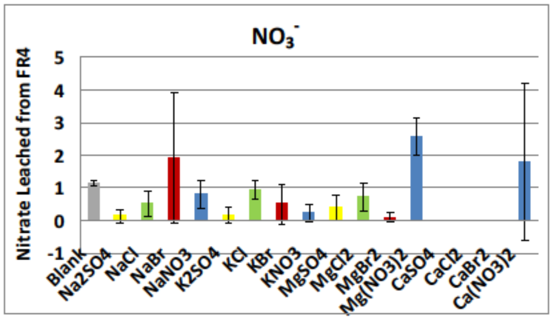
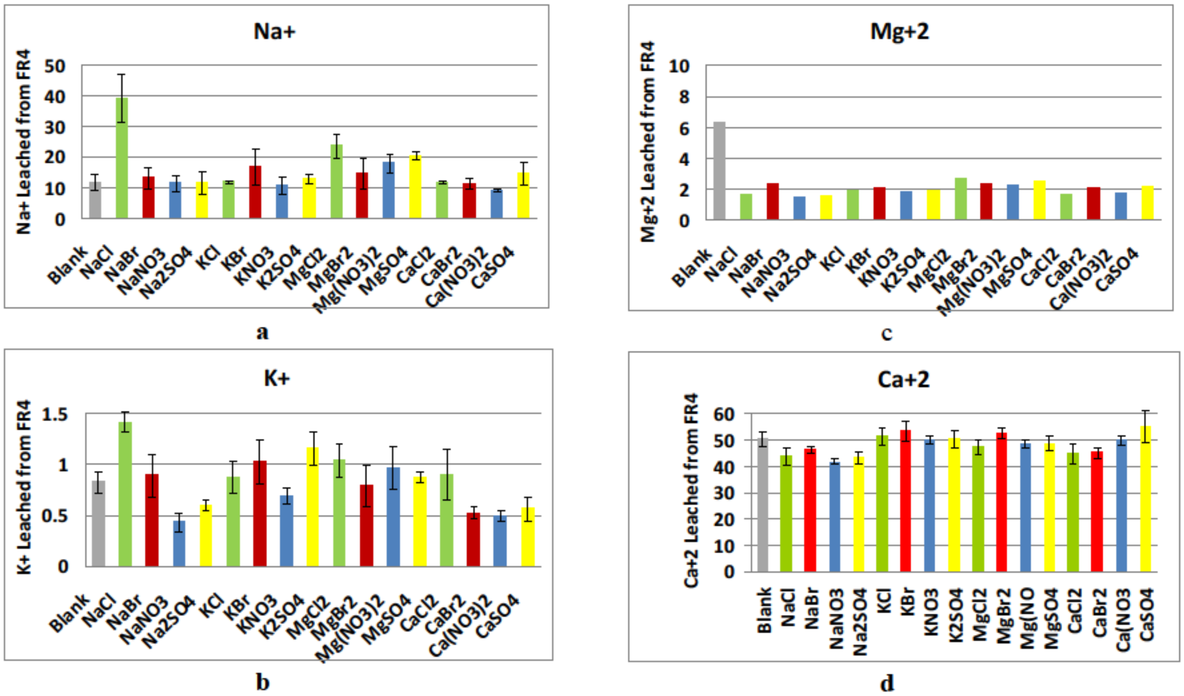


d

Figure 10 Anions Leached from FR4 Treated with the 16 Salts (Vertical axis µg/g FR4)
a) Bromide, b) Chloride, c) Sulfate, d) Nitrate



a c b d

Figure 11 Cations Leached from FR4 Treated with the 16 Salts (Vertical axis µg/g FR4)
a) Sodium, b) Potassium, c) Magnesium, d) Calcium

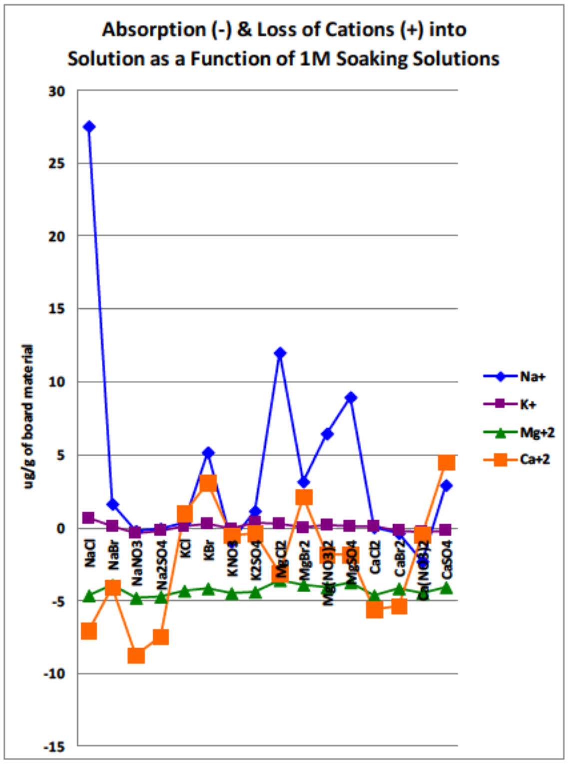


Figure 12 Cation Summary, Blank Subtracted from Each Data Point

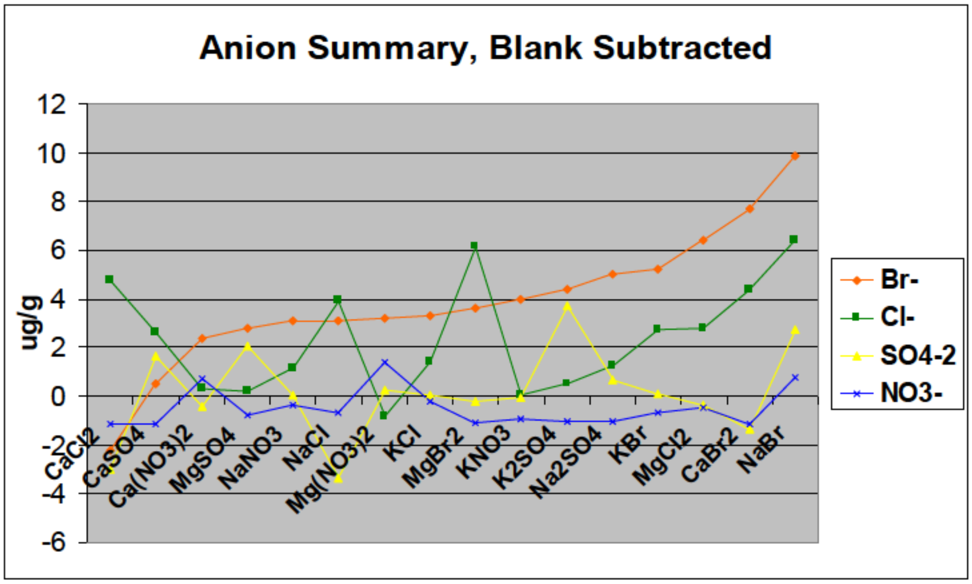


Figure 13 Anion Leaching Summary, Blank Subtracted from Each Data Point

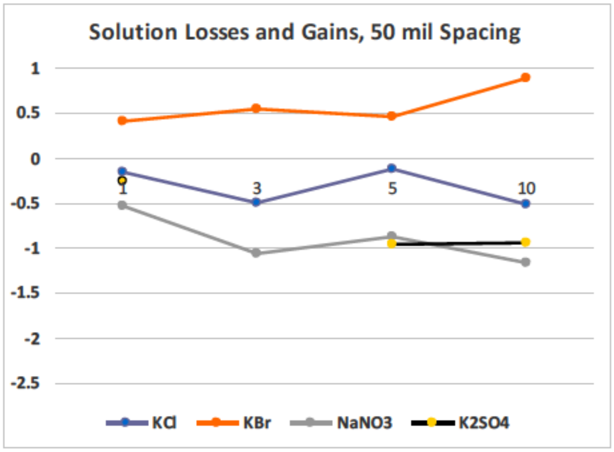


Figure 14 Ion Losses in ug/in^2 in Reference 15

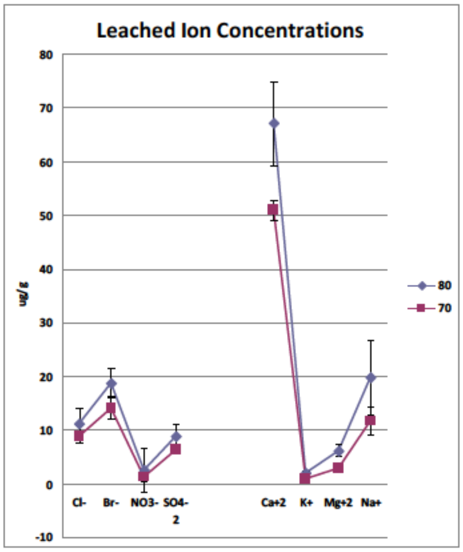


Figure 15 Results of Leaching as a Function of Temperature

Conclusions

The data confirms that a concentration of 7.5 x 10^{-4} M is a reasonable concentration to use for SIR testing involving inorganic, non-sublimating salts. The order of decreasing corrosiveness of the cations (as measured by SIR) is highest for sodium and follows the order $Na^+ > K^+ > Ca^{+2} > Mg^{+2}$. It is not as clear cut for the anions, but in the current study was generally in the order of $Br^- > Cl^- > NO_3^- \geq SO_4^{-2}$. Munson et al. (15) showed the order as $Cl^- \geq NO_3^- \geq SO_4^{-2} > Br^-$. However, the reader must be reminded that their work was based on ug/in^2 rather than formula units. Attempts to correlate the SIR results with ionic charge, crystal ionic radii, diffusion coefficients and molar ionic conductance values proved fruitless. A reasonable correlation of SIR results with $(R^+ * R^-)/(|Z^+ * Z^-|)$ where the R values are the radii of hydrated ions and the Z values are the individual ionic changes was found. However, with the somewhat suspect estimate for the hydrated radius of the sulfate ion and with a correlation coefficient less than 0.9, this may just be fortuitous and further studies are required to confirm or refute the applicability of this observation.

