Update to CR 13-04: Best Practices for Protecting DOT Equipment from the Corrosion Effect of Chemical Deicers

Final Report

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List of Abbreviations

ASTM	American Society for Testing and Materials
SAE	Society of Automotive Engineers
SSC	Salt Spray Chamber
$NaCl$	Sodium Chloride
$CaCl_2$	Calcium Chloride
$MgCl_2$	Magnesium Chloride
DDI	Distilled Deionized
DOT	Department of Transportation
EIS	Electrochemical impedance spectroscopy
NACE	The National Association of Corrosion Engineers
mpy	Mils per year (corrosion rate unit)
SMV	Snow Management Vehicle

Executive Summary

Department of Transportation (DOT) vehicles that are extensively used in snow management on the roadways, streets, and parking lots can receive excessive damage due to corrosion attack caused by humid, salt-laden environments. Therefore, to protect those DOT snow management vehicles (SMVs) and equipment, it is necessary to apply protective coatings on them. Although when received as new they have manufacturer's applied paints on them, it is often important to further protect their bodies with the help of additional protective coatings.

This project facilitated the research required to find coatings (currently available in the market) that are suitable for corrosion protection of the DOT SMVs and equipment. The research was done by conducting surveys, selecting products, performing standardized experiments, and discovering the best products in terms of corrosion protection after analyzing the results. A survey analysis as Task 1 was conducted, which found details on DOT snow management practices including the use of pre-wetted salts, corrosion inhibitors, materials used in equipment and their corrosion protection methods, washing and drying of SMVs and equipment, re-designing components to reduce corrosion losses, and the past experiences related to modes of corrosion protection. Followed by Task 1, another questionnaire (market analysis) as Task 2 was distributed, specifically focusing on the coatings used, their costs, mode of applications, and which ones were the best for several DOTs. Based on the responses of the two surveys, an experimental plan was developed, which highlighted the selected products to be tested, and the details of the testing procedures along with standard methods to be followed. The selected products included some sealants, lubricants, and an epoxy primer. To execute the experimental plan the selected products were applied to four types of materials (steel, stainless steel, alloys of aluminum, and copper) by carefully following the technical data sheets (TDS) of all products. They were then tested for their corrosion resistance by conducting accelerated cyclic tests including electrochemical impedance spectroscopy (EIS) and salt spray test (SAE J2334). Furthermore, some of the material-product combinations were also tested for their hardness and adhesion strengths. After the data yielded from these tests was analyzed, the results were compiled and compared.

Two comparison charts were developed based on the test findings and are added to the end of the report. Many details and research highlights were added to the Appendices. It was found that Fluid Film® provides the maximum corrosion protection to steel and aluminum alloys, as it showed remarkable performance in both electrochemical impedance spectroscopy (EIS) and salt spray testing. Based on EIS, the zinc-rich epoxy primer Aquapon® remained the best choice for steels in $NaCl\text{-}CaCl_2$ salt blend and also successfully passed all 60 cycles of SAE J2334, which are equivalent to 5 years in real-life corrosive environments. Although it costs more than Fluid Film for its mode of application and price per gallon, it has a higher resistance to abrasion, scratch, and indentations. Such coatings last longer than sealants and lubricants, which may or may not protect the metal surfaces in the long run. Sealants also do not have adequate adhesion strength and may not resist abrasion at all in sand, gravel, and snow-like conditions. Though Armour Seal® also showed excellent results in EIS and passed the salt spray testing for both stainless steel and aluminum alloys, its low adhesion strength and negligible hardness make it a vulnerable product to be used on DOT equipment and SMVs. Lubra Seal® is not recommended for steels over 6 months and must be reapplied at least twice a year. Minimum coating inspection is required for zinc-rich epoxy primer paint, while sealants and lubricants must be regularly inspected and re-applied

whenever necessary. Compared with Lubra Seal® and Fluid Film®, both Armour Seal® and Aquapon® are heavier products.

For copper, the selected products were Deox-IT (Gold G-series) and Permatex® di-electric grease. Both products failed to pass the 60 cycles of the salt spray test, starting to show signs of failure after only two weeks of SAE J2334 testing, which means in the field they may last only for 6 months after application. Their EIS results also did not indicate that they can resist corrosion in salt-laden wet/dry environments for long. Therefore, regular inspection of copper parts covered by these two lubricants must be done to avoid any sudden failures.

Finally, based on the survey responses, it is deduced that most of the DOTs do not dry the vehicles and equipment after washing or when exposed to wet conditions. Although it could be difficult to dry hundreds of vehicles on a daily basis, drying can prevent localized corrosion attacks and save money in the long run. Since the costs of corrosion losses within the DOTs are not known, it is difficult to conduct an accurate cost-benefit analysis for the installation of drying stations. Moreover, water with higher mineral content may aggravate corrosion on steel and could be treated before being used for the washing of vehicles.

Chapter 1: Introduction

1.1 Problem Statement

Since the issuance of "*CR 13-04 - Manual of Best Practices for the Prevention of Corrosion on Vehicles and Equipment (2015)*", new anticorrosion products have come to the market and there is increased use of non-sodium chloride deicers; as such, there is an urgent need to update some of the key sections in the manual. There are many coating technologies suitable for preserving the value of equipment asset in chloride-laden environments (Qian et al. 2015), but only some of them migrated into the market. There is increased use of beet juice/sodium chloride blends by highway agencies and the implications on equipment corrosion remain unknown at this stage. Our nationwide survey of highway agencies (Li et al. 2013) identified four anticorrosion coating products and the laboratory tests identified one best-performing coating (Rust Bullet®) in reducing the corrosion effect of magnesium chloride solution to carbon steel. Our recent laboratory study (Wu et al. 2016) revealed that the Rust Bullet® coating showed significant benefits in protecting the metallic substrates, and the cyclic exposure to magnesium chloride led to greater coating deterioration than did sodium chloride.

In this context, this Clear Roads (www.clearroads.org) project is designed to review department of transportation (DOT) members' current practices of using coatings to mitigate equipment corrosion and more importantly to point out which ones worked or failed. This is much needed to obtain a better understanding of what is needed to protect the large investment in equipment assets, which procedures were implemented, and whether or not they produced the anticipated results. To assist agencies and private contractors in selecting the proper coating(s), this project will also conduct tests to evaluate and compare the older products with the newer ones. Moreover, the project team will conduct a market analysis and a survey of Clear Roads' members' agencies, which would determine the products in use, reasons of their selection as well as their effectiveness, cost, ease of application, durability and any drawbacks.

Overall, this project aims to produce new data and renewed understanding to update the anticorrosion coating section of the 2015 Clear Roads Project 13-04 publication (Honarvar Nazari et al., 2015), *Manual of Best Practices for the Prevention of Corrosion on Vehicles and Equipment used by Transportation Agencies for Snow and Ice Control*. This would thus provide guidance to encourage good practices and procedures that may be implemented. The potential audience may include Managers, Middle Managers, Foreman and Operators; and the information will likely be integrated later into equipment specifications, purchase decisions of chemical deicers, guidance on equipment paint and washing practices for post-storm clean-ups, etc. Most of the emphasis is still required to be laid on finding optimum coatings for in-use DOT equipment and compare their efficiency in terms of corrosion protection.

Note that Clear Roads (www.clearroads.org) is an ongoing pooled-fund research program aimed at rigorous testing of winter maintenance materials, equipment, and methods for use by highway maintenance crews.

1.2 Background

Sodium chloride, calcium chloride, magnesium chloride and salt brines are the most commonly used chemical deicers used in maintaining the safety and mobility of winter highways in northern states. Though very effective and relatively affordable, they are quite corrosive, particularly to the metallic components of snow management vehicles (SMVs), equipment and machinery used to store, transport, and apply chemical deicers to roadways. Cast iron, carbon steel, stainless steel and aluminum are the primary metals used in the fabrication of the various components; copper electrical wiring is also prone to corrosion when exposed to the chemical deicers (Shi, Li, et al., 2013). One study published in 1992 estimated that road salt imposed infrastructure corrosion costs of at least \$615/t, vehicular corrosion costs of at least \$113/t, aesthetic costs of \$75/t if applied near environmentally sensitive areas, plus uncertain human health costs (Vitaliano 1992). Arguably, the cost of vehicular corrosion should have decreased since then, as vehicles today feature improved corrosion resistance via better design and materials selection.

Based on a nationwide survey we conducted (Li et al., 2013), for agencies that report deicer corrosion to equipment as a significant issue, the total cost of current corrosion management and corrosion risks related to deicer exposure was estimated to average \$1.06 million and \$14.05 million per year, respectively. On average, deicer exposure leads to risks in six areas: 17.3% depreciation in equipment value, 8.5% increase in equipment downtime, 11.9% reduction in equipment reliability, 17.3% reduction in equipment service life, 19.6% increase in premature repair and replacement, and 1.5% risk in safety from faulty parts on equipment.

Many highway agencies still use non-inhibited chemical deicers for winter road maintenance (WRM) operations. Products that are regarded as corrosion inhibitors are also used by many state, municipal, county, and other transportation agencies as additives to chloride-based deicers to reduce their corrosive effects on equipment, vehicles, and metallic infrastructures (Shi, Fortune, et al., 2013). Such infrastructures include bridges, overpasses, guardrails, signage, signals, light poles and other roadway fixtures. Notably, highway agency vehicles and equipment are at great risk of deicer corrosion due to cumulative exposure of the metals to deicers; for instance, state DOT trucks have observed the reduction of service life from 15 years to 12 years (Shi et al. 2016).

Although it is a common practice to wash the vehicles and equipment after every storm, whenever possible, it is often difficult to thoroughly clean every exterior surface. Often small grains of salt, treated sand and other solid materials are lodged in deep, hard-to-reach locations, especially on the frame. The thoroughness of washing can also vary considerably among operators and from storm to storm. Our recent laboratory study (Nazari et al. 2017) revealed that even with a salt remover to enhance the washing practice, the corrosion rate of aluminum alloy exposed to magnesium chloride deicer remained high.

For an “average agency” (e.g. a northern state DOT with an average size of fleet asset), the preliminary analysis by Shi et al. (2016) estimated a benefit/ cost ratio of 13.2 in further improving deicer corrosion control of DOT equipment fleet. For preventing the deicer corrosion of DOT equipment, anticorrosion coatings, spray-on corrosion inhibitors (Wu et al. 2016) and salt removers ((Nazari et al. 2017) were identified as the best practices (Shi, Li, et al., 2013). Proper application of anticorrosion coatings is critical for ensuring lower maintenance and repair costs, as well as minimizing the loss of available service time.

1.3 Actionable Guidance

Section 5.4 of the manual in the 2015 Clear Roads Project 13-04 publication (Honarvar Nazari et al., 2015) provides guidance on the use of coatings for corrosion control and protection of DOT equipment, with the focus mainly on sodium chloride deicer. For intricate geometries (like the bodies of DOT vehicles used in winter maintenance), electrochemical deposition as a coating method is effective. It is a generally understood that thicker coatings would last long, but it is not the case every time. An aluminum coating only ~2 µm thick would last longer than a ~10 µm dadmium or chromium coating on steel. Notably the coating's performance hinges on how well the surface is prepared prior to coating, as nearly three-fourth of coating failures occur due to poor surface preparation. The surfaces prior to the coating must be free of contaminants. Sand or grit blasting is the most efficient surface preparation technique (see Figure 1.1), and salt removers may be used as an alternative. Rust converters are helpful when the surface to be coated, is badly rusted. They can replace the less effective surface preparation methods (such as hand or mechanical wire brushing) and may also act as a primer coat prior to the coatings. A successful plan for painting a vehicle's body may include surface blasting, use of primers and then application of a top coat with a suitable thickness. Total thickness of all coats should not be less than 8 mm (Honarvar Nazari et al., 2015).

Figure 1.1 DOT vehicles - Sand blasted, had a primer coat and then painted (Honarvar Nazari et al., 2015)

It is important to update the Section 5.4 (Coatings for corrosion protection) in the manual to add information on the currently available protective coatings that would be suitable for use from cost, effectiveness, and long-lasting perspectives.

1.4 Deliverables

1.4.1 Surveying the State DOTs

The project's main focus was to identify the various coatings available on the market, from 2013 to the present, and conduct side-by-side comparison testing based on the application method. To do so, the research team conducted two surveys within State DOTs in the northern U.S. and a few other agencies associated with material protection and transportation departments. The first survey (Task 1) was heavily based on questions regarding the materials used in SMVs, snow control chemicals, use of corrosion inhibitors, winter maintenance practices, current use of protective coatings, washing and drying of SMVs, components, and equipment prone to corrosion, redesigning of components for corrosion prevention, and lessons learned in the past. However, the second survey (Task 2) was focused on extracting information based on the coatings used by State DOTs and other agencies to protect their SMVs and equipment from corrosion by various deicers.

1.4.2 Devising and executing the experimental plan

Based on the results gathered from both Task 1 and Task 2, an experimental plan was developed, which defined the scope of the experimental plan and the selected products to be tested on four types of materials – two ferrous-based and two non-ferrous. Steel and stainless steel were the two ferrous-based alloys, whereas aluminum alloys and copper (99% pure) were the non-ferrous materials. Coatings that are used by most DOTs for any given material were selected to be tested for various characteristics.

The methods used to test the selected coatings were mostly identified in the project's proposal. The aim was to test the coatings in rugged salt-laden and corrosive environments by using accelerated tests, for instance, electrochemical impedance spectroscopy (EIS) to test corrosion resistance of coatings in an accelerated corrosion environment, and cyclic salt spray test that can project a 5-year performance of coatings against salt-laden corrosion conditions. Furthermore, an adhesion test to assess the adhesion strength of coatings on materials, and a hardness test to validate whether coatings can last longer in abrasive conditions, were also conducted for a few selected products.

1.4.3 Developing comparison charts

After all the results were gathered and analyzed, a comparison chart was developed, which aims to help DOT personnel understand which coatings could be more useful in certain conditions and how to apply them properly. Moreover, based on the conclusions drawn and data gathered from this research project, section 5.4 of the Manual is updated.

Chapter 2: Project tasks – An overview

The project involves various tasks defined in the proposal and completed in full. A brief overview of each of those tasks is provided in this section.

2.1 Survey Analysis – An overview

Survey Analysis (Task 1) was conducted to gather information mainly about the materials used in State Department of Transportation (DOT) snow management vehicles (SMVs), the risk of corrosion for those SMVs and their components, whether or not DOTs redesign SMV components to avoid corrosion, washing and drying cycles for DOT SMVs, anti-corrosion coatings used for DOT equipment and SMVs, and any past experiences regarding corrosion of DOT equipment and SMVs. Detailed responses are added to Appendix B. However, this section presents the key highlights of Task 1.

2.1.1 Respondents – Geographic details

While a few responses were incomplete and lacked information about the agencies of respondents, most were thorough and with all of the questions answered. Figure 2.1 shows the US state members (DOTs/agencies) who responded to this survey. A few responses with no information regarding the agencies and partially completed are not counted in the total number of responses. Furthermore, the Ohio DOT recorded their response twice. In total about 20 different responses were gathered from various State DOTs and other agencies.