There should be no loss of cations from the salt solutions relative to the blank due to electrolytic reactions because any native metals, oxides and/or hydroxides produced would be soluble/reactive enough that they would still be in solution as the initial ion. However, there was a consistent decrease in the free magnesium ions, which suggests that the powdered FR4 either readily adsorbed or absorbed this particular ion. The effect on calcium ions was much more erratic. Potassium ions were not affected one way or the other. For the anions, if chlorine, bromine, nitrous oxides, sulfur dioxide and/or hydrogen sulfide were to be produced the minute quantities would most likely never leave the solution before reacting.

Both the current work and reference 15 show that bromide is being leached from FR4. And one of the base chemicals of many FR4 laminates is tetrabromobisphenol A. The mean bond energy for a carbon-bromine bond is 66 kcal/mole, as compared to C-Cl and C-C values of 79 and 83 kcal/mole, respectively.[16] The lower bond energy of bromine containing covalent bonds and the large number of them present in the FR4 used in the present study point in the direction of the likelihood of bromine leaching. However, 66 kcal/mole is still a significant energy barrier. Perhaps in the current study the destruction of carbon-bromine bonds was enhanced by:

1. the high concentrations of the salts the FR 4 squares were soaked in
2. by the use of 70°C for the immersion portion of the leaching study
3. the frictional heat generated in grinding the FR4 material.

Munson et al. used 40°C/90%RH and a 5 V bias while the current work used 85°C85%RH and no bias for the leaching portion of the study. Yet even with the lower temperature and using a moist atmosphere rather than immersion in water-based solutions they found bromide leached from the test boards. It must be noted that in and of itself the leaching of the bromide alone (no extra added) is not enough to cause SIR failure. If it was the case, then all the reference coupons used in the numerous SIR studies that have taken place over the last few decades would have failed.

Table 5 shows a comparison of the SIR results and leaching results from both the present work and the Munson study. The order of leaching of anions in both studies is the same. The corrosivity order of the cations as obtained by SIR and the leaching order of the cations in the present work are essentially the same. The corrosivity order of the anions as obtained by SIR and the leaching order of the anions in the present work are similar. The question then becomes is the order of corrosivity influenced by the leaching or absorption/adsorption of the ions. What was found to be different is the order for the corrosivity of bromide between the two research groups. Certainly, more investigation is required.

Table 5 Summary of SIR and Leaching Study Results

	SIR	Leaching into Solution
	Corrosive	
Cations	$Na^+ > K^+ > Ca^{+2} \geq Mg^{+2}$	$Na^+ > K^+ > Ca^{+2} > Mg^{+2}$
Anions	$Br^- > Cl^- > NO_3^- \geq SO_4^{-2}$	$Br^- > Cl^- > SO_4^{-2} > NO_3^-$
Anions[15]	$Cl^- \geq NO_3^- \geq SO_4^{-2} > Br^-$	$Br^- > Cl^- > SO_4^{-2} \geq NO_3^-$

Further work could be done using SIR patterns on a fluoropolymer, glass or ceramic base for comparison. This would allow a "pure" SIR investigation. FR4 materials of different Tg values could also be investigated. TBBPA free materials would also be another avenue for experimentation. Others salts should also be used. Lithium, ammonium, common transition metal, fluoride, sulfide and heteropolyatomic anion-containing salts would be possible candidates. The additional salts could be used to test the SIR to hydrated ion radius correlation. More leaching studies could be done in which the boards were no ground to powder. Doing more extensive leaching studies by immersing board materials in salt solutions of different temperatures could allow one to determine activation energies for the uptake/loss of ions from the materials. Insight into the ion leaching could be done by means of isotopic studies.

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The authors would like to thank Doug Pauls of Rockwell Collins and the facilitators of the High Density Packaging User Group for editing and helpful suggestions. This paper is dedicated to some of the directors, senior directors and a vice president in charge of the authors' group, the former Materials Interconnect Lab. at Blackberry. This work is based on the co-op work term report of Nathan Pajunen as an undergraduate student of the Department of Chemical Engineering, University of Waterloo at Blackberry.

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References

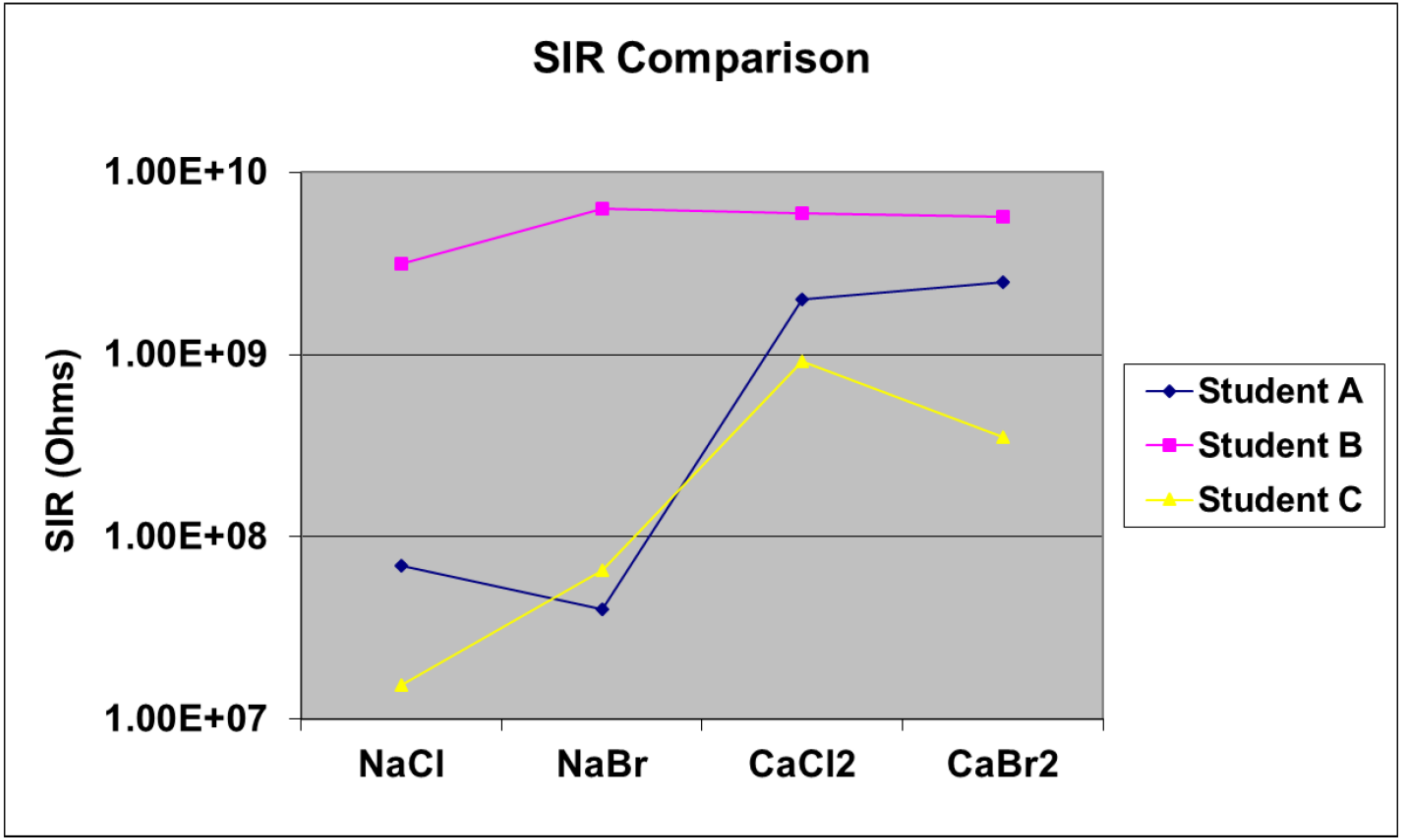
1. IPC TM-650 Test Methods Manual, Method TM 2.6.3F Moisture and Insulation Resistance, Printed Boards
2. IPC TM-650 Test Methods Manual, Method TM 2.6.3.5 Bare Board Cleanliness by Surface Insulation Resistance
3. IPC TM-650 Test Methods Manual, Method TM 2.6.3.2B Insulation and Moisture Resistance, Flexible Base Dielectric
4. IPC TM-650 Test Methods Manual, Method TM 2.6.3.3B Surface Insulation Resistance, Fluxes
5. IPC TM-650 Test Methods Manual, Method TM 2.6.3.6 Surface Insulation Resistance - Fluxes – Telecommunications
6. IPC TM-650 Test Methods Manual, Method TM 2.6.3.7 Surface Insulation Resistance
7. IPC TM-650 Test Methods Manual, Method TM 2.6.3.1E Solder Mask - Moisture and Insulation Resistance
8. IPC TM-650 Test Methods Manual, Method TM 2.6.3.4A Moisture and Insulation Resistance – Conformal Coating
9. IPC-CH-65 Guidelines for Cleaning of Printed Boards and Assemblies
10. IPC/EIA-J-STD-004, Requirements for Soldering Fluxes
11. IPC J-STD-005, Requirements for Soldering Pastes
12. Telecordia GR-78, Generic Requirements for the Physical Design and Manufacture of Telecommunications Products and Equipment
13. A. G. Volkov, S. Paula, and D. W. Deamer, Two mechanisms of permeation of small neutral molecules and hydrated ions across phospholipid bilayers, Bioelectrochem. Bioenergetics 42:153-160, 1997.
14. http://www.gly.uga.edu/railsback/Fundamentals/815HydratedCationRadii04LS.pdf, last referenced October 20, 2017.
15. Terry Munson, Paco Solis, Nick Munson, Steve Ring and Evan Briscoe, “How Clean is Clean Enough”, Proceedings of IPC APEX 2015.
16. William G.Davies, Introduction to Chemical Thermodynamics, W.B. Saunders Co., Toronto, 1972.

Correlations of Salt Composition and Surface Insulation Resistance Results

Nathan Pajunen, University of Waterloo, Waterloo, Ontario, Canada
Alexandre Romanov, Deepchand Ramjattan, Bev Christian

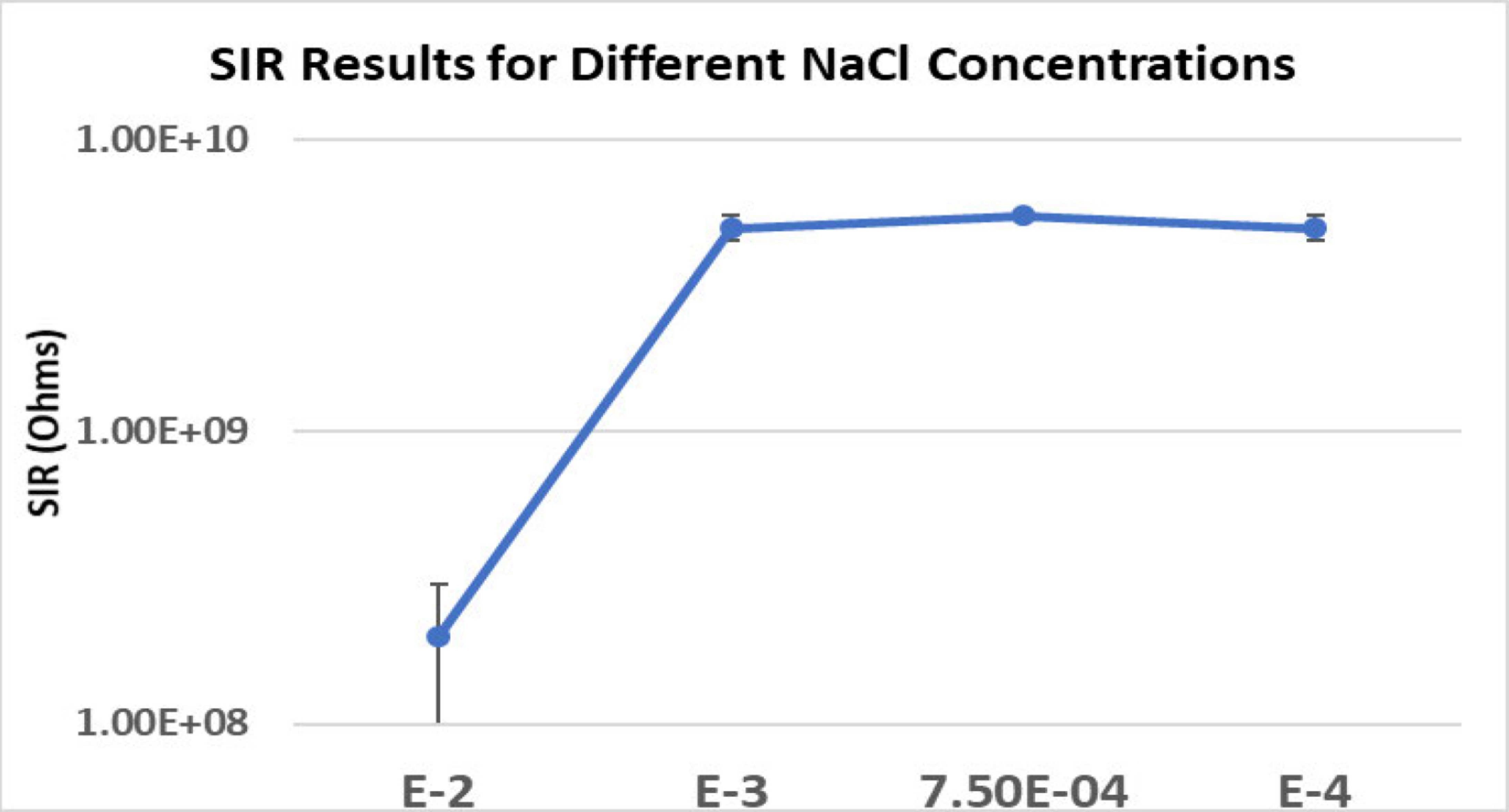
Uses of Surface Insulation Resistance Testing

- Checking for potential corrosion issues for laminate materials & bare boards
- Flexible base dielectric
- Fluxes, paste fluxes, core wire fluxes
- Underfills
- Solder masks (both temporary and permanent)
- Conformal coatings
- The efficacy of various cleaning protocols


SIR Comparison
SIR (Ohms)
1.00E+10
1.00E+09
1.00E+08
1.00E+07
NaCl
NaBr
CaCl2
CaBr2
Student A
Student B
Student C

Conditions of SIR Testing

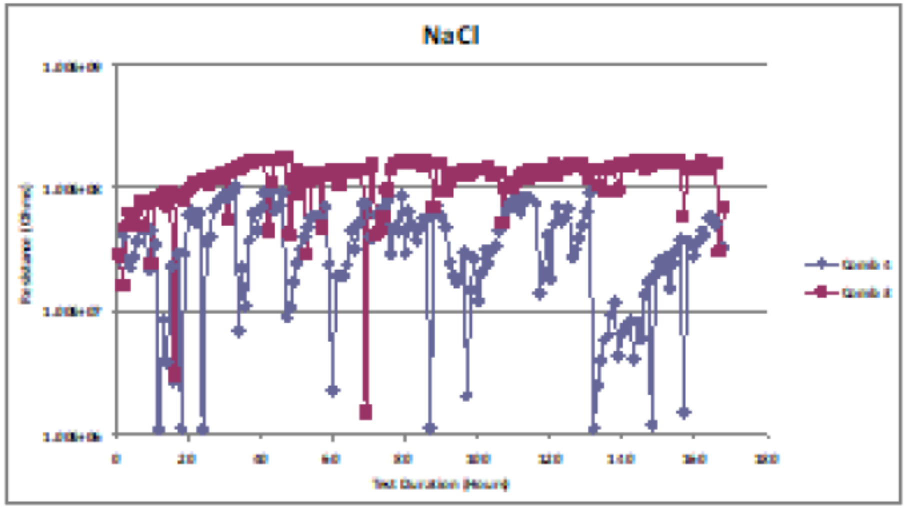
- Number of Hours – 168 hours
- Temperature – 85°C
- Humidity – 85%RH
- Voltage Bias – 100 V
- Test Voltage – 100V
- 150µL of solution applied to each comb pattern
- Concentration? – Next slide

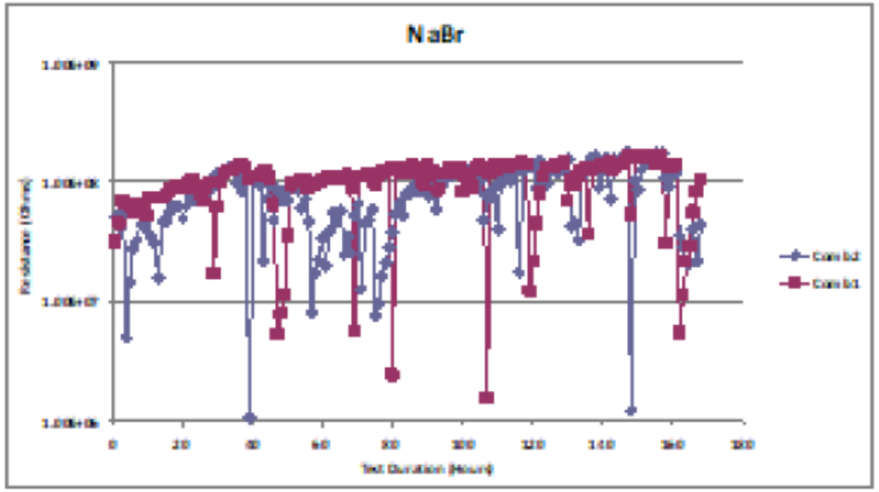

SIR Results for Different NaCl Concentrations
SIR (Ohms)
1.00E+10
1.00E+09
1.00E+08
E-2
E-3
7.50E-04
E-4