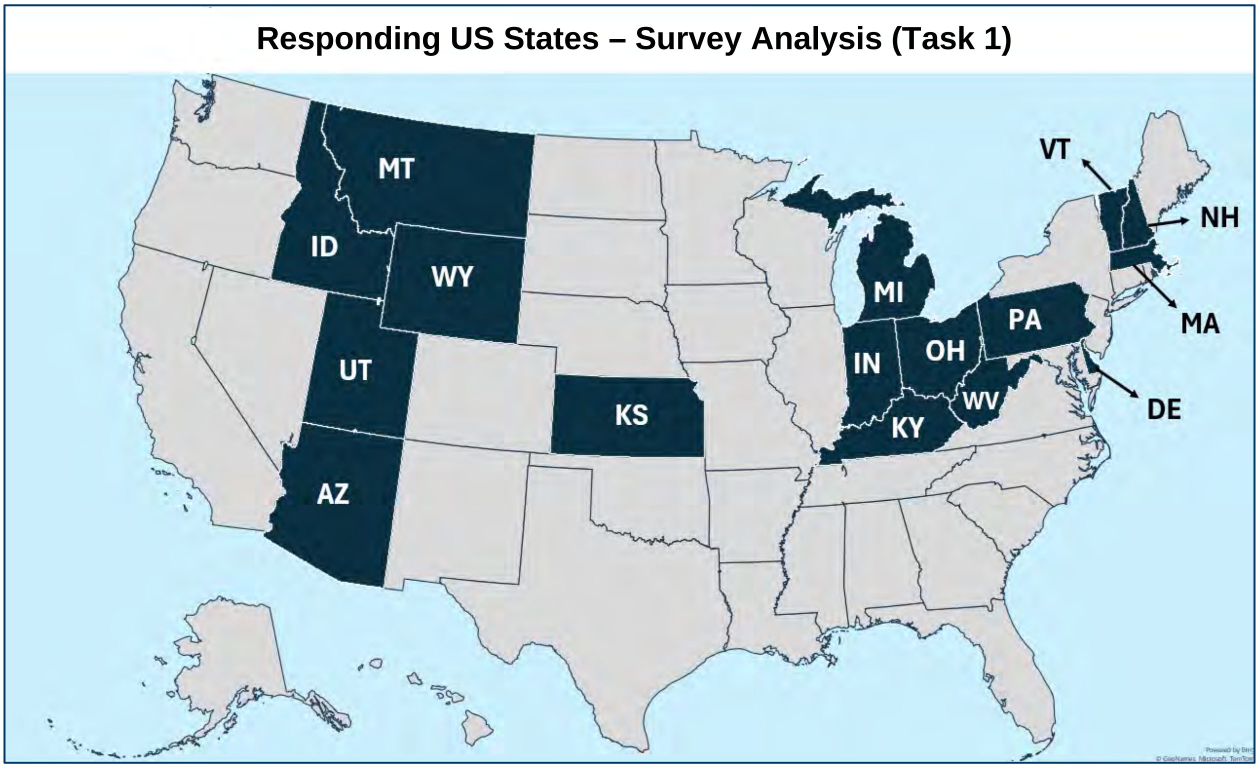

Responding US States – Survey Analysis (Task 1)

Figure 2.1 The U.S states (DOTs/agencies) responded to the survey questionnaire – Task 1

2.1.2 Questions asked – Background & Importance

Detailed responses to most of the questions are provided in Appendix B. However, here only some of the important findings from the Survey Analysis are discussed.

2.1.3 Questions regarding snow and ice control practices

Questions related to snow and ice control practices revealed that more agencies are doing pre-wetting of solid salts compared to the use of dry salt only, which may aggravate corrosion if corrosion inhibitors are not used in the liquids (pre-wetting agents). Notably, the majority of the agencies (52%) use corrosion inhibitors (e.g., Beet Juice) in snow-melting products.

2.1.4 Questions regarding agency vehicles and equipment

From questions regarding agency vehicles and equipment, we found that carbon steels are still the most widely used followed by stainless steel, whereas magnesium alloys are the least used by the agencies. The importance of this question is linked with the high susceptibility of carbon steels towards corrosion. Later in the survey a question regarding redesigning equipment to avoid corrosion revealed that many agencies replaced several carbon steel parts with stainless steel. The DOT equipment that is at risk of being corroded was ranked and we found that dump trucks, liquid deicer applicators, front-end loaders, and hoppers are at the highest risk of being corroded. More specifically the top five components that are at high risk of being corroded include brackets and supports, frames, fittings, brake drums and discs, and electrical components (e.g., wiring). Table B 2 in Appendix B provides those rankings.

Four agencies often replace their vehicles due to corrosion damage within 10 years of first use. While 9 responses said they do not replace their vehicles prematurely solely due to corrosion issues.

Questions regarding corrosion management practices

Sixty-two percent of the respondents said they have redesigned the components to avoid corrosion mostly by replacing materials that are prone to corrosion with corrosion resistance materials. Table B 4 in Appendix B has the complete details, and the most common redesigning was replacing steel with stainless steel for several equipment/components. Corrosion-related vehicle inspections are carried out mostly twice every year or after each storm.

Questions regarding washing & drying

Ninty percent of agencies have implemented a washing program for their SMVs, which is mostly based on washing after every storm. Specifics on washing cycles, frequency, and any associated policy are added to Tables B5 to B7 in Appendix B.

Types of water may affect the rate of corrosion for certain materials. The Handbook of Corrosion Engineering (Roberge, 2000) has a whole chapter on how different water sources and their distinct properties (e.g., dissolved oxygen, dissolved mineral content, and so on) can impact metals that come in contact with them. One important aspect of this project is to know how much water contact is made with the SMVs and if it is dried properly or not. Also, it was imperative to know what type of water is used in washing, because some types could be more corrosive to steel, stainless steel, and aluminum alloys than others (ASM Handbook Committee & ASM International, 1978). In industries, often water purification and conditioning techniques are followed to make sure the water is fit to use to avoid maximum corrosion losses. The presence of chlorides, free mineral acids, and dissolved carbon dioxide can affect corrosion rates for steels (Roberge, 2000). The ASM Metals Handbook of Corrosion (Volume 13) also discusses the importance of water chemistry and properties that affect corrosion rates for steel and stainless steel. Wet corrosion is caused by water pockets, or layers of water formed on metal surfaces after rain, spray, or dew drops (ASM Handbook Committee & ASM International, 1978). Moreover, atmospheric factors can contribute to atmospheric corrosion and carbon steel can lose more weight due to corrosion loss in one state than the other within the US (ASM Handbook Committee & ASM International, 1978).

The importance of drying the SMVs when they are parked and not in use for days comes from the fact that the size of a water molecule is 2.75 angstroms (^{o}A) which is 0.275 nm. The pore size of an average paint film or rust protective film lies somewhere between micro (1μm to 100μm) to nano (10 nm to 500nm, for more advanced coatings). This means most of the coatings and paints may not be able to stop water from being penetrated or absorbed through the surface of paints, ultimately reaching the metal surface. Therefore, the drying of SMVs (when not in use) after washing would make a great difference in minimizing corrosion.

Most agencies use tap water for washing followed by a post-washing treatment done by only a few. Over 65% of agencies in the survey do not do any drying of the vehicles. The ones that are drying the SMVs are relying on a self-drying mechanism. Note that if there are over a thousand vehicles (2,500 to 5,000 in some agencies), drying all of them after washing and when not in use could be a cumbersome job.

Questions regarding anti-corrosion coatings

Many respondents (43%) indicated that they do not know whether or not additional coatings (other than OEMs) are applied to the SMVs, whereas 33% of the responding agencies do not apply them and only 23% make use of additional protection on SMVs. The use of fluid film on SMVs and their components is common. Those coatings for additional protection are mostly applied manually by spray guns or paint brushes. Cost was the top reason for not applying additional coatings. Fluid Film® was named the best additional coating in terms of corrosion protection and the most used onsite coating; however, metalizing and a proper coating system were found longer lasting.

In the end, the responding agencies shared their past experiences and lessons learned. Once again, Fluid Film was found as an effective product by a few agencies. However, the typical service life of Fluid Film was said to be 6 months, whereas a good paint system life was mentioned as 5 years.

2.2 Market Analysis – An overview

A market analysis (Task 2) was conducted to gather information specifically related to the coatings or paints used on the DOT equipment and SMVs. The questions developed were generated after gathering results from the Survey Analysis. The total number of participants in this survey was 17 and a geographic map indicating the responding state members is given in Figure 2.2.

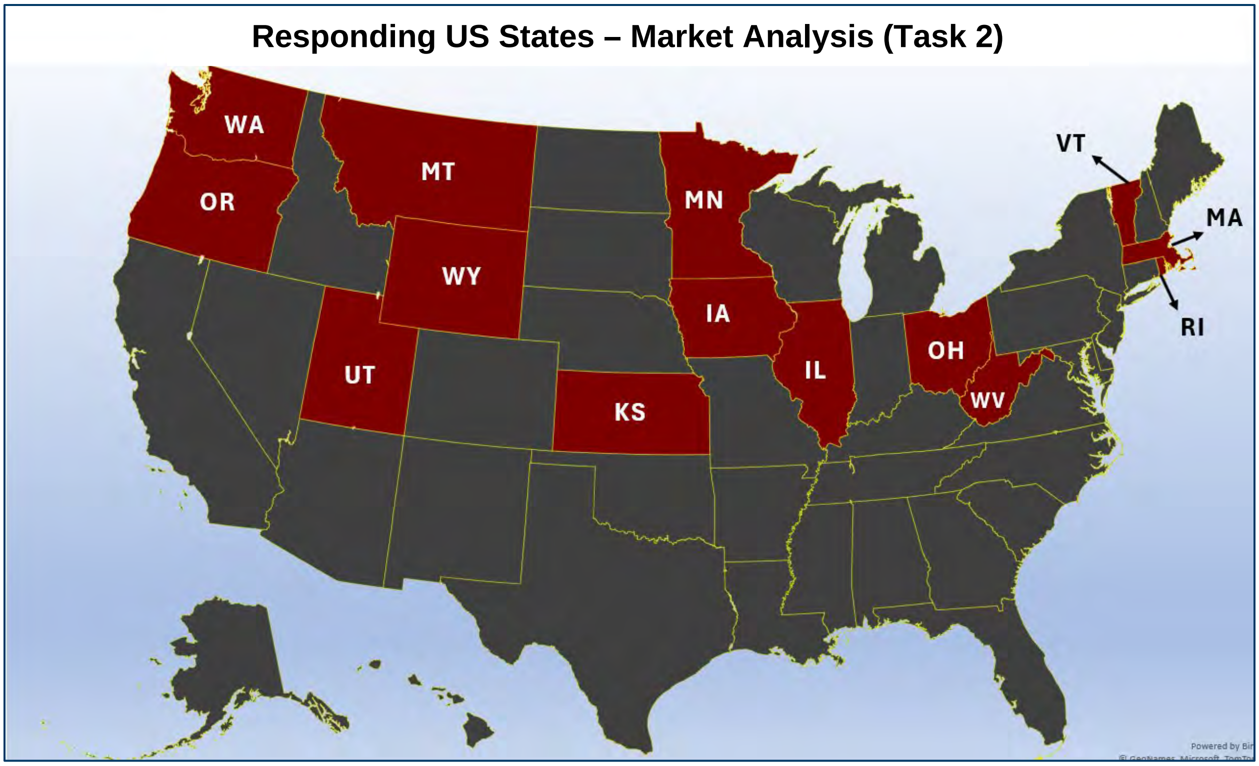


Figure 2.2 The U.S states (DOTs/agencies) responded to the survey questionnaire – Task 2

2.2.1 Benefits reaped from Task 2

The market analysis revealed that several coatings currently used by the State DOTs and other agencies on different materials. The benefit of this survey was that it remained focused on the coatings and therefore respondents had a greater chance to provide information on coatings-related specifics.

2.2.2 Important findings – Task 2

For the 4 different types of materials (steels, stainless steels, and alloys of aluminum and copper), most coatings are utilized for steels, followed by stainless steels. For steel, Fluid Film is used by most of the State DOTs and is applied at the lowest cost compared to other coatings used for steel, stainless, and aluminum alloys. Dielectric grease is widely used for copper and copper alloys (e.g., electrical wiring) and costs about $2.00 per square foot. Armour and Lubra seals are also used by several agencies, but they do not last longer and can be washed away easily. Fluid Film needs at least two applications per year but can be used in both hot and cold environments. Only a few use paint systems on truck frames and cabs, though they can last longer than lubricants and sealants. Ceramic coating was mentioned as a product lasting for 3 years, however, the same agency indicated that Fluid Film is the easiest to apply.

In the responses gathered from the question on coatings used in the last ten years, Fluid Film was mentioned 6 times in the 20 responses in total. The most common method of coating/paint application within the agencies is spray coating, followed by brush application. Conventional spraying is more common than airless spray. Thirty-one percent of agencies in the survey are using sandblasting as a surface preparation technique, before applying coatings on materials, while some also use acid cleaning and washing. The coatings are usually applied by in-house crews and only 29% of state DOTs have them applied by the contractors.

Overall, Fluid Film, Lubra Seal, Armour Seal, dielectric grease, and a zinc-rich primer (or galvanizing) turned out to be the most used products on various materials. Tables B 14 to B 27 in Appendix B provide some more details gathered from Task 2 – Market Analysis.

2.3 Development of experimental plan – An overview

Based on the results of Task 1 and Task 2, an experimental plan as Task 3 was developed to outline the selected coatings for each of the materials, and the standard test methods to be employed for evaluating each coating. The plan highlights the coatings selected for each material, as provided in Table 2.1.

Table 2.1 Selected coatings for different materials

Materials		Selected coatings	Manufacturer
Steel	1.	Lubra Seal ®	Rhomar Industries, Inc.
	2.	Fluid Film ®	Eureka Chemical Company
	3.	Aquapon ® | 97-670 series (zinc-rich epoxy)	Pittsburgh Plate Glass (PPG) Industries, Inc.
	4.	Kem Kromik ^{TM} 255 (primer finish)	Sherwin Williams ®
Stainless Steel	1.	Armour Seal ®	Rhomar Industries, Inc.
	2.	Lubra Seal ®	Rhomar Industries, Inc.
Aluminum	1.	Fluid Film ®	Eureka Chemical Company
	2.	Armour Seal ®	Rhomar Industries, Inc.
Copper	1.	Di-electric Grease	Permatex ®
	2.	DeoxIT ®	Caig Laboratories, Inc.

These coatings were selected based on tables generated from the Task 2 results, which are added to Appendix C (Tables C1 and C2). Only the top choices for each material were selected. Steel testing was carried on with only three choices since Kem Kromik^{TM} 255 was not available in the U.S.

To apply each of the selected coatings, technical data sheets (TDS) for each product were carefully followed and vendors were directly contacted whenever necessary. To apply both Lubra Seal® and Armour Seal®, a pistol grip spray gun was purchased from Rhomar Industries, Inc. Fluid Film® was sprayed using a handheld airless paint sprayer (Graco TrueCoat 360 single speed) and was also brushed gently whenever necessary for touching smaller coupons. For spraying heavy metal-based (zn-rich epoxy) primer (Aquapon®) on steel, a one-quart pressure pot with a conventional spray gun was used. The pressure pot was equipped with an agitator to mix the paint during painting constantly. Products selected for copper came with their application kits, however, Permatex® dielectric grease was applied with a soft paintbrush on cleaned coupons. Coupons were cleaned or sandblasted only if recommended by the vendors in TDS. Some of these coatings can be regarded as soft coatings or lubricants (such as Fluid Film, dielectric grease, and Deox IT), while others are hard coatings. Some of the highlights of the paint job done in the paint shop at Washington State University are shown in Figures C1 to C3 in Appendix C.

The experimental plan highlights the test procedures used to evaluate the performance of coatings. Table 2.2 presents those test procedures, along with the standard test methods, and types of alloys used for each test. The table also indicates the coupon sizes and the deicer solutions used for some test methods. The information regarding the coupon sizes in inches (in or ”) and alloy types were mostly extracted from the standard test methods (ASTM International, 2016, 2017, 2019, 2020, 2022; SAE International, 2016).