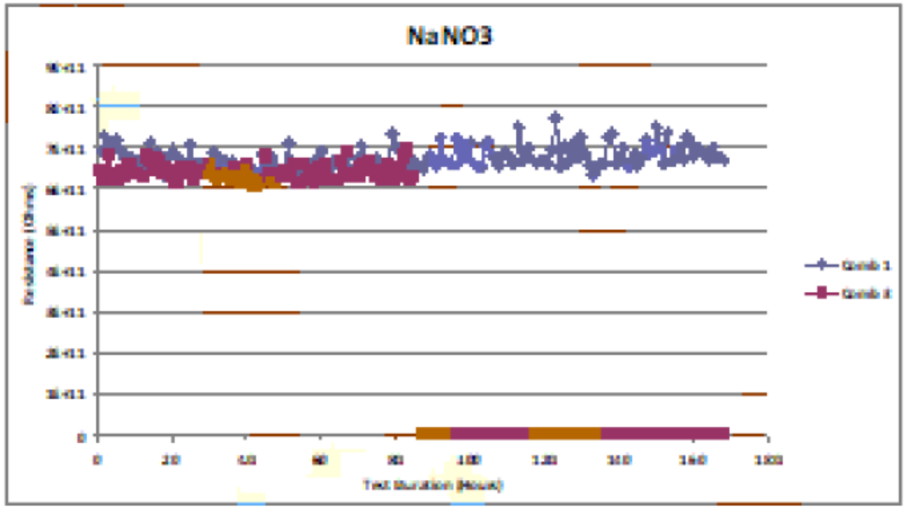
Salts Chosen for the Study

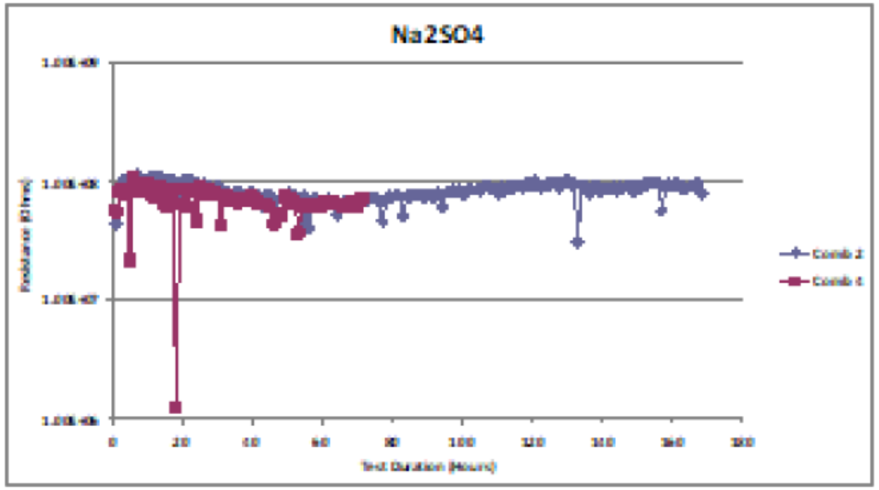
	Na^+	K^+	Mg^{+2}	Ca^{+2}
Cl^-	√	√	√	√
Br^-	√	√	√	√
NO_3^-	√	√	√	√
SO_4^{-2}	√	√	√	√

SIR Results for Sodium Salts

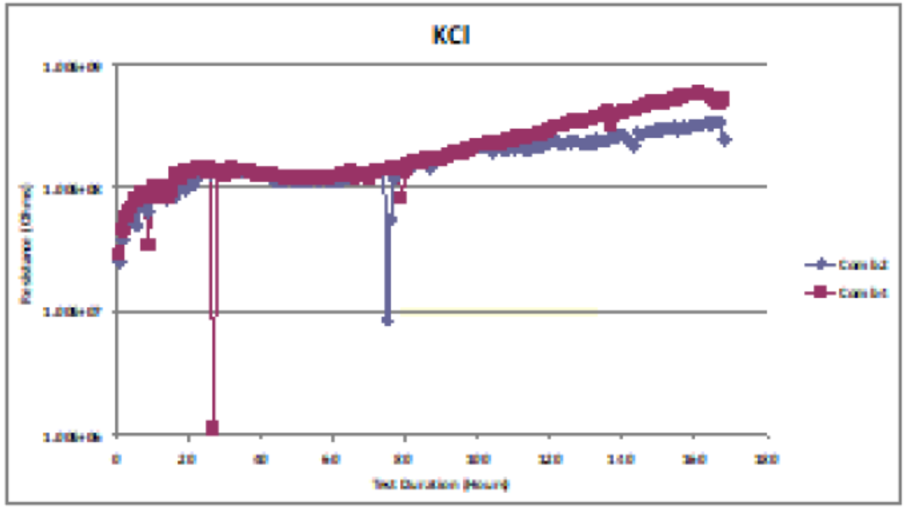


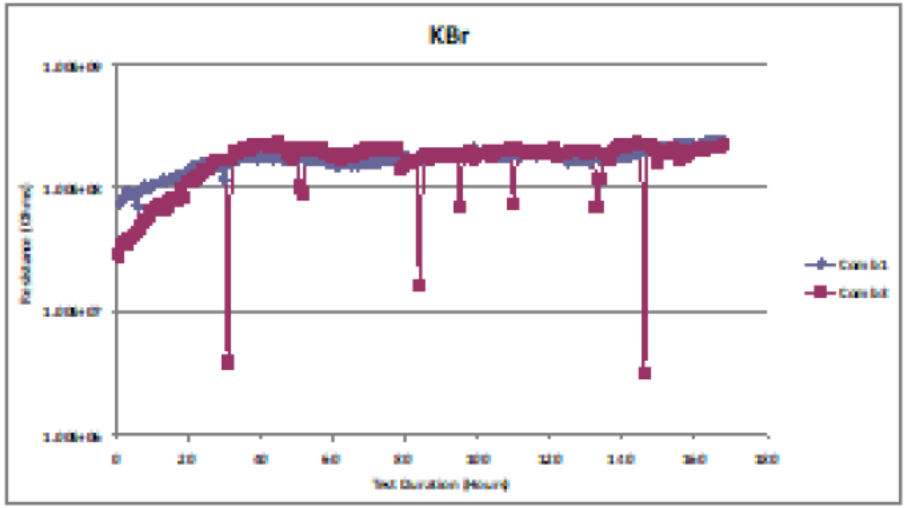


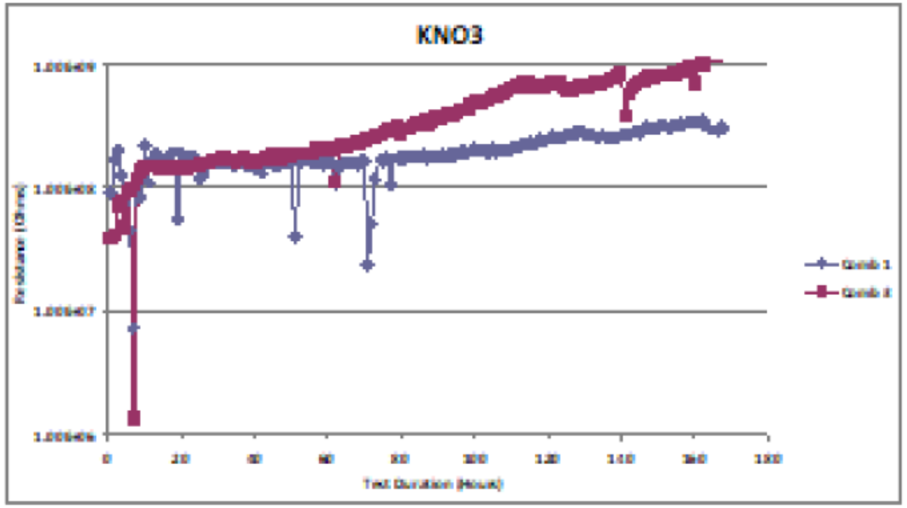


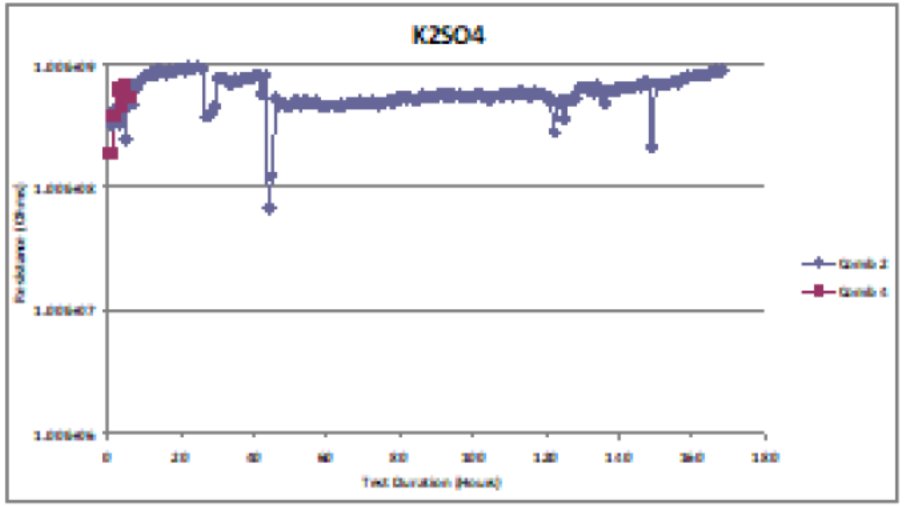


SIR Results for Potassium Salts

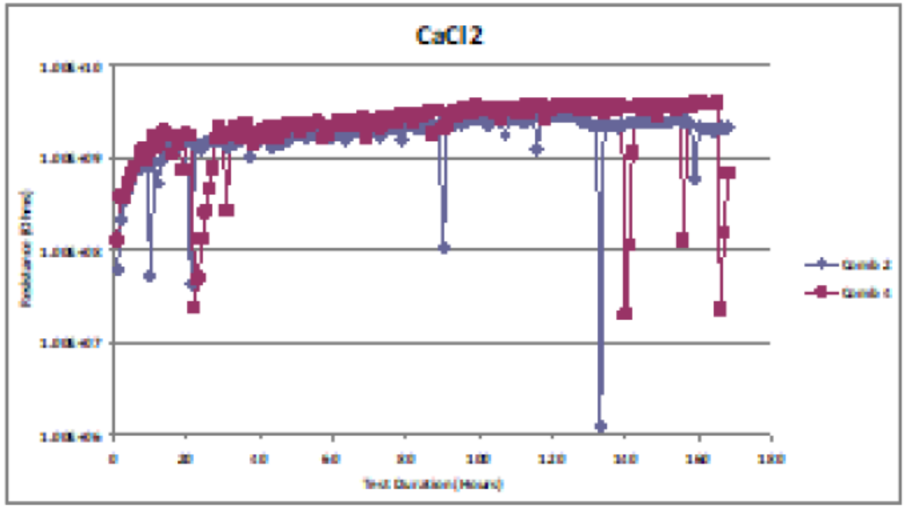

KCl

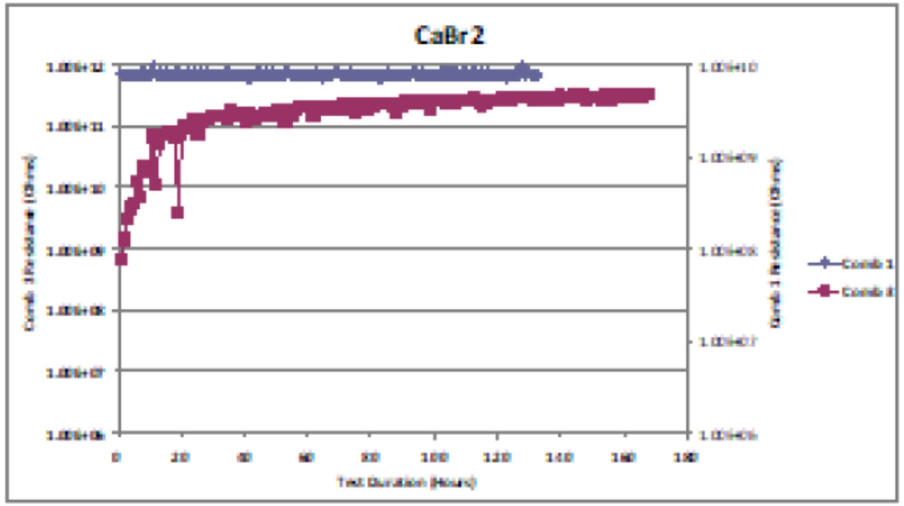

KBr

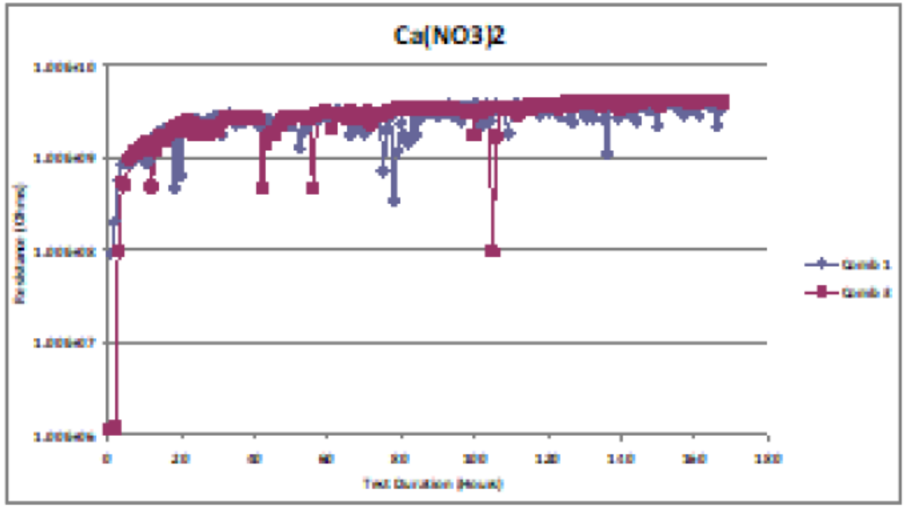

KNO3

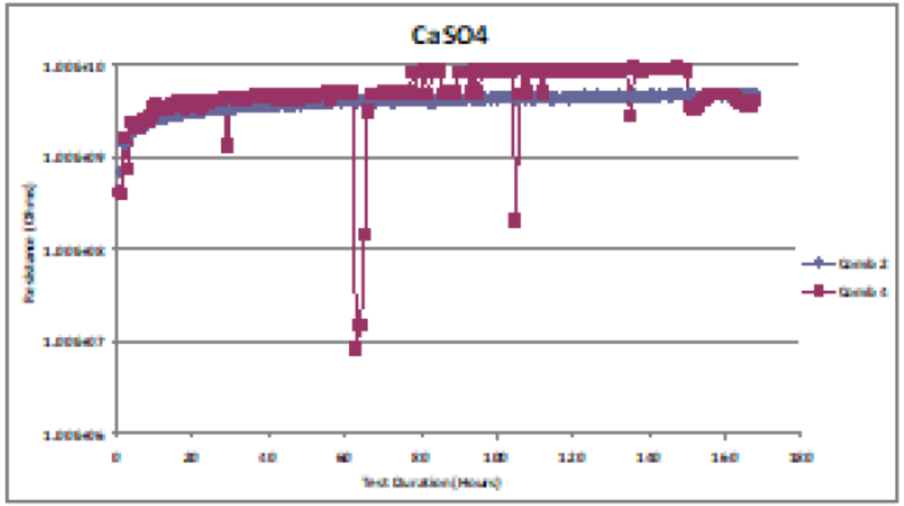

K2SO4

SIR Results for Calcium Salts