Table 2.2 Test procedures and standard methods for evaluating the selected coatings

Sr. #	Test procedures & standard methods	Materials	Alloy types	Coupon sizes	Deicer types
1.	Electrochemical Impedance Spectroscopy (EIS) ASTM G59 & ASTM G106 - 89	Steel	ASTM A1008, cold rolled	1 in x 2 in x 0.036 in	CaCl$_2$ + NaCl salt blend & MgCl$_2$ + Beet Juice blend
		Stainless Steel	AISI 430	1 in x 2 in x 0.035 in	
		Aluminum	6061 – T6*	1 in x 2 in x 0.032 in	
		Copper	C11000	1 in x 2 in x 0.032 in	
2.	Salt Spray Test (with scribed coupons) SAE J2334	Steel	ASTM A1008, cold rolled	3 in x 2.5 in x 0.036 in	0.5 % NaCl + 0.1 % CaCl$_2$ + 0.075 % NaHCO$_3$ + water balance**
		Stainless Steel	316, brush polish	3 in x 2.5 in x 0.036 in	
		Aluminum	5052 – H32*	3 in x 2.5 in x 0.032 in	
3.	Salt Spray Test (without scribed coupons) SAE J2334	Steel	ASTM A1008, cold rolled	1 in x 2 in x 0.036 in	0.5 % NaCl + 0.1 % CaCl$_2$ + 0.075 % NaHCO$_3$ + water balance**
		Aluminum	5052 – H32*	1 in x 2 in x 0.032 in	
		Copper	C11000	1 in x 2 in x 0.032 in	

Sr. #	Test procedures & standard methods	Materials	Alloy types	Coupon sizes	Deicer types
4.	Adhesion Test ASTM D4541	Steel	ASTM A36, plate	2 in x 2.5 in x 0.25 in	Not applicable
		Stainless Steel	316, mill finish	2 in x 2.5 in x 0.25 in	
		Aluminum	5052 – H32*	2 in x 2.5 in x 0.25 in	
5.	Pencil Hardness Test ASTM D3363	Steel	ASTM A36, plate	1 in x 3 in x 0.25 in	Not applicable
		Stainless Steel	316	1 in x 3 in x 0.25 in	
		Aluminum	5052 – H32*	1 in x 3 in x 0.25 in	
6.	Vickers Hardness Test ASTM E384	Steel (only for Aquapon®)	ASTM A36, plate	1 in x 3 in x 0.25 in	Not applicable

** T6 and H32 are the heat treatment routes that are used for aluminum alloys.*
*** The amount is added by weight percent*

The information on the sizes of coupons for each test was taken from the standard test methods listed in Table 2.2. The $CaCl_2$ + NaCl salt blend was the deicer formulation category by the Pacific Northwest Snowfighters Association, and it had 23 wt. % NaCl brine and 30 wt. % $CaCl_2$ brine in a 100:30 mass ratio. This blend was further diluted to 3:20 by volume (3 parts blend and 20 parts DDI water) to obtain a final salt concentration of about 3.5 wt. %, before using for the EIS test. The other deicer type used for the EIS test was the 29 wt. % $MgCl_2$ brine inhibited with beet juice and diluted with DDI water to a certain ratio.

Only one deicer formulation was used for the salt spray test and is mentioned in the standard test method, SAE J2334. It was a blend of low-concentrated $CaCl_2$, $NaHCO_3$, and NaCl solutions (SAE International, 2016), as indicated in Table 2.2. The chemical names and product types for each selected coating are given in Table C 3 along with their images (Figure C 4) in Appendix C.

A Gantt Chart was developed to define a timeline and monitor the percentage completion of the tests conducted. Moreover, Excel sheets were developed to monitor the progress of each test daily, to avoid missing any data recording.

2.4 Execution of experimental plan – An overview

This section provides details of all the test methods, aided with necessary figures and tables. How each test method was conducted, the number of replicates, the equipment used and its figures, and so forth, are all briefly described here.

In Table 2.3 the materials and the selected coatings for them represent a total of 9 types of coated coupons, referred to as combinations – 3 for steel, 2 for S.S., 2 for aluminum, and 2 for copper. Their replicates are noted as A, B, C, and D. In some cases, only 3 replicates were used. The abbreviations used for each combination are given in Table 2.3.

Table 2.3 Abbreviations used for material-coating combinations for all tests

Materials	Material – Coating combinations	Material – Coating abbreviations
Steel	Steel – Lubra Seal	S-LS
	Steel – Fluid Film	S-FF
	Steel - Aquapon	S-AP
Stainless steel	Stainless steel – Armour Seal	SS-LS
	Stainless steel – Lubra Seal	SS-AS
Aluminum	Aluminum – Armour Seal	Al-AS
	Allunimum – Fluid Film	Al-FF
Copper	Copper – Copper-dielectric grease	Cu-DG
	Copper – Deox IT	Cu-DO

So, for a steel coupon coated with Lubra Seal, three replicates (triplicates) are noted as S-LS-A, S-LS-B, and S-LS-C, where S is for steel, LS for Lubra Seal and A, B, and C are the triplicates used for added accuracy. Another example is Cu-DG-A, where Cu is copper, DG is di-electric grease, and A is a replicate out of the three or five.

2.4.1 Electrochemical impedance spectroscopy – ASTM G59 & G106 – 89

Electrochemical impedance spectroscopy (EIS) is a well-known testing technique used to measure various coating properties including coating resistance, and pore resistance. Moreover, it can also determine the resistance of the solution which is used to submerge coated coupons. It can quite accurately predict the status of a coating over a known period, whether the coating is still intact, or it has failed.

EIS test procedure

To conduct this test a special electrochemical cell is used as shown in Figure 2.3 This cell usually consists of a reference electrode (RE), a working electrode (WE), and a counter electrode (CE); all connected while submerged in a solution carrying various ions promoting a corrosion-laden environment. An

Ag/AgCl (silver/silver chloride) RE filled with potassium chloride (KCl) and a graphite CE was used for this EIS test (Loveday et al., 2004a). Reference electrodes were tested and adjusted by using a new RE and a multimeter (*Reference Electrodes*, n.d.). The working electrode is the coated coupon, either electrically wired to make a connection or directly connected. A salt solution is usually used to immerse all the electrodes connected to a potentiostat before any EIS measurement is taken.

An AMETEK® potentiostat was used for this test as shown in Figure 2.4. The Potentiostat was calibrated by using both internal and external dummy cell calibration procedures (*How-to Guides | Princeton Applied Research*, n.d.). Reference and counter electrodes are only immersed in the solution when an EIS measurement is to be taken. However, coated coupons remain immersed in the solution all the time till the end of the test period (30 days), unless they need to be dried for any dry cycle. For that, they are kept out of the solution for a specified time.

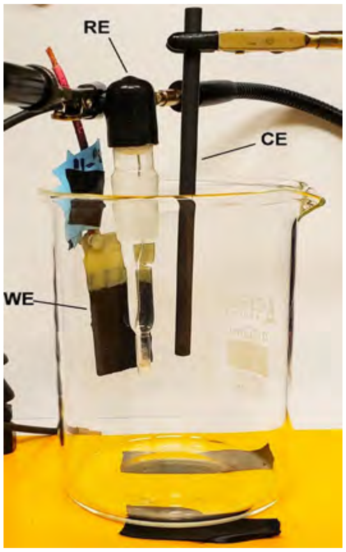


Figure 2.3 The EIS cell used in the testing

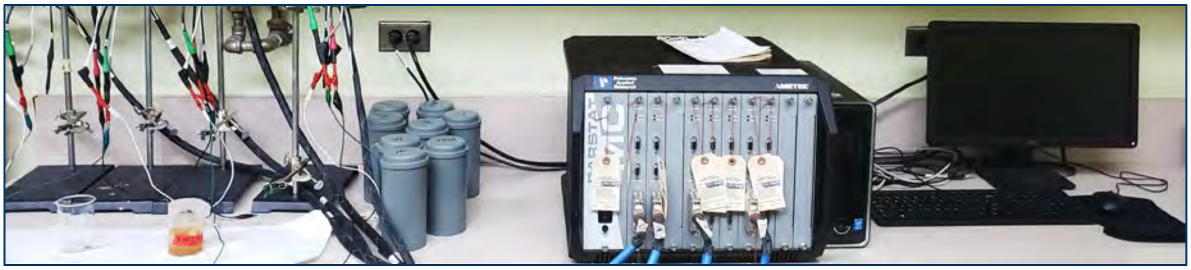


Figure 2.4 Potentiostat (AMETEK®) used to conduct EIS test

For this test, two sets of measurements were taken to judge the performance of coatings in two salt solutions – a blend of $CaCl_2$ and NaCl brine, and $MgCl_2$ brine with added beet juice as a corrosion inhibitor. Both solutions had salt concentrations of about 3 wt. %.

Coupons were first wired electronically, using a pure copper wire, and then coated by following the manufacturers' guidelines in the TDS. A mixture of beeswax and gum rosin was used to cover the coupon area that was supposed to remain unexposed to the salt solution. After that, the wired-coated coupons were ready to be used as working electrodes. Figure 2.5 shows a few of the wired-coated coupons.

The total number of combinations (9) for all coated coupons can be deduced from Table 2.3 – 3 for steel, 2 for S.S., 2 for aluminum, and 2 for copper. For each combination, three replicates (triplicates) were used for the EIS test; therefore, to run two sets of measurements in two salt solutions, a total of 54 (27 x 2) coated coupons were prepared.

Wet and dry cycles were used in this EIS testing to enhance the stress on the coatings. For this, coupons were left immersed in the salt solution for 2 days and were kept out of them for 1 day. This was repeated till one month of testing was completed, for each set of measurements.

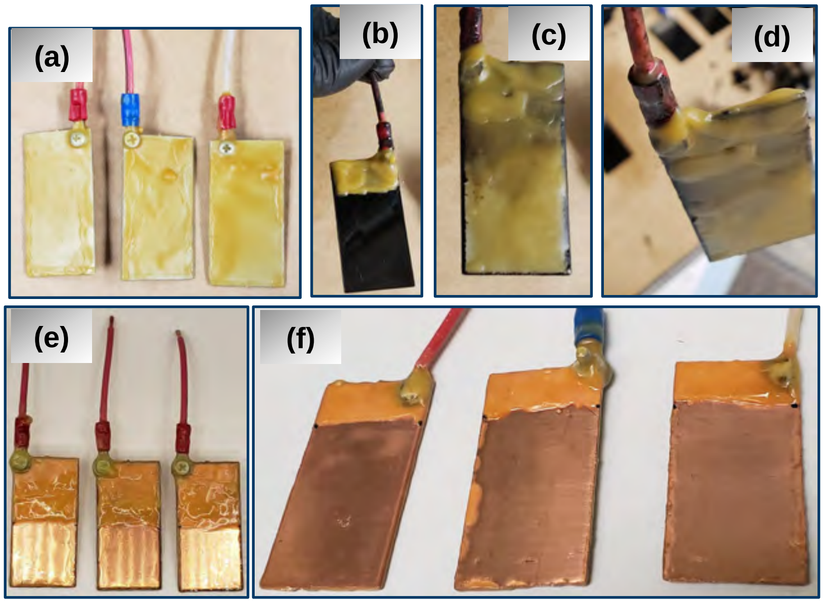


Figure 2.5 EIS test coupons wired and sealed with beeswax: (a) Al-FF backsides, (b) SS-AS-A exposed and sealed area front, (c) SS-AS-A fully sealed backside, (d) SS-AS-A fully sealed connection, (e) Cu-DO-NaCl blend, (f) Cu-DO-$MgCl_2$ blend

The amount of solution to be used was calculated by using the area of the coupon exposed to the salt solution (ASTM International, 2021). The minimum coupon area exposed to the salt solution was found from the standard test method (ASTM International, 2021). On average, the area exposed for coupons was usually 9 cm^2 and 200 mL to 400 mL of salt solution (depending on the exposed area) was used in beakers to immerse the coupons.

For both sets of measurements, approximately 400 EIS readings were taken for all 54 coated coupons, including triplicates. This includes tests performed on day 0 or day 1, and then every 5 days until one month of testing was completed, which gives a total of 7 to 8 days of testing for each set of measurements. Each EIS test takes approximately 90 minutes, including setting the electrochemical cell. A user does not have to be present at all times while the test is running but should monitor intermittently for any abnormal cell behavior. An Excel file used to organize these test series helped avoid excessive workload on one day and made sure no test measurement was missed on the given day and time.

Nyquist and Bode plots

The potentiostat is usually equipped with a software program that processes the EIS test data and develops the plots. The AMETEK® potentiostat used in this test was equipped with the software program called VersaStudio^{TM}. The EIS test provides an assessment of the coating in the form of plots or graphs generated by VersaStudio^{TM}, based on the impedance (Z, ohm-cm^2) and frequency (*f*, hz) values. Two common types of plots are Nyquist (real impedance vs imaginary impedance, Z_{re} vs Z_{im}) and Bode. When the impedance is measured at several frequencies and is plotted on the x-y axis, the resulting plot is known as a Nyquist plot (Loveday et al., 2004a). The Bode plot is a merger of two plots, one is plotted between the logarithmic *f* (Hz) and magnitude of impedance (|Z|) in ohm-cm^2, and the other is plotted between the logarithmic *f* (Hz) and the phase shift (ϕ) commonly known as phase angle in degrees (^{o}). The Bode plots are individually plotted by VersaStudio^{TM} but are often merged into one by the software program employed to model the raw EIS data.

A quick analysis of a coating can be made by just visualizing the plot shapes. For example, an intact coating would have a Nyquist plot with a relatively straight vertical line, instead of a semi-circle or two semi-circles both of which represent a failed coating. From the Bode plots, the values of ϕ^o and |Z| (ohm-cm^2) can also reflect the condition of a coating. The higher the values of ϕ and |Z|, the better the condition of a coating. An example of Nyquist and Bode plots (SS-AS-NaCl blend-30 min) of an intact coating is shown in Figures 2.6 and 2.7 respectively. The Nyquist plots indicate that the coating after only 30 minutes of immersion in the blend salt brine shows no signs of failure because it is an almost straight vertical line. In Nyquist plots, a diagonal line with a slope of 45^o represents Warburg impedance (Gamry Instruments, 2010).

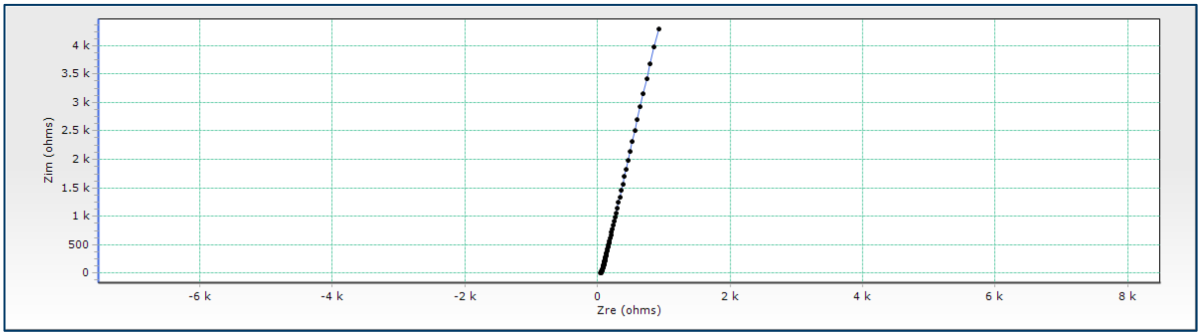


Figure 2.6 Nyquist plot (Z_{real} vs $Z_{imaginary}$) for SS-AS-C after only 30 minutes of immersion in $CaCl_2$+NaCl blend salt brine

However, the very low impedance magnitude in the Bode magnitude plot of the same coupons (Fig 2.7a) indicates that the pore resistance of this coating may not be good enough. Sometimes it can improve over time, which it did after 5 days, but it was still very low. The Bode phase plot (Fig 2.7b) indicates an increase in the phase angle from 0^o to nearly 80^o, which means that the coating is doing very well.