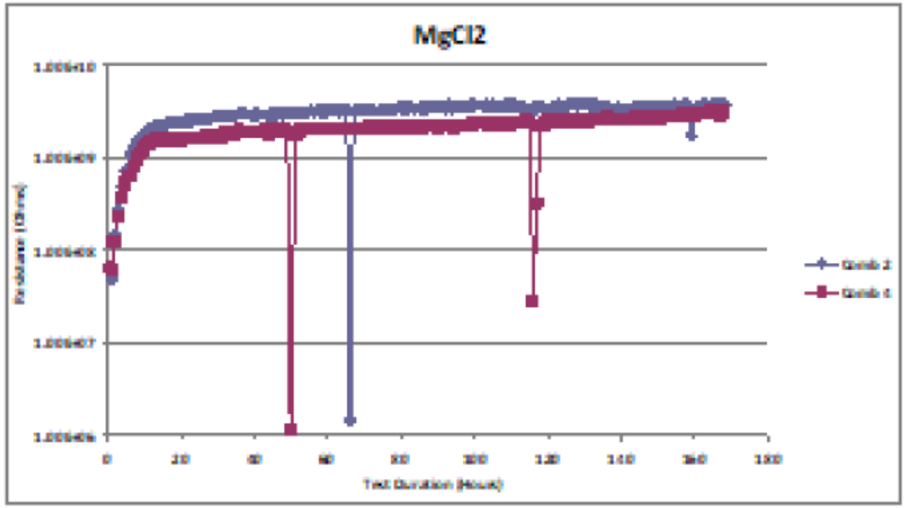


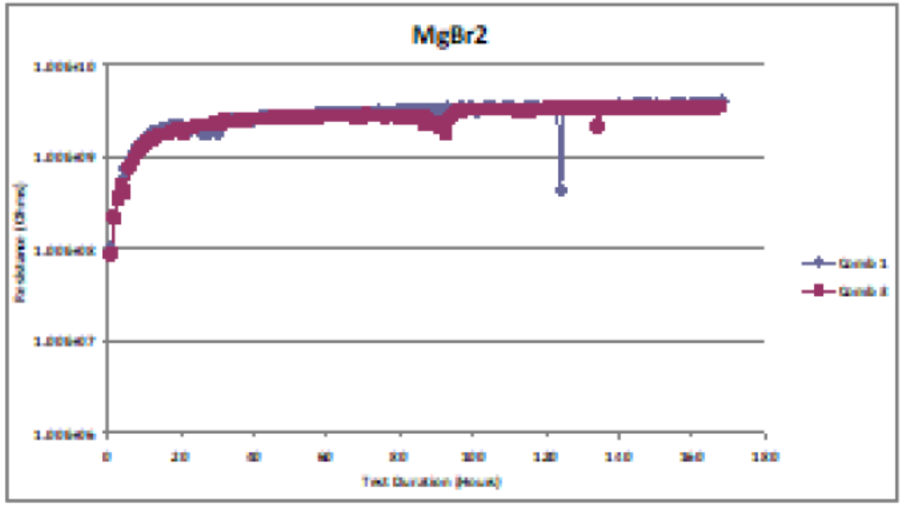


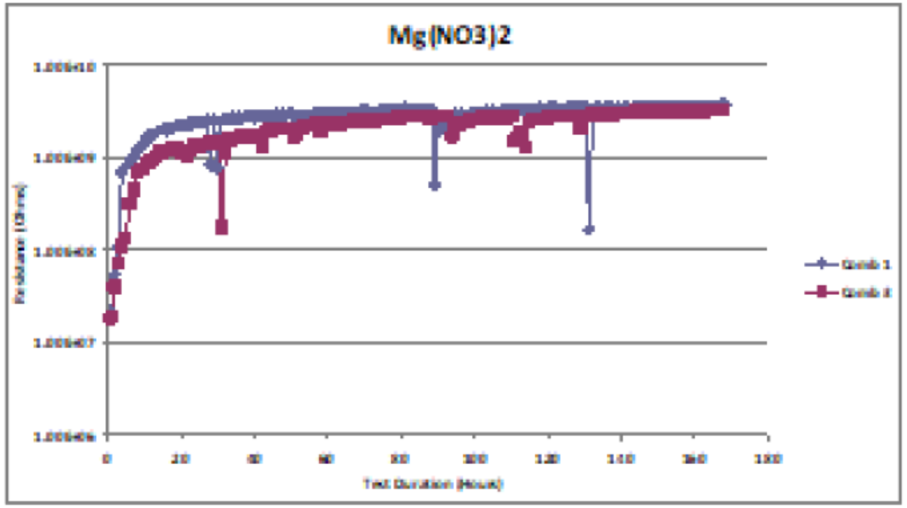


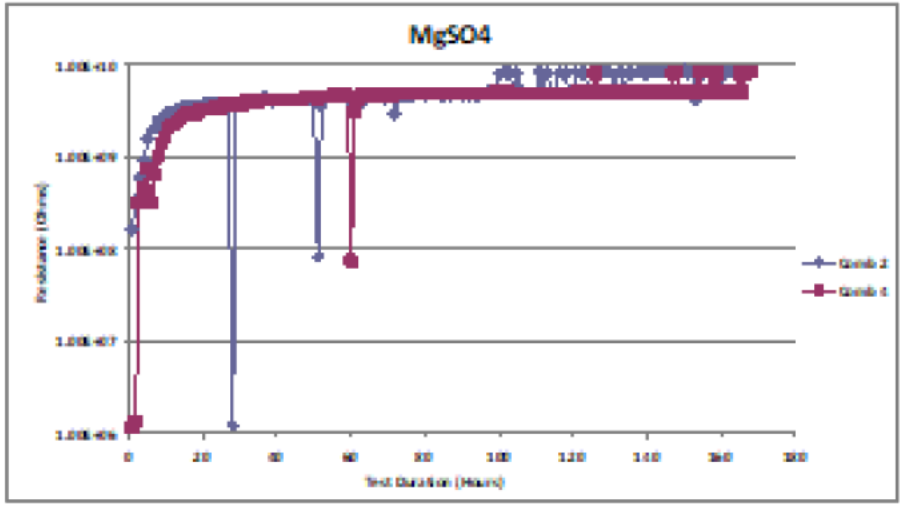


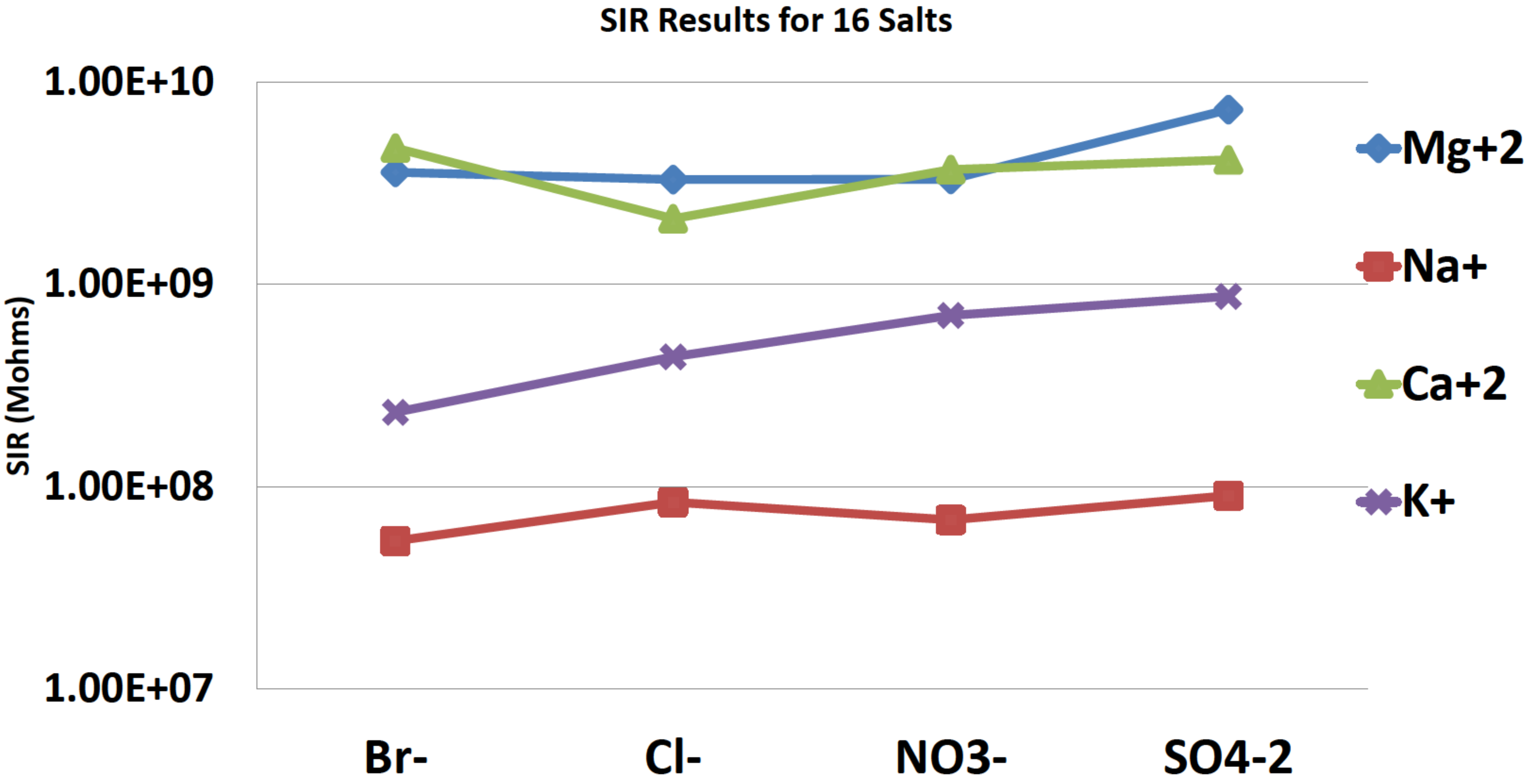
SIR Results for Magnesium Salts

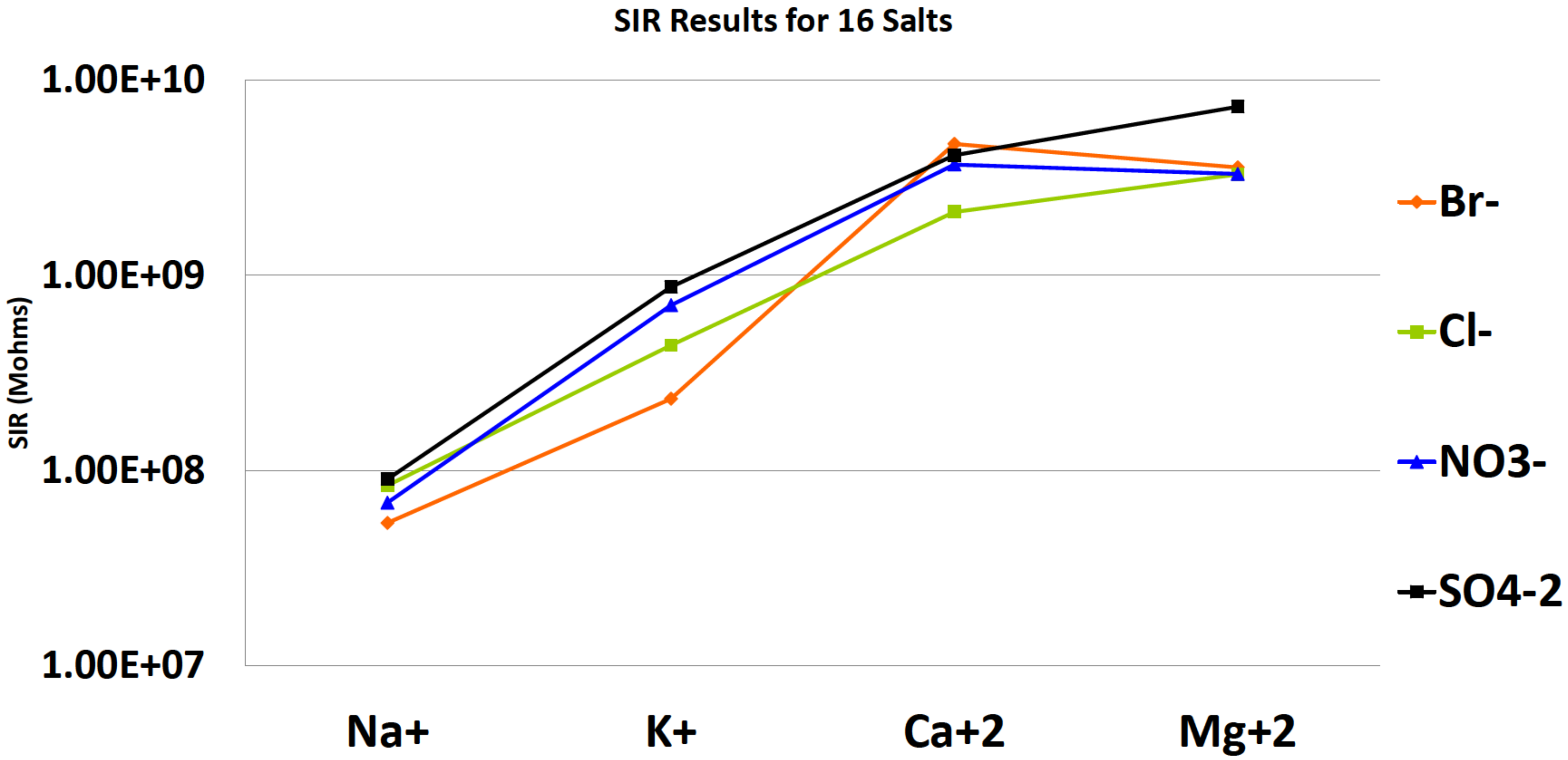

MgCl2

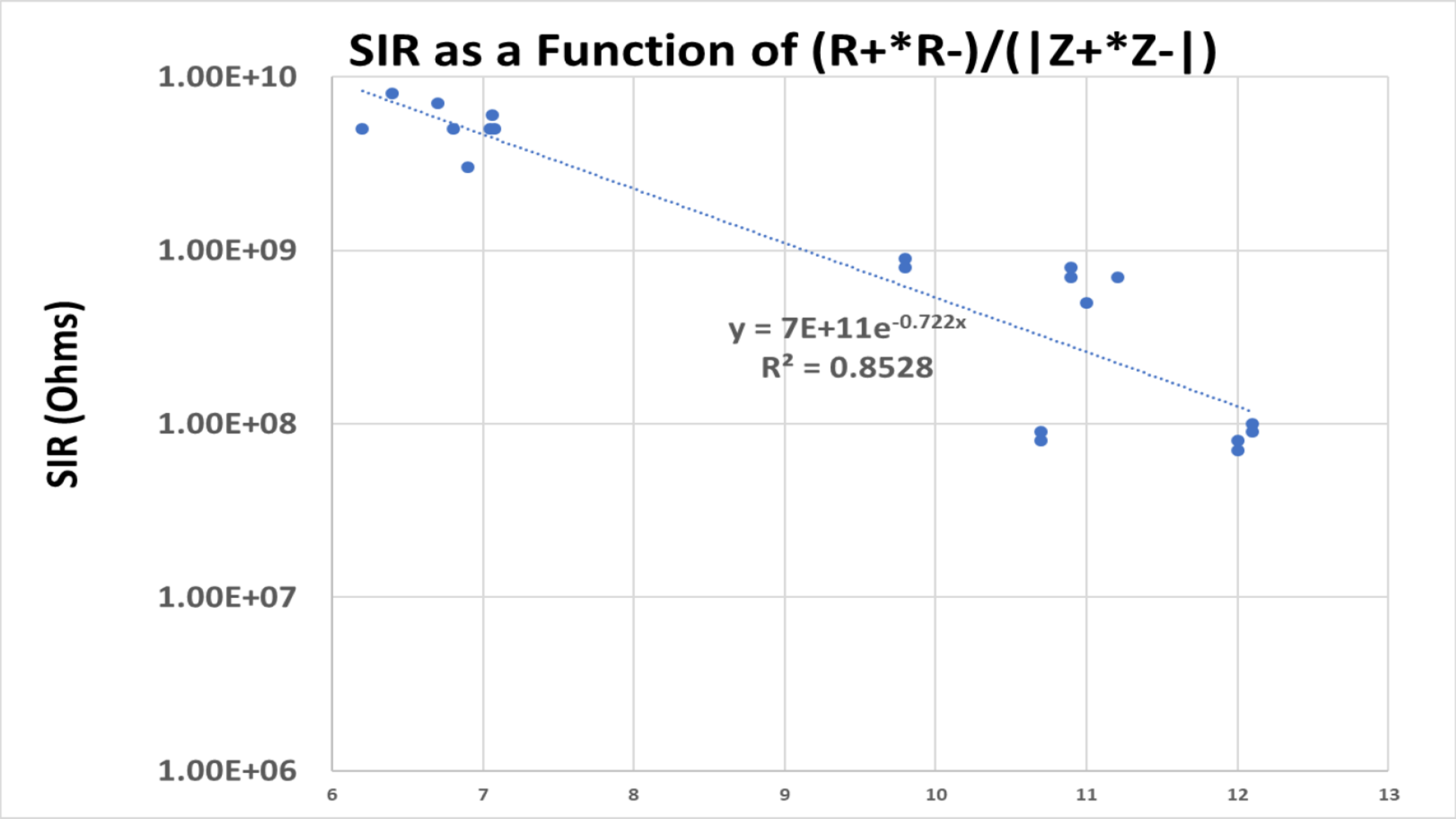

MgBr2


Mg(NO3)2


MgSO4


SIR Results for 16 Salts
SIR (Mohms)
1.00E+10
1.00E+09
1.00E+08
1.00E+07
Br-
Cl-
NO3-
SO4-2
Mg+2
Na+
Ca+2
K+


SIR Results for 16 Salts
SIR (Mohms)
1.00E+10
1.00E+09
1.00E+08
1.00E+07
Na+
K+
Ca+2
Mg+2
Br-
Cl-
NO3-
SO4-2


SIR as a Function of (R+*R-)/(|Z+*Z-|)
SIR (Ohms)
1.00E+10
1.00E+09
1.00E+08
1.00E+07
1.00E+06
6
7
8
9
10
11
12
13
y = 7E+11e^-0.722x
R² = 0.8528

IC and OES-ICP Parameters

- Guard column 4mm
- 4mm column
- 4mm suppressor
- Injection loop 20um
- Eluent: 8.0mM Na_2CO_3/1mM $NaHCO_3$
- Eluent flow rate 1mL/min