Some electrochemical process parameters can also be estimated from these plots. These include solution and polarization resistances. However, for this study focus was kept on the coating resistance (the pore resistance) of each coating on each coupon, which can be more accurately obtained after modeling of raw EIS data.

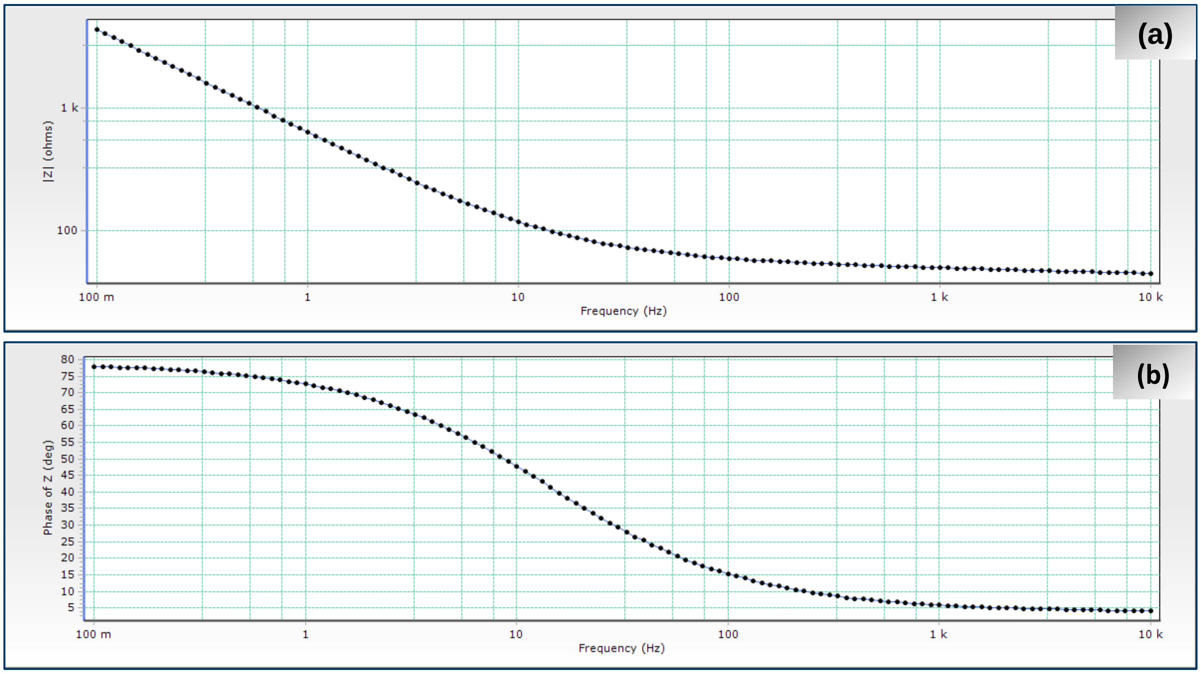


Figure 2.7 Bode plots for SS-AS-C after only 30 min of immersion in $CaCl_2$+NaCl blend salt brine (a) Bode magnitude (b) Bode phase

How the raw EIS data obtained from VersaStudio^{TM} was analyzed is briefly explained in section 2.5 – Data analysis.

2.4.2 Salt spray test – SAE J2334

This test was carried out to evaluate the 5 years of projected performance of each coating for its corrosion resistance against a typical marine-laden environment. The test can be performed in two broad methods: (1) without scribed coated coupons, and (2) with the scribed coated coupons.

The first method is the conventional salt spray testing method (ASTM International, 2019b; SAE International, 2016) which follows the exposure of coated coupons to the salt solution (usually 5 wt. % NaCl brine) for a known number of hours. The other method follows a similar methodology as the first one, only the coupons are scribed after being coated following the ASTM D-1654 standard method (ASTM International, 2016). For this project, based on their nature, some coatings were scribed, and others were not before testing. SAE J2334 was followed for both methods, however, ASTM B-117 (ASTM International, 2019b) is a commonly used salt spray test method. The salt spray chamber used in this test is shown in Fig. 2.8a.

SAE J2334 – Test parameters

Initially, it was decided to use 5 replicates for each material-coating combination, but because of a faulty new SSC, which never worked, the available chamber space was reduced, and the number of replicates had to be decreased to 4. All coated coupons were placed at once in the SSC (Fig 2.10a) for the salt exposure cycle, and in the humidity chamber for the other two cycles. A humidity chamber (Memmert, USA, LLC®) in Figure 2.8b, was used to run the humid and dry cycles each day. The conditions used for all cycles and the ramp times in between each cycle are added to Table 2.4.

Table 2.4 Conditions and ramp times used for each cycle of SAE J2334

Conditions	Cycles	Values
	Humid Cycle – 6 hours	
Temperature (^{o} C)		48 – 50^{o} C
Relative humidity (R.H., %)		85 – 95 %
Time of exposure (T, hr)		6 hrs.
Ramp time – Humid cycle and salt exposure cycle		20 min
	Salt Exposure Cycle – 15 minutes	
Temperature (^{o} C)		35^{o} C
Relative humidity (R.H., %)		95 – 99 %
Time of exposure (T, hr)		0.25 hr. (15 min)
Ramp time – Salt exposure cycle and dry cycle		20 min
	Dry Cycle – 17 hours	
Temperature (^{o} C)		60^{o} C
Relative humidity (R.H., %)		50 %
Time of exposure (T, hr)		17 hrs.
Ramp time – Dry cycle and humid cycle		15 min

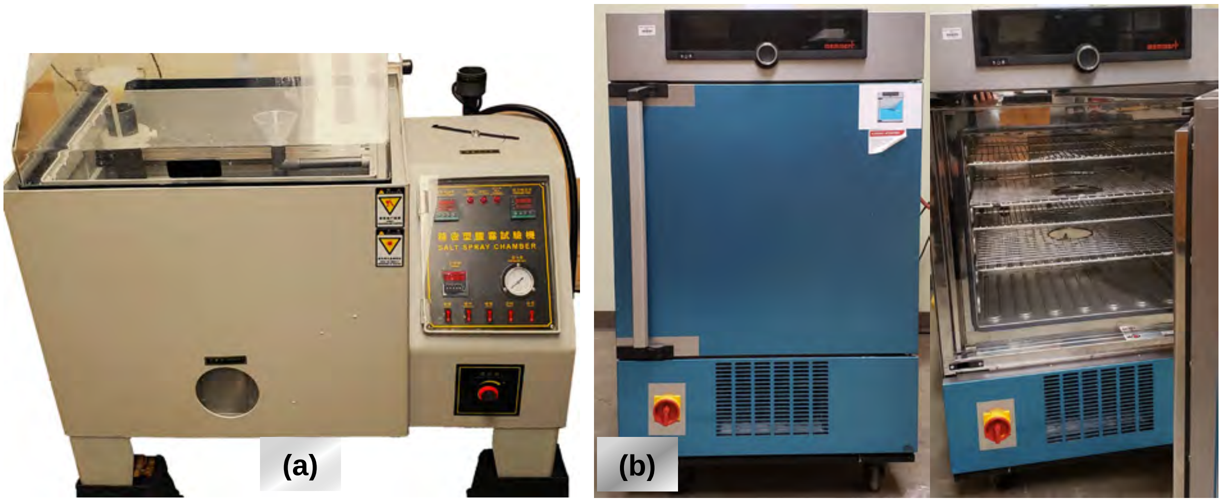


Figure 2.8 Equipment used to conduct SAE J2334 (a) Salt spray chamber, 110L capacity (110V) (b) Humidity chamber (Memmert, USA, LLC®) for humid and dry cycles

SAE J2334 – Test procedure

SAE J2334 salt spray test is a cyclic test procedure, which requires a pre-defined number of cycles to be completed for evaluating a coating's performance against corrosion. Each cycle lasts about a day and involves three different stages – The humid cycle, the salt spray cycle, and the dry cycle. The cycles are shown in Figure 2.9. The standard test method (SAEJ2334) is an accelerated test that indicates a minimum exposure for the coupons scribed or un-scribed should be 60 cycles or days (SAE International, 2016), which would be equivalent to 5 years of real-life exposure.

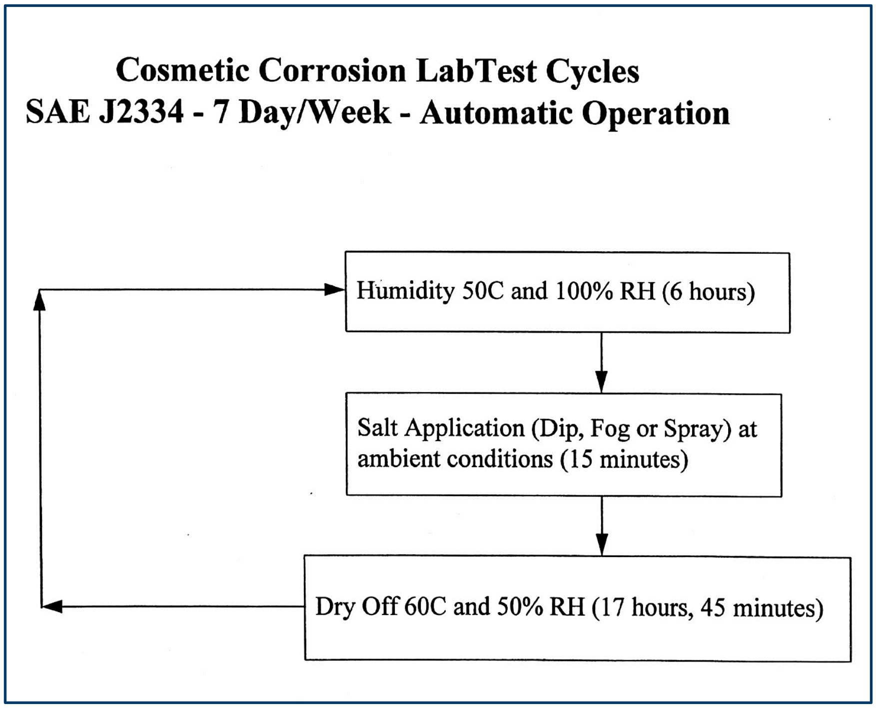


Figure 2.9 SAE J2334 daily test cycles for automatic operations (including weekends) (*J2334_201604*, n.d.)

For this test, 60 test cycles were completed that lasted for 2 months. Weekends and federal holidays were also utilized. Prepared coupons were placed daily in the humidity chamber for the 6-hour humid cycle, taken out of it, moved to the SSC for the 15-minute salt exposure cycle, and then moved back to the humidity chamber to run the dry cycle. This was repeated every day for two months.

The salt spray chamber (SSC) used for this test had a fog deploying method, instead of spraying. It had a brine chamber filled with approximately 5 liters of freshly prepared SAE salt solution, and a heated water chamber filled with tap water to create a humid environment. The salt solution must be made with distilled water only and was changed once a week (SAE International, 2016). The conductivity of the freshly prepared SAE salt solution ranged between 9.8 mS – 10.30 mS, which narrowly satisfied the conditions in the standard method (SAE International, 2016). The fog collection rate of the SSC used in this test was 4 mL/hr.

Coupons coated with Armour Seal, Lubra Seal, and Aquapon (S-LS, S-AP, SS-AS, SS-LS, Al-AS, and their replicates A to D) were scribed using the method defined in the ASTM D1654 (ASTM International, 2016). Other coupons coated with Fluid Film, dielectric grease, and Deox IT, were not scribed and therefore were smaller in size. The guidance on the sizes of scribed and non-scribed coupons was taken from standard test methods (ASTM International, 2016, 2019b; SAE International, 2016). Figure 2.10 shows coupons inside the SSC (just after the salt exposure cycle) and humidity chamber.

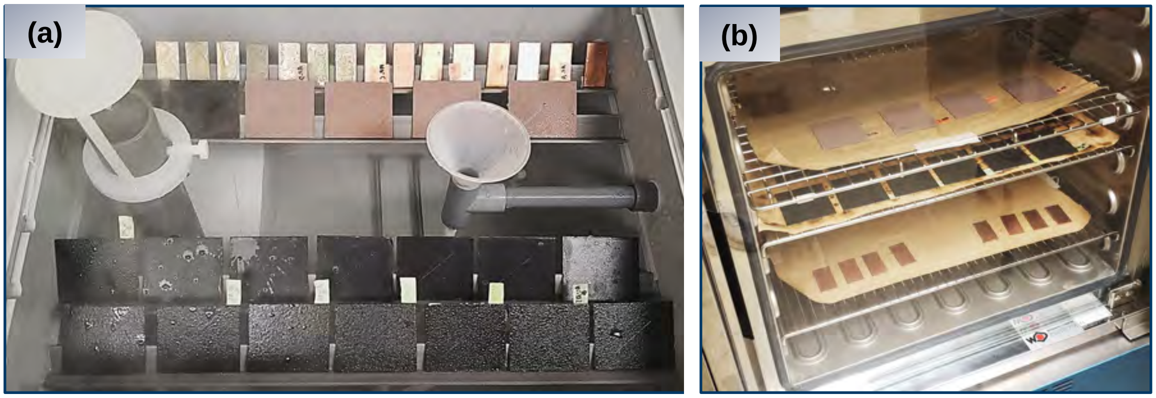


Figure 2.10 All coupons (scribed and non-scribed) placed in (a) SSC and (b) humidity chamber

Daily monitoring of coupons, weighing, and intermittent cleaning

After first preparing the coupons, they were weighed and photographed before beginning the test. To monitor the progress of the test every day, an Excel spreadsheet was prepared and can be shared as electronic media, on demand.

The standard describes that the weights of the coupons should be taken after a fixed number of days (SAE International, 2016). In this case, the weights of all coupons were taken after every 10 to 12 days. However, once the rusting started on steel coupons coated with Lubra Seal, their cleaning was done more frequently, and their weights were taken before and after each cleaning. The intermittent cleaning of coupons with rust on them (S-LS) was done by gently scrubbing the rusted areas with sandpaper of grit size 2000 and then blasting compressed air on them. Before taking the final weights on the last day of testing (December 19, 2023), rusted and corroded coupons were thoroughly cleaned, by scrubbing 1,200 grit sandpaper (for Cu-DO), 1,500 grit sandpaper (for Cu-DG), and 600 grit followed by 1,200 grit sandpaper (for S-LS), washing, and cleaning with acetone using cotton. All coupons were dried after thorough cleaning at 60C for 12 hours, before taking their final weights for corrosion rates (C.R.) calculations. Rugged sandblasting or chemical cleaning methods, mentioned in ASTM G1-03 (ASTM International, 2017b) were not employed as those could also have removed the intact paint from the coupons, which could have been misleading in C.R. calculations. All 60 cycles of the SAE J2334 test were completed by daily exposing the coated coupons to humid, salt exposure, and dry cycles, regularly taking photographs, and doing intermittent cleaning.

2.4.3 Adhesion test – ASTM D4541

The adhesion test was done for hard coatings including Lubra Seal, Armour Seal, and Aquapon. Other coatings used (Fluid Film, dielectric grease, and Deox IT) were soft and did not require adhesion testing. To conduct this test a standard test method ASTM D4541 (ASTM International, 2017a) was followed.

Pull-off adhesion test procedure

To perform this test a manual pull-off adhesion tester kit (PosiTest® AT-M by DeFelsko®) was purchased, which comes with adhesive glue, dollies, and a manual pull-off pump equipped with an actuator assembly and a digital display, as shown in Figure 2.11.

After the coupons were coated, they were mildly abraded with sandpaper provided with the adhesion tester kit, to improve the bonding between the adhesive and the dolly. A 20 mm dolly (also rubbed on the sandpaper) was used to adhere to the coated coupon with the adhesive glue, by following the guidelines in the standard test method. The dollies were left to bond fully onto the coated coupons by letting the adhesive set, for 48 hours. The manual provided by the PosiTest® AT-M was carefully followed to operate the tester and complete the test. Five replicates were used for each material-coating combination.

The results for the adhesion strength of the coatings were obtained in pounds per square inch (psi). The adhesion test for Lubra Seal coupons was not much successful due to glue failure occurring on those coupons. Details on the data analysis are provided in section 2.5. Some of the coupons used in the adhesion test are shown in Figure 2.12.

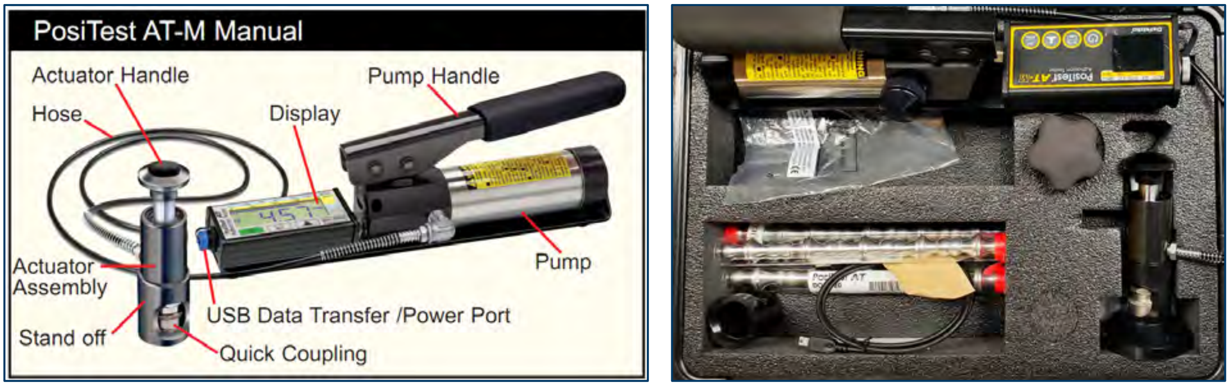


Figure 2.11 PosiTest® AT-M pull-off adhesion tester to conduct ASTM D4541 (*Instruction Manuals | DeFelsko*, n.d.)

Figure 2.12 Coupons used in pull-off adhesion test: (a) S-LS (black coupons) and S-AP coupons (dark pink), (b) SS-AS, and (c) Al-AS

2.4.4 Pencil hardness test – ASTM D3363

To test the hardness of the hard coatings (sealants and zinc-rich epoxy primer), the ASTM D3363 standard test method (ASTM International, 2022a) was followed. The hardness test for the soft coatings (lubricants and grease) was not done.

ASTM D3363 – Test procedure

A pencil hardness test kit was purchased to conduct this test, which included a stainless steel block of known weight having a slot to hold a lead pencil at an angle of 45^o, sandpaper, and a set of lead pencils. The standard procedure of the pencil hardness test is easy to follow and some help can be found on a YouTube video by Elcometer® inspection equipment (Elcometer Inspection Equipment - Coatings Industry, 2019). According to the standard method, a set of lead pencils were sharpened using a special sharpening method which protected the lead from being sharpened and only removed the wood. To sharpen the pencils that way a designated sharpener was also purchased separately. All pencils were then rubbed on a sandpaper provided with the kit, by holding each pencil at an angle of 90^o, to flatten the end of the exposed lead. The exposed lead's length was kept between 5 to 6 mm.

The hardness scale followed is illustrated in Table 2.5 in which 6B is the softest lead and 6H is the hardest.

Table 2.5 Hardness scale for the lead pencils used in ASTM D3363

Lead Pencils – Hardness Numbers													
6B	5B	4B	3B	2B	B	HB	F	H	2H	3H	4H	5H	6H
			Softer							*Harder*			

Before commencing the test, the weighted block was fully rested on the coated coupons lying flat on a horizontal surface. To begin with the testing, the hardest lead pencil was placed in the weighted block at an angle of 45^o and locked by a nut when its tip touched the coated surface. The block was then pushed forward, away from the operator, up to a distance of at least 6.5 mm on the coated surface. This procedure was repeated on a new area of the same coupon, using pencils one after another with decreasing lead hardness following hard to hard-to-soft scale. Figure 2.13 shows some of the coupons tested with this method and the pencil hardness kit utilized.

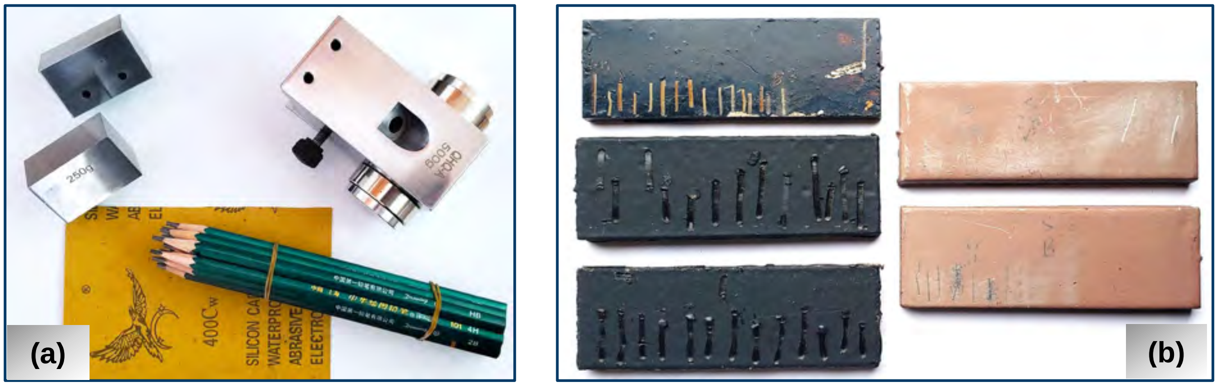


Figure 2.13 Pencil hardness test: (a) test kit, and (b) some coupons (S-LS, SS-AS, Al-AS, and S-AP) tested

The standard method describes that the test may be stopped when a pencil is found that will neither cut through for a distance of at least 3 mm nor scratch the film (ASTM International, 2022a). The last pencil to cut through the coated film for at least 3 mm of distance is the Gouge hardness. Furthermore, the first pencil that does not scratch the coated film, while continuing the test from hard to soft scale, is the scratch hardness. The types of scratches or cuts made to the coated surface are described in section 2.5.

The test was conducted for coupons S-LS, S-AP, SS-LS, SS-AS, and Al-AS; with 5 replicates each. The data analysis is further described in section 2.5.

2.4.5 Vickers hardness test – ASTM E384

After the pencil hardness test did not provide any concrete results for S-AP coupons, due to the very hard coated surface, it was decided to perform the Vickers hardness test for them. Though there is a standard test method (ASTM E384) (ASTM International, 2022b) available that describes the Vickers and Knoop hardness methods for hard-coated surfaces, it was not entirely followed or consulted. Micro Vickers (microindentation) hardness test was conducted for the S-AP coupons.

Micro Vickers hardness test procedure

To perform the Micro Vickers hardness test, the coupons prepared for the pencil hardness test were used. A square pyramidal-shaped diamond indenter was equipped with the Vickers hardness tester. The load applied on each coated coupon was 1Kg$_f$, whereas the dwell time was 15 seconds. The indentations were observed using a light microscope equipped with the tester and diagonals were measured precisely. Figure 2.14 shows the indentations made on the coated coupon of S-AP.

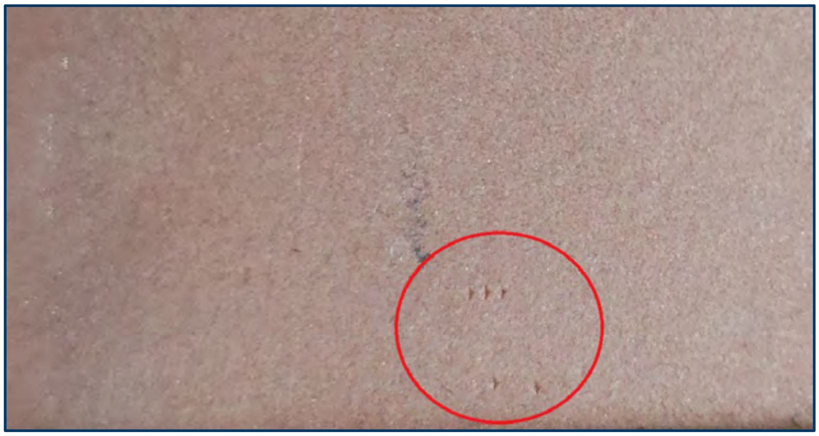


Figure 2.14 Micro indentations made on the S-AP coupon, after the Vickers hardness testing

2.5 Data analysis – An overview

This section will elaborate on how data gathered from each test was analyzed. For instance, for the photographs taken during the SAE J2334 test, ASTM methods were followed to determine the percentage of blistering or the amount of rust. Also, the modeling of raw EIS graphs was conducted to analyze raw EIS data, and so forth.

2.5.1 EIS raw data analysis

To analyze the raw EIS data obtained from VersaStudio^{TM}, a software program ZsimpWin was used. To save time, not all the raw plots (over 400) were modeled, rather the data only from day 0 or 1 and day 30 was analyzed, which reduced the number of modeled graphs to about 120.

Modeling of the EIS plots & electrochemical circuits

For in-depth analysis, the raw EIS plots were modeled in a software program. Once the raw EIS data is modeled, important coating parameters that define the integrity of a coating are available. Those parameters include the coating capacitance (C_c), double layer capacitance (C_{dl}), charge transfer resistance (R_{ct}), and pore resistance (R_{po}), which is also known as coating resistance (Loveday et al., 2004b). The value of C_c is much lower (usually 1nF/cm^2) than C_{dl}, which is usually between 10-40 µF/cm^2 (Loveday et al., 2004b). This is because C_{dl} is the capacitance of the metal-electrolyte interface called the 'double layer' and is sometimes related to the delamination of the coating. The higher the capacitance (either C_c or C_{dl}), the lower the integrity of the coating. Typically for an immersed coated sample, when the coating absorbs water (water uptake) over time, its capacitance (C_c) increases due to the dielectric constant of water which is often higher than that of a coating. The Brasher-Kingsbury equation can determine the volume fraction of the water absorbed by a coating over time:

$$Volume\ Fraction\ (H_2O) = \frac{log\left(\frac{C_t}{C_o}\right)}{log\ \varepsilon_w} \qquad (1)$$

where C_t is the coating capacitance at time t, C_o is the initial coating capacitance, and ε_w is the dielectric constant for water, which is 80.

To model the raw EIS plots from Versa Studio^{TM}, an appropriate circuit was selected in ZsimpWin depending on the raw plot curves or shapes. The various elements used in such circuits were mostly resistances (R), capacitances (C), diffusion coefficients, inductors (L), constant phase element (Q), and the Warburg impedance (W). The aim was not to complicate the circuit choices and to limit the number of resistances used to three – a solution resistance (R_s), a pore resistance (R_{po}), and a charge transfer resistance (R_{ct}) or polarization resistance (R_p). A few common circuits often used for modeling are shown in Figure 2.15, where C_{dl} is the double layer capacitance and C_c is the intact coating's capacitance.

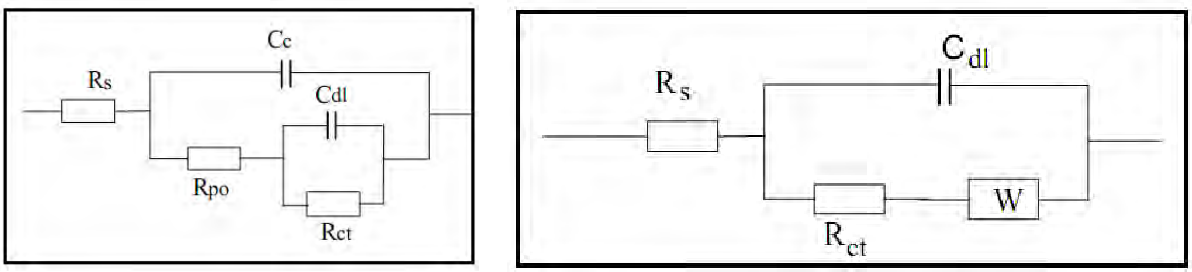
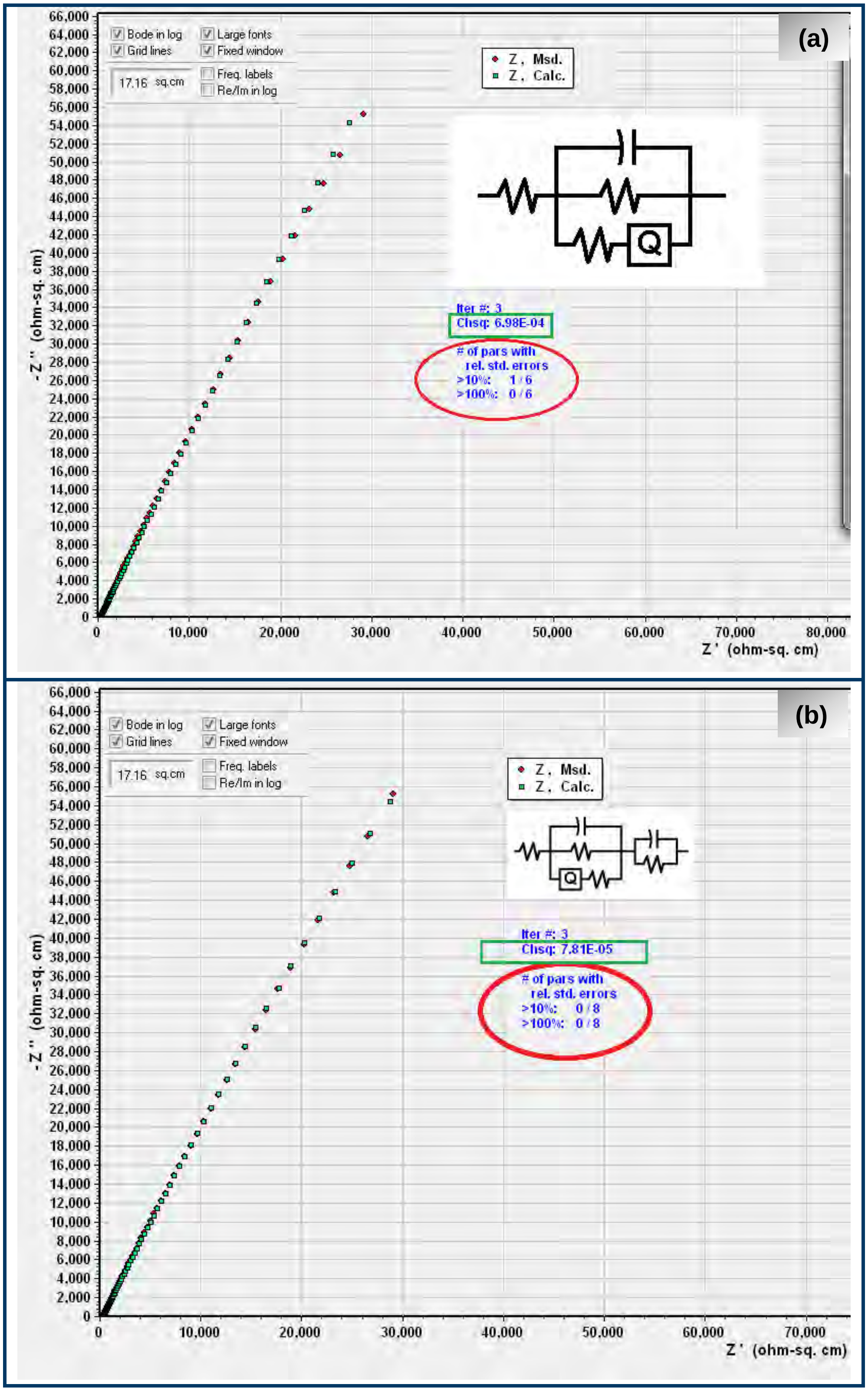


Figure 2.15 Commonly used equivalent electrical circuits for modeling raw EIS plots (Gamry Instruments, 2010)

After modeling the EIS data using a proper equivalent circuit, keeping the percent relative standard errors to a minimum, and obtaining a "perfect" fit, the data on coating parameters became available. The fit of the modeled plot indicates how accurately the modeling was done. The lower the value of chi-squared (x^2), the better the fit for the modeled plots, as shown in Figure 2.16. A good value for x^2 would be below 1×10^{-3} or 0.001.