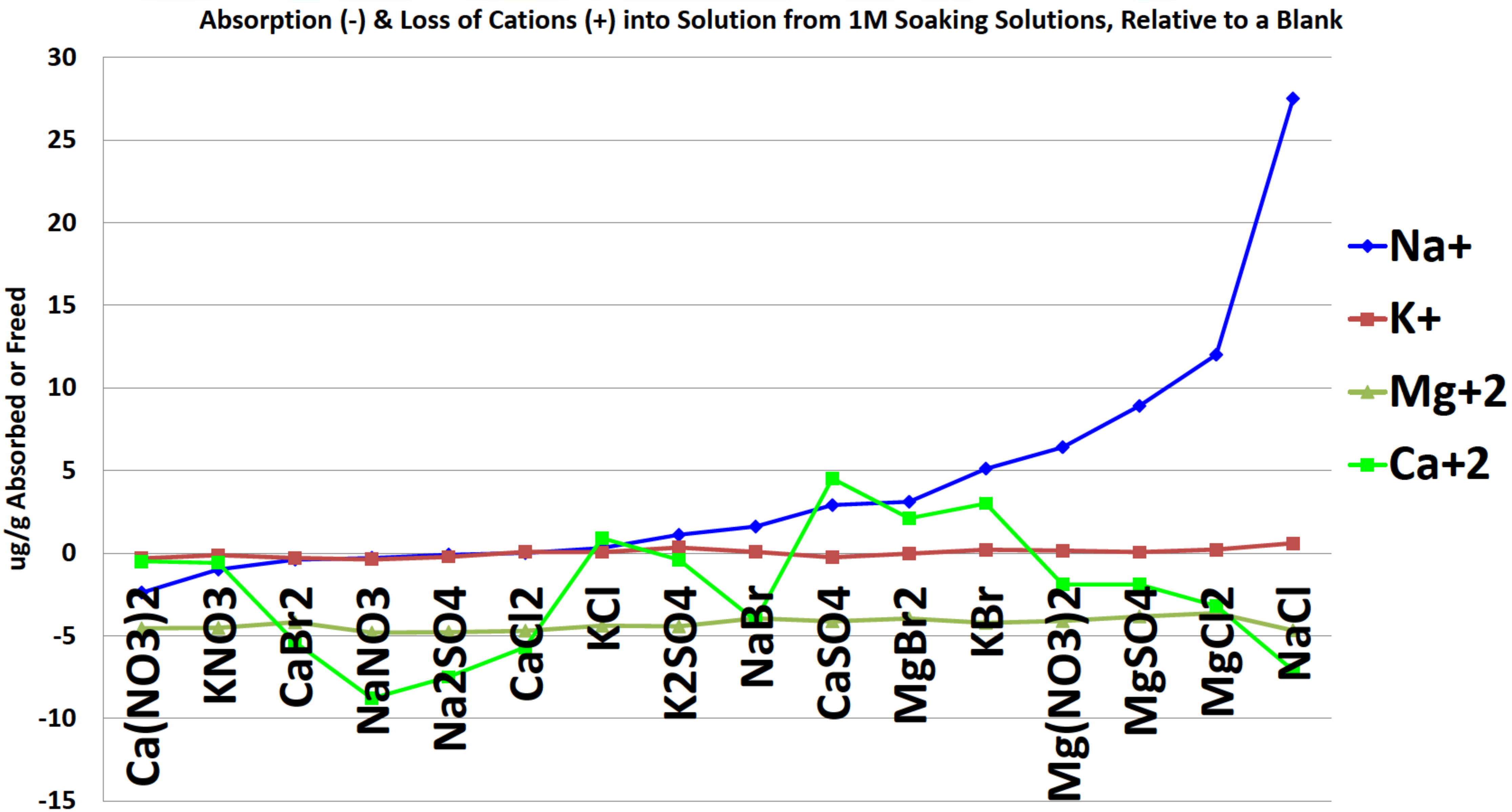
Plasma gas flow: 15mL/min

Auxiliary gas flow: 0.2 mL/min

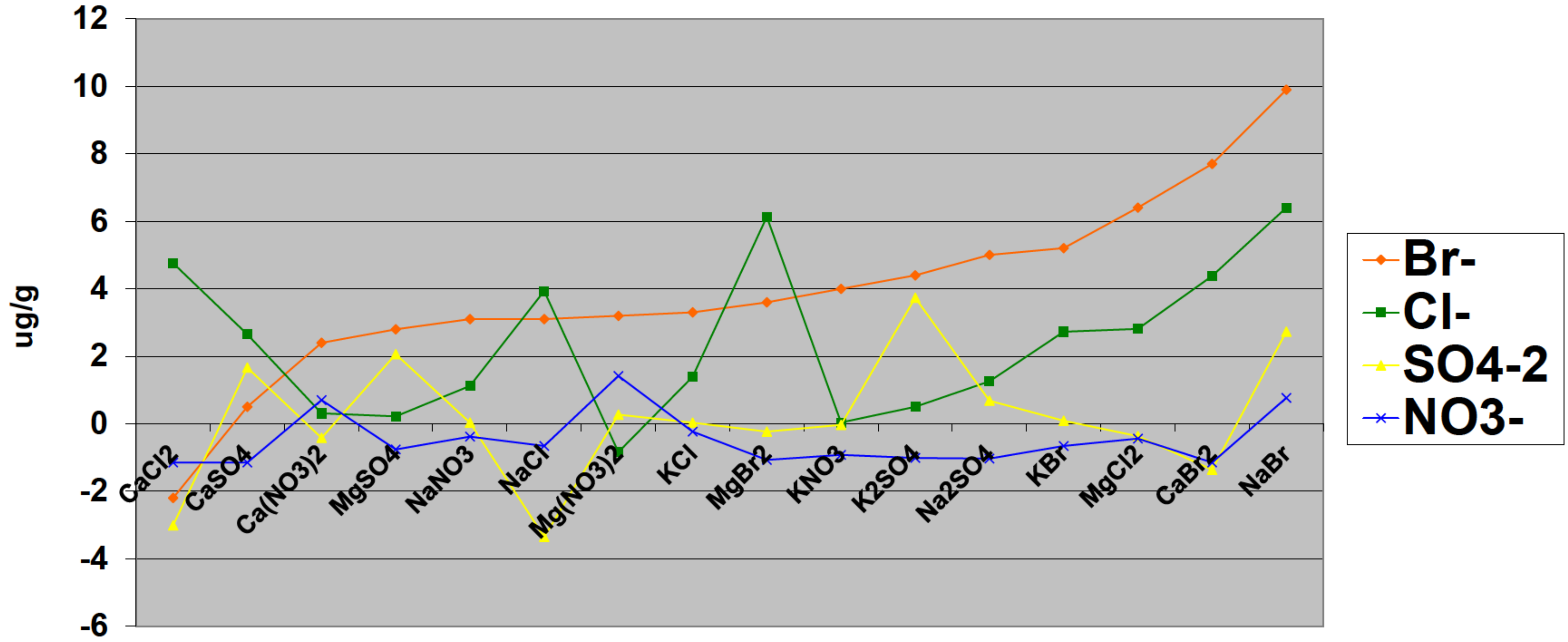
Nebulizer gas flow: 0.5 mL/min

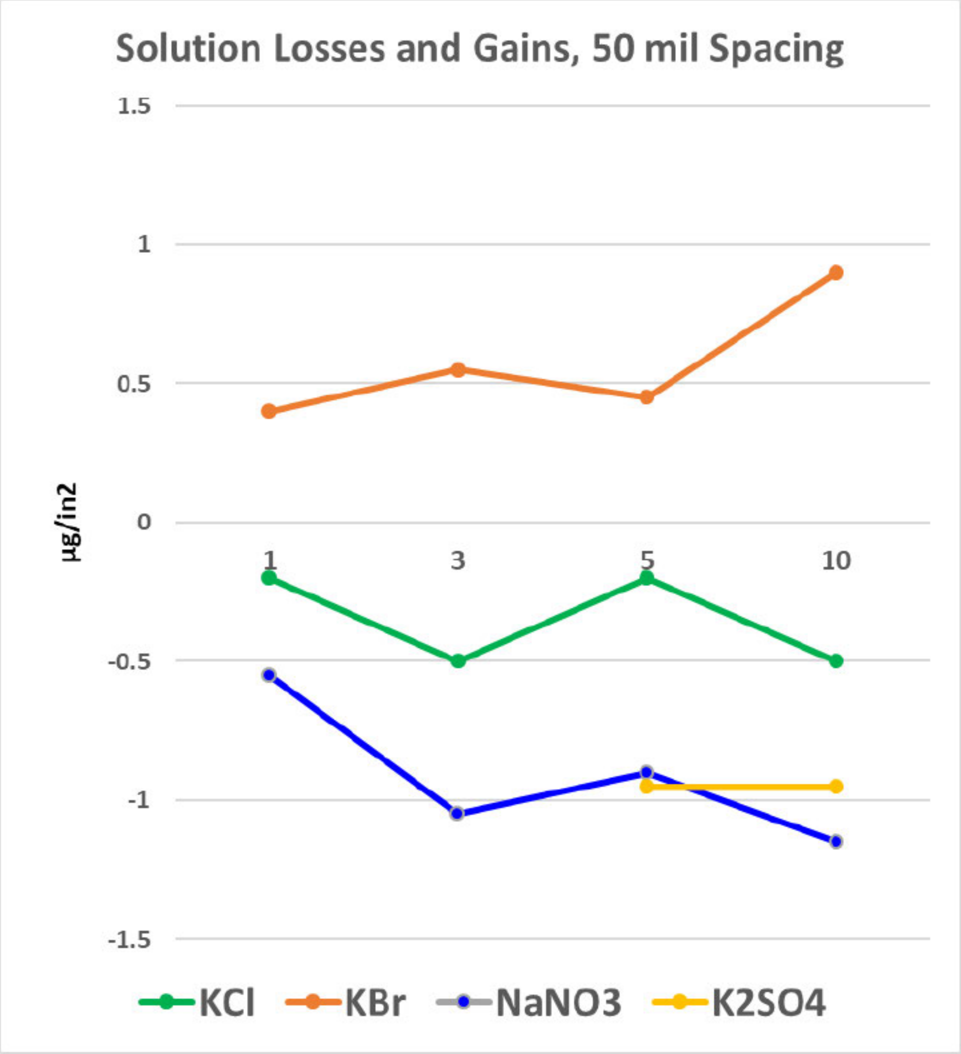
RF: 1450 Watts

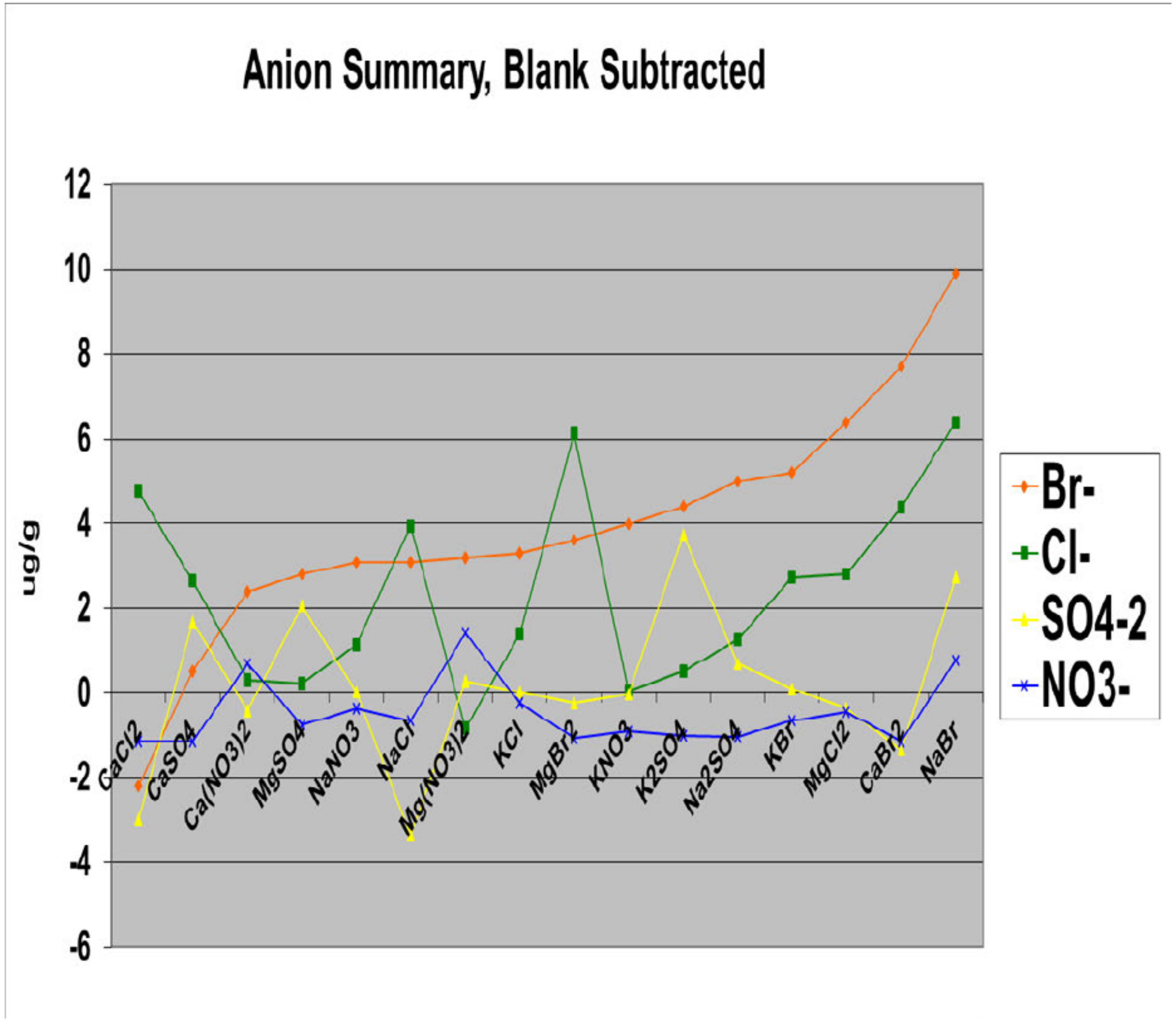
Pump speed: 2mL/min


Absorption (-) & Loss of Cations (+) into Solution from 1M Soaking Solutions, Relative to a Blank
ug/g Absorbed or Freed
30
25
20
15
10
5
0
-5
-10
-15
Ca(NO3)2
KNO3
CaBr2
NaNO3
Na2SO4
CaCl2
KCl
K2SO4
NaBr
CaSO4
MgBr2
KBr
Mg(NO3)2
MgSO4
MgCl2
NaCl
Na+
K+
Mg+2
Ca+2

Anion Summary, Blank Subtracted




Solution Losses and Gains, 50 mil Spacing


Anion Summary, Blank Subtracted

Summary of SIR and Leaching Study Results

	SIR	Leaching into Solution
	Corrosive	
Cations	$Na^+ > K^+ > Ca^{+2} \geq Mg^{+2}$	$Na^+ > K^+ > Ca^{+2} > Mg^{+2}$
Anions	$Br^- > Cl^- > NO_3^- \geq SO_4^{-2}$	$Br^- > Cl^- > SO_4^{-2} > NO_3^-$
Anions[15]	$Cl^- \geq NO_3^- \geq SO_4^{-2} > Br^-$	$Br^- > Cl^- > SO_4^{-2} \geq NO_3^-$

Overall Conclusions

- 7.5 x 10^{-4} M is a reasonable concentration for SIR studies
- Halides are more corrosive than the other 2 common anions
- Alkali metal ions are more corrosive than alkaline earth cations
- The order of cation corrosivity matches the order of leaching from FR4 laminate

Overall Conclusions

- Bromide leaches out of FR4 laminate
- The relationship between anion corrosivity and leaching is not as clear
- There is, as yet, no definitive equation for predicting SIR corrosion results

Questions?