Figure 2.16 shows the modeled Nyquist plots for SS-AS after 2 days of EIS testing. The raw plot curve (in red dots) indicates that the coating is very much intact as it is a relatively straight line, more vertical than horizontal, and is not parabolic or circular. The figure shows the same raw plot modeled twice using two equivalent circuits. Chi-squared, illustrated as 'Chsq' marked in the green box and the percent relative standard error are circled red. In Fig. 2.16a when the chi-squared is higher (6.98×10^{-4}) the fit is not perfect, and the number of errors (though still very low) is also higher than the modeled plot in Figure 2.16b; which has a lower chsq (7.8×10^{-5}), therefore a better fit and no relative standard errors above 10% or 100%.



(a)

☑ Bode in log ☑ Large fonts
☑ Grid lines ☑ Fixed window
17.16 sq.cm ☐ Freq. labels
☐ Re/Im in log

Iter #: 3
Chsq: 6.98E-04
# of pars with
rel. std. errors
>10%: 1 / 6
>100%: 0 / 6

(b)

☑ Bode in log ☑ Large fonts
☑ Grid lines ☑ Fixed window
17.16 sq.cm ☐ Freq. labels
☐ Re/Im in log

Iter #: 3
Chsq: 7.81E-05
# of pars with
rel. std. errors
>10%: 0 / 8
>100%: 0 / 8

Figure 2.16 Modeled EIS Nyquist plots for SS-AS-2 days-NaCl |CaCl₂ blend salt brined: (a) Higher x^2 (chsq) and errors (b) lower x^2 and reduced errors

For this study, not only x^2 values were kept as low as possible by finding the proper circuit giving a good fit, but the percentage relative standard errors were also kept lower than 10% in most cases. Ideally, this error should not be more than 1 to 2 %, which is hard to obtain provided that the salt-laden environment for coatings has a complex chemistry of ions. Moreover, other factors like the stability of the electrochemical cell, physical interruptions, nature and type of coatings, and the type and quality of electrodes also play a key role in minimizing the % error for each result.

2.5.2 Salt spray test – Data analysis

Data gathered from the SAE J2334 test was based on the weights of the coupons before and after intermittent cleaning and the photographs showing the areas rusted and physical conditions for some coupons. This data was used to calculate the corrosion rates and analyze the degree of rusting (if present) on any of the coupons.

Method of determining corrosion rates

According to the standard method (SAE International, 2016), the weight loss due to rusting or corrosion should be calculated by following the guidelines provided in ASTM G1-03 (ASTM International, 2017b), which also defines the corrosion rate in terms of weight loss as below:

$$Corrosion\ rate\ (C.R.) = \frac{(K x\ W)}{(A\ x\ T\ x\ D)} \tag{2}$$

where D is the density of the material in g/cm3, T is the time of exposure in hours, A is the area of the coupon exposed in cm^2, and W is the weight loss due to corrosion. The value of K (a constant) depends on the desired units of the corrosion rate. If the corrosion rate is intended to be in mils per year (mpy) then the value of the K should be 3.45×10^6. Mils is one-tenth of an inch.

Moreover, rust creepage (for scribed coupons) was also analyzed only for S-LS coupons because they were corroding. To find creepage, ASTM D1654 (ASTM International, 2016) was followed. The width of the rusted area around the scribe was measured at 5 different points along the scribe and their mean was calculated. Then using equation (3), rust creepage was calculated.

$$c = \frac{w_c - w}{2} \tag{3}$$

where c is rust creepage, w_c is the mean width of the rusted area around the scribe, and w is the initial width of the scribe (0.762mm). Based on creepage values, creepage ratings were determined based on rating guidance (Figure C 4, Appendix C) provided in ASTM D1654 (ASTM International, 2016). Since only S-LS coupons showed rusting for most of the test duration, they were cleaned more frequently. However, Cu-DG and Cu-DO coupons also showed signs of corrosion and were cleaned only at the very end of the test. Weights of coupons before and after intermittent cleaning were used in an Excel spreadsheet to calculate the weight loss and C.R. in mpy using equation (2).

Method of evaluating the degree of rusting on painted steel surfaces

To determine the percentage of rust on a painted steel coupon, ASTM D610 – 08 (ASTM International, 2019a) was followed. The method defines the various rust distribution types (spot, general, pinpoint, and hybrid) and the rust grades by using visual examples. A few visual examples are provided in Appendix C (Figure C5), however, the rust grades for various percentages of surface rusted are provided in Table 2.6. To quantify the % area covered by rust on steel coupons painted with Lubra Seal, an image processing software program called Image J was used.

As shown in Figure C 5 (Appendix C), spot rusting occurs when most of the rusting is concentrated in a few localized areas of the painted surface, general rusting occurs when different sizes of rust spots are randomly distributed across the painted surface; and pinpoint rusting as the name suggests, occurs when the rust is distributed across the surface as tiny individual specks of rust. Pinpoint rusting was the case for scribed S-LS coupons, during the first few weeks. Hybrid rusting is the combination of two or more types of rusting described above.

Table 2.6 Rust ratings based on the rust grades and rust distribution types (ASTM International, 2019a)

Rust Grade	Percent of Surface Rusted	Visual Examples		
		Spot (S)	General (G)	Pinpoint (P)
10	Less than or equal to 0.01 percent		None	
9	Greater than 0.01 percent and up to 0.03 percent	9-S	9-G	9-P
8	Greater than 0.03 percent and up to 0.1 percent	8-S	8-G	8-P
7	Greater than 0.1 percent and up to 0.3 percent	7-S	7-G	7-P
6	Greater than 0.3 percent and up to 1.0 percent	6-S	6-G	6-P
5	Greater than 1.0 percent and up to 3.0 percent	5-S	5-G	5-P
4	Greater than 3.0 percent and up to 10.0 percent	4-S	4-G	4-P
3	Greater than 10.0 percent and up to 16.0 percent	3-S	3-G	3-P
2	Greater than 16.0 percent and up to 33.0 percent	2-S	2-G	2-P
1	Greater than 33.0 percent and up to 50.0 percent	1-S	1-G	1-P
0	Greater than 50 percent		None	

In this study, for the scribed S-LS coupons, the rust grade was mostly between 0 and 4. Further comments on the rust ratings and corrosion rates for S-LS, Cu-DG, and Cu-DO coupons are made in Chapter 3 – Results.

2.5.3 Adhesion test – Data analysis

Data analysis for the adhesion test was straightforward. The values recorded in the PosiTest AT-M were noted down to be reported in an Excel sheet. The different types of fractures and the glue failure are shown in Figure… and are defined below.

Types of fractures and glue failure – Pull off adhesion test

A cohesive fracture (Figure 2.17a) occurs within a coated layer, rupturing the coated layer. This type of fracture would leave a smooth layer of coating on the dolly face, regardless of the number of layers coated or painted on the coupon. In this study, all coupons had only one layer of coating on them, either the epoxy primer (Aquapon), the Armour Seal, or the Lubra Seal.

An adhesive fracture occurs between the layers of a coating system (a primer or tie/tack and topcoat), as shown in Figure 2.17b. So, if this type of fracture would have occurred for any of the coupons coated with Aquapon, Lubra and Armour Seals, the surface of the bare coupons must have become visible after the dolly was detached.

A glue failure, as shown in Figure 2.17c, occurs when the glue separates from either itself, the coating, or the dolly; leaving no signs of coating on the dolly face. This could happen if the glue is not properly mixed, or the coating was not applied properly. This may also happen if the surface of the coating is too shiny, or the glue material is not bonding properly with the specific paint or coating.

These types of fractures or glue failures are reported for all the test coupons in Chapter 3.

2.5.4 Pencil hardness and Vickers hardness tests – Data analysis

Pencil hardness test analysis

The pencil hardness test data was based on Table 2.7 and the hardness is reported in terms of either Gouge or Scratch hardness. Figure 2.17d shows the visual illustration of both types of deformation made to the coated film by the lead.

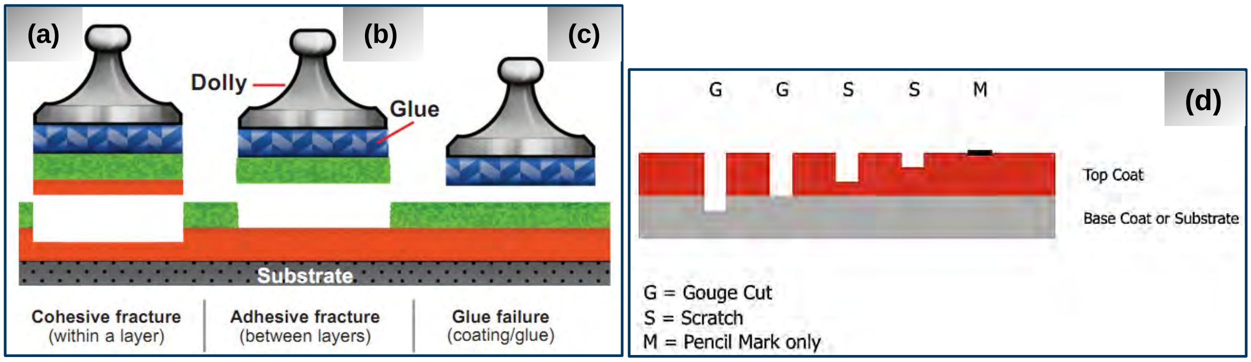


Figure 2.17 Adhesion test analysis: (a) cohesive fracture, (b) adhesive fracture, and (c) glue failure (ASTM International, 2017a) (d) an illustration of Gouge, Scratch, and a Mark on the coated coupon, left behind by the lead (ASTM International, 2022a)

A cut of gouge is a result in which the coating film is removed, revealing the surface of the coupon. However, when the deformation is made to the coated layer by the lead, but it is not deep enough to reveal the surface of the coupon, it is regarded as a scratch. A mark would not cut through or scratch the coated film and can be removed by an eraser; therefore, not considered a permanent deformation or a hardness result. Moreover, to examine the cut, scratch, or mark, a magnifying glass with added light was used. Gently rubbing the finger or thumbnail also helped identify between a scratch or a mark. Results are provided in Chapter 3.

Vickers hardness test analysis

For the Micro Vickers hardness test, done only for S-AP coupons, the microindentations made on the coupons were analyzed right after applying the load on them, using a light microscope equipped with the tester. Figure 2.18 shows the calculations made for finding the diagonals of the pyramidal shape produced by the pyramidal-shaped diamond indenter.

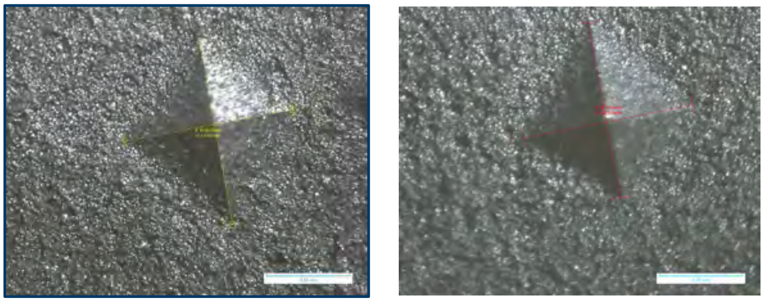


Figure 2.18 Precisely measured diagonals of the pyramidal-shaped microindentation made on the S-AP coupon

Chapter 3: Results

Results of the various tests performed to evaluate the selected corrosion protection coatings are presented and described in this chapter.

3.1 EIS results

3.1.1 EIS modeling results – Plots and data for NaCl-CaCl$_2$ salt blend

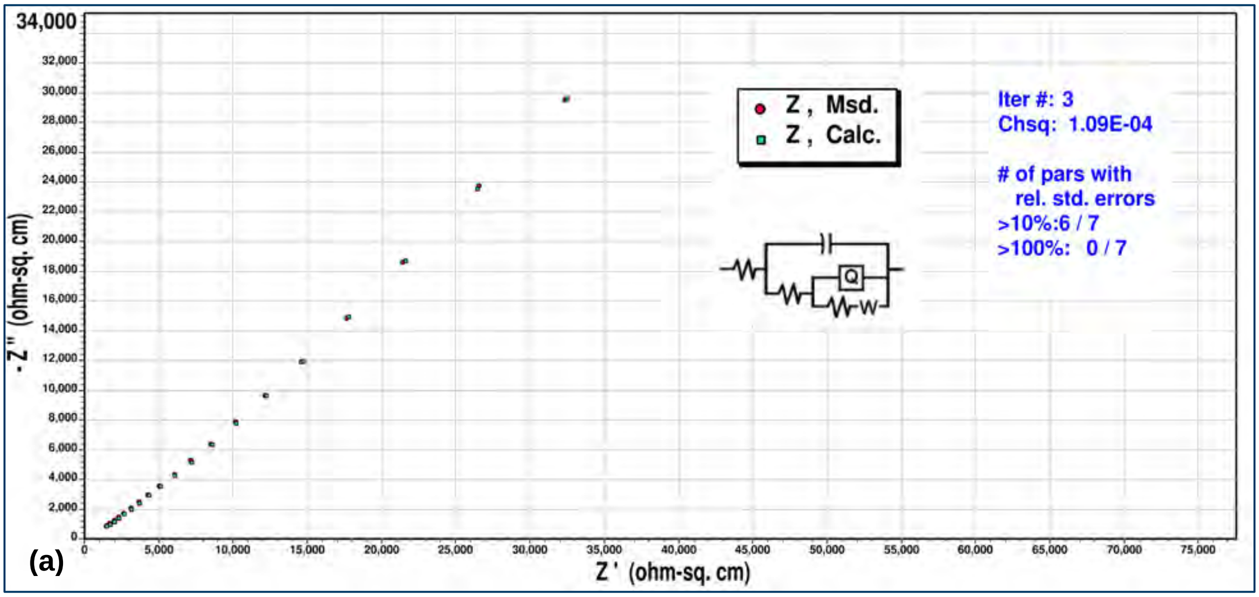
This section presents only some of the results from day 0 or 1 and day 30. Because it is not feasible to add all the plots (~200) for all replicates of coated coupons from the EIS test done for both salt solutions, complete results (plots and data) can be provided electronically to Clear Roads on demand. Moreover, the modeled graphs of one of the replicates (either A, B, or C) for the first and last day of testing are added only for the S-LS coupons, to give an example of what they illustrate. The rest of the modeled graphs are added to Appendix A. Also, a screenshot showing the results obtained after modeling in ZsimpWin along with the modeled Nyquist plot and the raw graph from VersaStudio is added as Figure C 6, Appendix C. The data gathered after modeling each test for all types of material-coating combinations in both salt solutions are provided in this section. Note that the “end value” instead of the “start value” indicates the EIS parameters of interest.

All the EIS tests were conducted for 30 days following a similar trend of wet-dry cycles for all coupons, as mentioned in Chapter 2.

3.1.1.1 Steel-Lubra Seal – NaCl-CaCl$_2$ blend

Day 1 - Results

The modeled Nyquist and Bode plots for S-LS-C after 1 hour of immersion are shown in Figure 3.1.



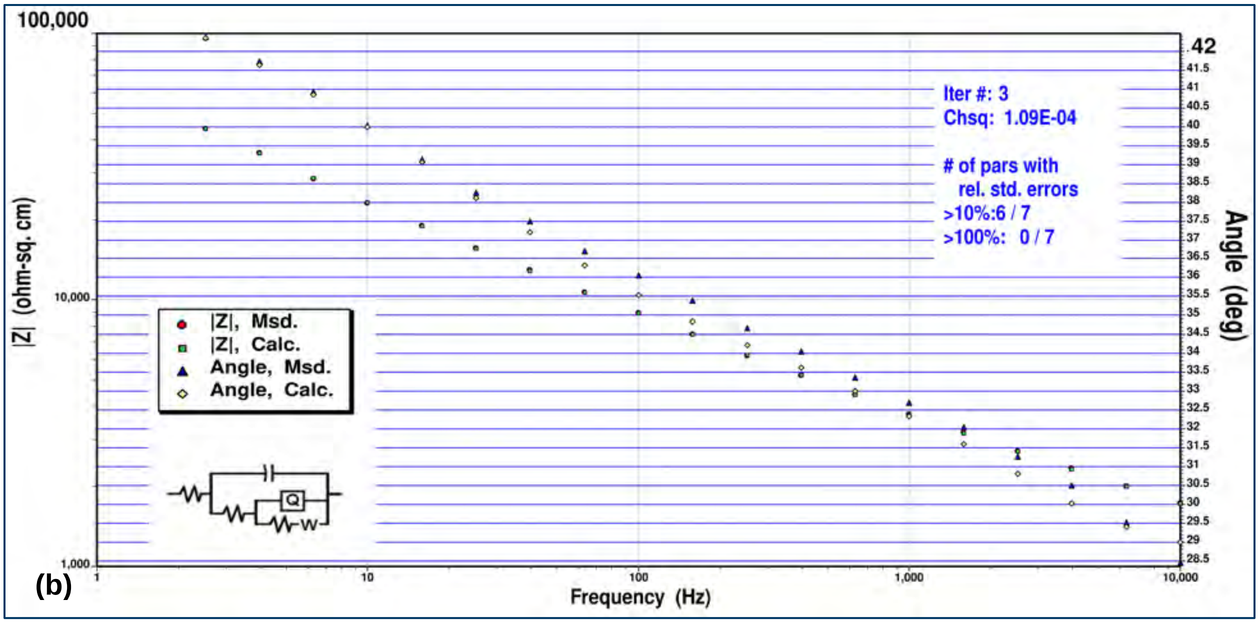


Figure 3.1 EIS modeled plots for S-LS-C-1 hr, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

The straight line with a slope of 45° in the Nyquist plot of S-LS-C-1hr is the Warburg impedance. The impedance on the Nyquist plot is between 30 to 40,000 ohm-cm^2, which can be regarded as good, but not excellent for an hour-old sample inside the salt solution. The Bode plots, however, indicate that the coating is intact, though not showing a very high resistance against the salt solution depicted by only a 100 K-Ohm-cm^2 of an impedance magnitude and a poor phase angle that reaches 42°.

The coating parameters obtained after modeling the raw data of S-LS-A-day 1 are added to Table 3.1, where R $_{sol}$, R $_{po}$, and R $_{c\,t}$ are the solution, pore, and charge transfer resistances. Warburg component (W-Yo) is used in modeling plots that have Warburg impedance. The CPE (Q-Yo) is used when there is semi-infinite diffusion or general diffusion is occurring. It may also be considered as capacitance in special cases.

Table 3.1 Coating-related parameters obtained after modeling raw EIS data for S-LS-A-day 1

Exposed area of coupon S-LS-A | 9.68 cm^2			**Chi-Squared (x^2)** | 1.09 x 10^{-4}	
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
1	R$_{sol}$ (ohm-cm^2)	6.19E+02	6.19E+02	49.6
2	C$_c$ (F/cm^2)	4.23E-09	4.22E-09	85.0
3	R$_{po}$ (ohm-cm^2)	6.60E+01	6.60E+02	21.3
4	Q-Yo (S-sec^n /cm^2)	3.37E-06	3.37E-06	15.6
5	Q-n	5.16E-01	5.16E-01	4.2
6	R$_{c\,t}$ (ohm-cm^2)	1.67E+04	1.68E+04	18.5
7	W-Yo (S-sec$^{0.5}$ /cm^2)	2.63E-06	2.63E-06	16.8

The higher the R_{po}, and $R_{c\,t}$, the greater the integrity of the coating. The solution resistance indicates whether corrosion products (less or more) are developing in the solution. A low R_{sol} is usually due to low corrosion products in the solution or a less complex system of ions; both factors may boost the coating's performance.

Day 30 - Results

The modeled plots of S-LS-C after 30 days of EIS test with wet/dry cycles are shown in Figure 3.2 and the corresponding data obtained including process parameters are provided in Table 3.2.

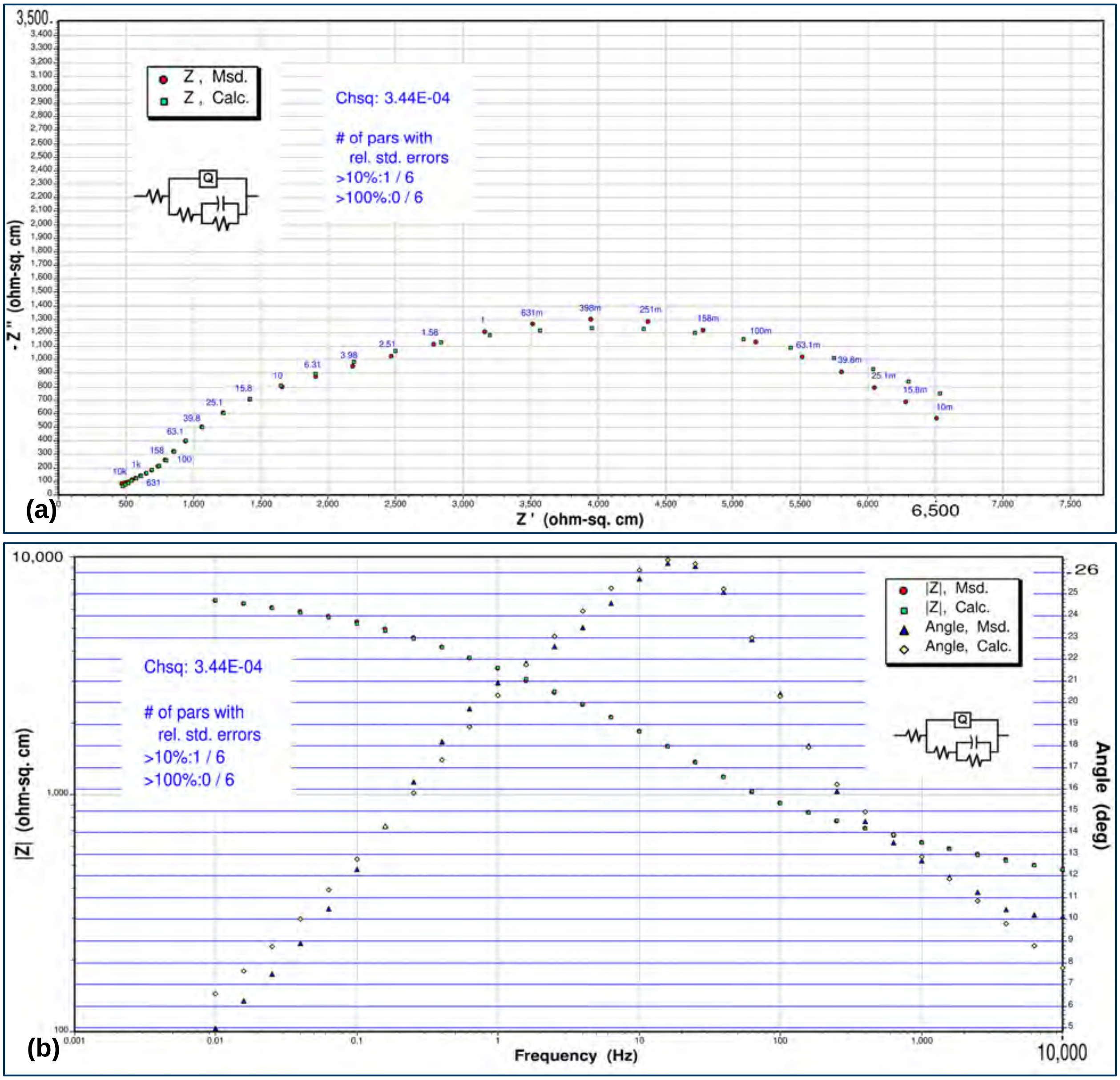


Figure 3.2 EIS modeled plots for S-LS-C-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

Table 3.2 Coating-related parameters obtained after modeling raw EIS data for S-LS-C-day 30

Exposed area of coupon S-LS-A | 9.68 cm^2			**Chi-Squared (x^2) |** 3.44 x 10^{-4}	
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
1	R_{sol} (ohm-cm^2)	3.79E+02	3.79E+02	2.7
2	Q-Yo (S-sec^n /cm^2)	1.00E-02	1.00E-04	2.6
3	Q-n	8.00E-01	3.97E-01	2.1
4	R_{po} (ohm-cm^2)	1.81E+03	1.81E+03	9.5
5	C_{dl} (F/cm^2)	1.52E-06	1.52E-06	11.2
6	R_{ct} or R_p (ohm-cm^2)	5.72E+03	5.72E+03	4.8

Note that the coating's performance was reduced quite significantly based on the reduced values of impedance (from 100 K-ohm-cm^2 to 10 K-ohm-cm^2) and phase angle (from 42^o to 10^o) in the Bode plots (Fig 3.2 b) compared to the values obtained after 1 hr of immersion. Also, the Nyquist plot in Figure 3.2a indicates a semicircle (one-time constant) which represents a deteriorated coating. Note that this one-time constant is also fairly visible in the Bode magnitude (|Z|) plot, where the plot breaks between 100 and 1000 Hz of frequency. Due to the corrosion of metal occurring in S-LS, corrosion products blocking the pores led to an increase in pore resistance (Loveday et al., 2004b), which should not be confused with improved performance. A decrease in charge transfer resistance, meaning that charge or ions were transferred between the metal and electrolyte easier than before; also indicates increased corrosion rate of the metal.

3.1.1.2 Steel-Fluid Film – NaCl-CaCl$_2$ blend

The raw data obtained for Fluid Film coupons was often cluttered due to disturbances in the cell and on the working electrode coated with Fluid Film. Moreover, the coating was easily notched or scratched with slight contact with other electrodes present in the cell. The Fluid Film also was dissolving into the solution over time and increased the complexity of the solution ions; hence resulting in not smooth Nyquist and Bode plot curves. Such cluttered data is very difficult to model and sometimes cannot be modeled because no circuit fits the shape of the plots.

The modeled Nyquist and Bode plots for S-FF-B-day1 and day 30 are added to Figures A1 and A2 in Appendix A, respectively. The corresponding values of key parameters obtained after the modeling of S-FF-B after day 1 and day 30 are provided in Table 3.3.

Table 3.3 Coating-related parameters obtained after modeling raw EIS data for S-FF-B-day 1 and day 30

Exposed area of coupon S-FF-B | 6.45 cm^2	**Chi-Squared (x^2) – day 1 plots |** 6.08 x 10^{-3} **Chi-Squared (x^2) – day 30 plots |** 2.08 x 10^{-4}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	7.01E+09	7.01E+04	83.9
2	C_c (F/cm^2)	1.68E-11	1.68E-11	3.1
3	R_{po} (ohm-cm^2)	4.71E+07	4.71E+07	25.9
4	Q-Yo (S-sec^n /cm^2)	6.28E-10	6.28E-10	19.0
5	Q-n	5.28E-01	5.28E-01	10.6
6	R_{ct} (ohm-cm^2)	7.93E+08	7.93E+08	17.5
7	W Yo (S-sec$^{0.5}$ /cm^2)	6.69E-09	6.68E-09	16.8
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	8.35E+03	8.35E+03	3.9
2	C_c (F/cm^2)	4.64E-08	4.64E-09	21.5
3	$R_{c\,t}$ (ohm-cm^2)	1.13E+05	1.13E+05	1.4
4	Q-Yo (S-sec^n /cm^2	2.63E-07	2.63E-07	5.6
5	Q-n ($0<n<1$)	8.57E-01	8.57E-01	1.0
6	R_{po} (ohm-cm^2)	6.54E+03	6.54E+03	8.2
7	L (Henri-cm^2)	1.70E+05	1.70E+05	17.1
8	R_p (ohm-cm^2)	6.08E+05	6.08E+05	7.5
9	C (F/cm^2)	7.40E-09	7.39E-09	31.4
10	*R_m (ohm-cm^2)	1.12E+04	1.12E+04	14.3

R_m is the magnetic resistance in parallel to the magnetic inductance in the circuit

After one day of testing, the Nyquist plot in Figure A 1(a) indicates very high impedance values for S-FF-B, nearly reaching 1 giga Ohm-cm^2, which suggests that the coating corrosion resistance is outstanding after one day of immersion in the NaCl+$CaCl_2$ blend salt brine. Moreover, the Bode plots (Figure A1 b) indicate the same coating's excellence by reflecting very high final |Z| and Φ values of 1.0 x 10^9 Ohms-cm^2 and 86^o respectively indicating that the coating is intact. Note that the maximum Φ is 90^o. Table 3.3 shows that very high pore resistance (R_{po}) and $R_{c\,t}$ values reflect that the Fluid Film layer is very much impervious with minimum pores available for water uptake and for corrosive ions to pass through, as well as providing a very high corrosion resistance.

Note that after 30 days of immersion in the blend salt brine (following the wet-dry cycles, as mentioned in Chapter 2), the Fluid Film layer showed significant deterioration as reflected by lower final values of Φ and |Z| (100,000 ohms-cm^2) in the Bode plots. Also, the Nyquist plot in Figure A 2(a) shows a complete semicircle indicating that the coating's performance has been compromised after 30 days of wet-dry cycles in the blend salt brine. Note that this unique shape of the Nyquist plot (semicircle curling inside at the end) occurs with magnetic materials and requires a special element (magnetic inductance, L) to be added to the circuit for better modeling results and plot fit. Steel was the only material of choice that was magnetic at room temperature and its Nyquist plots often showed these curves. Despite the deteriorating coating's performance evident from the shape of the Nyquist plot and decreasing impedance and phase angle values in the Bode plot, the coating resistance (R_{po}), though decreased, is still high relative to other coatings in the blend salt brine after 30 days. Coating capacitance is still showing a usual value of 4.6 nF / cm^2, which indicates limited level of water uptake.

3.1.1.3 Steel-Aquapon – NaCl-$CaCl_2$ blend

The modeled EIS plots for S-AP-C on day 1 after 2 hours of immersion and day 30 are shown in Figures A 3 and A 4 in Appendix A, respectively. The corresponding values of key parameters obtained after modeling raw EIS plots for S-AP-C-2hr and S-AP-C-day 30 are provided in Table 3.4.

Table 3.4 Coating-related parameters obtained after modeling raw EIS data for S-AP-C-2 hr and day 30

The exposed area of coupon S-AP-C | 15.5 cm^2	**Chi-Squared (x^2) – day 1 plots |** 9.49 x 10^{-4} **Chi-Squared (x^2) – day 30 plots |** 5.13 x 10^{-4}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 (after 2 hours) – Results				
1	R_{sol} (ohm-cm^2)	1.31E+02	5.57E-03	> 100 %
2	Q-Yo (S-sec^n /cm^2	5.96E-08	5.97E-08	15.5
3	Q-n (0<n<1)	6.25E-01	6.25E-01	4.9
4	$R_{c\,t}$ or R_p (ohm-cm^2)	1.28E+05	1.28E+05	6.7
5	W Yo (S-sec$^{0.5}$ /cm^2)	2.68E-04	2.68E-04	22.6
6	C_c (F/cm^2)	3.26E-09	3.26E-09	46.2
7	R_{po} (ohm-cm^2)	1.40E+04	1.40E+04	42.4
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	1.03E+03	1.03E+03	11.6
2	C_c (F/cm^2)	4.85E-09	4.85E-09	17.4
3	R_{po} (ohm-cm^2)	9.03E+04	9.03E+04	10.9
4	Q-Yo (S-sec^n /cm^2	3.45E-06	3.45E-06	15.7
5	Q-n (0<n<1)	8.00E-01	4.09E-01	4.4
6	$R_{c\,t}$ or R_p (ohm-cm^2)	2.21E+04	2.21E+04	2.9
7	W Yo (S-sec$^{0.5}$ /cm^2)	8.44E-05	8.44E-05	8.6

The zinc-rich epoxy primer shows good pore resistance (~10 K-ohm) on the first day of immersion, whereas the coating's capacitance is also very low and closer to the usual value which is 3 nF / cm^2.

After 30 days of wet/dry cycles in the blend salt brine, unchanged R_{po} and only a small decrease in $R_{c\,t}$ values for S-AP-C indicate that the zinc-rich epoxy primer showed excellent performance against corrosive electrolyte in wet/dry conditions and also did not absorb a lot of water as evident from almost unchanged coating capacitance (C_c). A relatively small increase in Q (from 10^{-8} to 10^{-6}) which may be considered as the C_{dl} for this case, indicates some layer formation between the metal and electrolyte.

3.1.1.4 Stainless Steel-Lubra Seal – NaCl-$CaCl_2$ blend

The modeled EIS plots for SS-LS-A on day 1 and day 30 are shown in Figures A 5 and A 6 in Appendix A, respectively. The Bode magnitude plot shows a high ending value of the impedance (10 mega ohm – cm^2), which indicates good condition of the coating. Moreover, the Bode phase angle plot shows that the angle kept increasing constantly and reached a good value of 64°, though better values could be 80° and above. 90° is the highest possible value for phase angle.

The corresponding values of key parameters obtained after modeling raw EIS plots for SS-LS-A-day 1 and day 30 are provided in Table 3.5.

Table 3.5 Coating-related parameters obtained after modeling raw EIS data for SS-LS-A-day 1 and day 30

Exposed area of coupon SS-LS-A | 9.76 cm^2	**Chi-Squared (x^2) – day 1 plots |** 6.77 x 10^{-5} **Chi-Squared (x^2) – day 30 plots |** 8.79 x 10^{-5}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	2.67E+03	2.67E+03	6.7
2	C_c (F/cm^2)	1.05E-09	1.05E-09	2.0
3	R_{po} (ohm-cm^2)	3.23E+04	3.23E+04	7.1
4	Q-Yo (S-sec^n /cm^2)	8.51E-08	8.51E-08	1.6
5	Q-n (0<n<1)	5.61E-01	5.61E-01	0.5
6	R_{ct} (ohm-cm^2)	5.27E+06	5.27E+06	2.0
7	C_{dl} (F/cm^2)	6.28E-06	6.28E-07	14.3
8	R_p (ohm-cm^2)	1.02E+06	1.02E+06	10.1
9	W (S-$sec^{0.5}$/cm^2)	1.21E-06	1.21E-06	3.3
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	1.37E+03	1.37E+03	11.8
2	C_c (F/cm^2)	2.19E-09	2.19E-09	6.5
3	R_{po} (ohm-cm^2)	7.61E+04	7.60E+03	16.8
4	Q-Yo (S-sec^n /cm^2	2.06E-07	2.06E-07	0.4
5	Q-n (0<n<1)	5.73E-01	5.73E-01	0.2
6	R_{ct} (ohm-cm^2)	4.42E+07	4.42E+07	2.2
7	C_{dl} (F/cm^2)	8.46E-08	8.46E-08	13.4
8	R_p (ohm-cm^2)	6.75E+03	6.75E+03	13.6

Stainless steel showed interesting results with Lubra Seal in NaCl+$CaCl_2$ blend salt brine over 30 days. The coating's parameters indicate that the coating remained intact with very little signs of deterioration, given the small drop in the pore resistance (from 3.23 x 10^4 to 7.6 x 10^3 ohm-cm^2) and only a small increase in the coating capacitance, which indicates a slight water uptake in 30 days. However, the decreased double-layer capacitance (C_{dl}) and the increased charge transfer resistance (R_{ct}) indicate that the stainless steel passivity increases the integrity of the coating system against corrosion. Finally, a notable decrease in the polarization resistance was observed, regardless of how good stainless steel is against corrosion. This is because of some water uptake, evident by the increase in C_c and reduced R_{po}, leading to an overall decline in the R_p.

3.1.1.5 Stainless Steel-Armour Seal – NaCl-$CaCl_2$ blend

Figures A 7 and A 8 in Appendix A show the Nyquist and Bode plots for SS-AS-C after 2 days and 30 days of testing, respectively. The corresponding values of key parameters obtained from the modeling of raw data of SS-AS-C-2D and 30 days are given in Table 3.6.

Table 3.6 Coating-related parameters obtained after modeling raw EIS data for SS-AS-C-day 2 and day 30

Exposed area of coupon SS-AS-C | 17.16 cm^2

Chi-Squared (x^2) – day 1 plots | 8.26×10^{-5}
Chi-Squared (x^2) – day 30 plots | 1.98×10^{-4}

Index	Parameters	Start value	End value	Relative standard error %
		Day 2 – Results		
1	R_{sol} (ohm-cm^2)	1.83E+02	1.83E+02	0.3
2	C_c (F/cm^2)	1.63E-06	1.63E-06	3.3
3	R_{ct} (ohm-cm^2)	2.17E+04	2.17E+04	10.4
4	Q-Yo (S-sec^n /cm^2)	5.75E-05	5.75E-05	1.5
5	Q-n (0<n<1)	8.00E-01	7.11E-01	0.3
6	R_{po} (ohm-cm^2)	5.03E+01	5.03E+01	2.4
7	C_{dl} (F/cm^2)	3.07E-05	3.07E-05	2.0
8	R_p (ohm-cm^2)	9.44E+05	9.45E+05	19.8
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	1.86E+02	1.86E+02	3.1
2	C_c (F/cm^2)	1.10E-07	1.10E-07	7.7
3	R_{po} (ohm-cm^2)	1.95E+03	1.95E+02	5.4
4	Q-Yo (S-sec^n /cm^2)	1.14E-05	1.14E-05	1.3
5	Q-n (0<n<1)	6.27E-01	6.27E-01	0.4
6	R_{ct} (ohm-cm^2)	2.30E+05	2.30E+05	6.7
7	W-Yo (S-$sec^{0.5}$ /cm^2)	1.10E-05	1.10E-05	3.4

Although the impedance values in Nyquist and Bode magnitude plots (Figures A 7a and A 7b) after 2 days of testing are not very high, the Nyquist curve (a straight line, with a slope of ~ 70^o) indicates that the coating is fully intact. Moreover, the Φ value of 75^o is excellent for a sealant immersed in a salt solution for 2 days.

After 30 days both charge resistance and pore resistance improved, relative to day 2. This means the sealant formulation has a self-healing characteristic. Moreover, stainless steel shows passivity that is a natural barrier formed that protects against corrosion. Both (R_{ct} and R_{po}) increasing and Cc decreasing indicate an overall excellent performance and resistance to the corrosive salt electrolyte.

3.1.1.6 Aluminum-Armour Seal – NaCl-CaCl$_2$ blend

Figures A 9 and A 10 in Appendix A show the Nyquist and Bode plots for Al-AS-B after 2 hours of immersion and 30 days of testing, respectively. The corresponding values of key parameters obtained from the modeling of raw data of Al-AS-B after 2 hours and 30 days of testing are given in Table 3.7.

Table 3.7 Coating-related parameters obtained after modeling raw EIS data for Al-AS-B-2 hr and day 30

Exposed area of coupon Al-AS-B | 17.17 cm^2		**Chi-Squared (x^2) – day 1 plots** | **Chi-Squared (x^2) – day 30 plots** |		5.2 x 10^{-4} 8.45 x 10^{-4}
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	2.41E+02	2.40E+02	5.3
2	C_c (F/cm^2)	7.76E-08	7.76E-08	8.1
3	R_{po} (ohm-cm^2)	4.32E+02	4.32E+02	13.4
4	Q-Yo (S-sec^n /cm^2)	1.83E-06	1.83E-06	1.3
5	Q-n (0<n<1)	7.93E-01	7.93E-01	0.4
6	R_{ct} (ohm-cm^2)	2.42E+06	2.42E+06	5.4
7	W (S-sec$^{0.5}$/cm^2)	3.93E-06	3.93E-06	14.3
8	C_{dl} (F/cm^2)	9.23E-07	9.22E-07	35.6
9	R_p (ohm-cm^2)	2.27E+02	2.27E+02	17.8
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	2.26E+02	2.26E+02	5.7
2	C_c (F/cm^2)	6.37E-08	6.37E-08	6.7
3	R_{po} (ohm-cm^2)	5.77E+02	5.77E+02	7.3
4	Q-Yo (S-sec^n /cm^2)	4.21E-06	4.21E-06	5.4
5	Q-n (0<n<1)	6.58E-01	6.58E-01	1.1
6	R_{ct} (ohm-cm^2)	1.03E+07	1.02E+06	76.1
7	W-Yo (S-sec$^{0.5}$/cm^2)	7.89E-06	7.85E-06	79.4
8	C_{dl} (F/cm^2)	6.76E-06	6.75E-06	20.3
9	R_p (ohm-cm^2)	5.95E+05	5.96E+05	29.4

The modeled Bode plots for Al-AS-B-2 hr indicate a high ϕ of 70^o though it eventually dropped to a very low phase angle. Impedance is high, nearly 1 mega ohm-cm^2, which indicates good coating health. Nyquist plot shows a Warburg impedance, but since it is not a full semicircle but rather a parabolic curve, the coating performed well. However, the data for Al-AS-B after 2 hours of immersion shows that the coating resistance (R po) is not high enough, though it could improve after a few days. Coating capacitance is at a usual value of 0.7 nF/cm^2 and C_{dl} is higher than usual at 92 µF/cm^2.

After 30 days of EIS testing including the wet/dry cycles, the coating capacitance (0.6 nF/cm^2) has a minute decrease and C_{dl} has significantly decreased to 6 µF/cm^2, which is a good indication of a superior resistance of Al-AS towards corrosion. Both C_c and C_{dl} values after 30 days show that the water uptake was very low for Al-AS and no corrosion products were formed on the metal surface due to salt solution penetrating through the pores of Armour Seal. This conclusion is cemented by the R_{po} did not change much after 30 days but rather increased a little, which also reflects the high integrity of Al-AS in a salt-laden (wet/dry) environment. An increase in R_{po} and no decrease in R_{ct} once again imply the self-healing characteristic of Armour Seal in aqueous media.

3.1.1.7 Aluminum-Fluid Film – NaCl-$CaCl_2$ blend

Day 1 (2 Hours) – Results

Figures A 11 and A 12 in Appendix A show the Nyquist and Bode plots for Al-FF-A after 5 days and 30 days of testing, respectively. The data for early hours of immersion or one day into testing could not be plotted due to cluttering in the raw plots. This happened often for Fluid Film coupons as they disturbed the solution chemistry by getting dissolved into it, causing disturbances in the cell.

The corresponding values of key parameters obtained from the modeling of raw data of Al-FF-A-5D and 30 days are given in Table 3.8.

Table 3.8 Coating-related parameters obtained after modeling raw EIS data for Al-FF-A-day 5 and day 30

The exposed area of coupon AL-FF-A | 6.45 cm^2		**Chi-Squared (x^2) – day 1 plots |** 8.58 x 10^{-3} **Chi-Squared (x^2) – day 30 plots |** 7.38 x 10^{-4}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 5 – Results		
1	R_{sol} (ohm-cm^2)	8.27E-02	8.27E-02	> 100 %
2	Q-Yo (S-sec^n /cm^2)	9.34E-12	9.34E-12	19.1
3	Q-n (n=1) *	1.00E+00	1.00E+00	2.2
4	R_{po} (ohm-cm^2)	8.28E+08	8.28E+08	43.7
5	C_{dl} (F/cm^2)	9.15E-12	9.15E-12	63.0
6	R_{ct} or R_p (ohm-cm^2)	9.05E+08	9.05E+08	39.1
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	4.31E+06	4.31E+04	22.1
2	C_c (F/cm^2)	1.47E-10	1.47E-10	7.0
3	R_{po} (ohm-cm^2)	1.82E+06	1.82E+06	4.3
4	Q-Yo (S-sec^n /cm^2)	6.58E-09	6.58E-09	1.3
5	Q-n (0<n<1)	6.52E-01	6.52E-01	0.6
6	R_{ct} (ohm-cm^2)	4.63E+08	4.63E+08	2.5
7	C_{dl} (F/cm^2)	9.67E-11	9.67E-11	5.9
8	R_p (ohm-cm^2)	3.75E+05	3.75E+05	10.6

* *For special cases (when n = 1) CPE (Q) behaves as a capacitor and from a very low value it could be the coating's capacitance*

The Fluid Film is the only coating investigated that showed the highest phase angle (90^o) after a few hours or days of immersion. The Bode plot showed very high impedance values, indicating very high pore resistance of the coating, however, the Nyquist plot indicates a coating failure that could be due to some physical defects on the coating. Keeping in mind that the Fluid Film can easily be removed by a slight touch of a fingernail or any electrode while setting up the EIS cell, it is possible the specific coupons had some defects on the film applied. Regardless of that, the data shows an R_{po} of 100 mega ohm-cm^2 and a very low C_{dl} of 9 pF/cm^2. The C_{dl} is usually above 1 µF/cm^2, but in the case of Fluid Film, it is extremely low, suggesting the difficulty of forming a metal/electrolyte interface.

After 30 days of EIS testing, Al-FF showed a drop of R_{po} (1.8 mega ohm-cm^2), but it is still high relative to several other material-coating combinations in the NaCl+$CaCl_2$ blend salt brine. The C_{dl} (and C_c) increased, indicating some water uptake and the possibility of an interface developing between the metal and the electrolyte underneath the coating. Note that the solution resistance increased manyfold (from 0.008 ohm-cm^2 to 43 K ohm-cm^2) and this is normal for a solution with Fluid Film inside. As mentioned, this refined petroleum-based protectant starts dissolving in the salt solution after a while, creating a complex network of ions, which increases the R_{sol}. Therefore, this increase is not necessarily due to the corrosion products inside the solution.

3.1.1.8 Copper-Dielectric grease – NaCl-$CaCl_2$ blend

Figures A 13 and A 14 in Appendix A show the Nyquist and Bode plots for Cu-DG-B after 1 hour and 30 days of testing, , respectively. The corresponding values of key parameters obtained from the modeling of raw data of Cu-DG-B after 1 hour of immersion and after 30 days of testing are given in Table 3.9.

Table 3.9 Coating-related parameters obtained after modeling raw EIS data for Cu-DG-B-1Hr and day 30

Exposed area of coupon Cu-DG-B | 6.45 cm^2		**Chi-Squared (x^2) – day 1 plots** | 1.68×10^{-2} **Chi-Squared (x^2) – day 30 plots** | 1.17×10^{-3}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
	Day 1 – Results			
1	R_{sol} (ohm-cm^2)	2.13E+02	2.13E+02	77.3
2	C_c (F/cm^2)	1.06E-06	1.06E-08	22.1
3	R_{po} (ohm-cm^2)	4.46E+06	4.46E+06	43.3
4	Q-Yo (S-sec^n /cm^2)	2.38E-07	2.38E-07	67.2
5	Q-n (0<n<1)	5.88E-01	5.88E-01	19.1
6	R_{ct} (ohm-cm^2)	3.43E+05	3.43E+05	31.5
7	W-Yo (S-sec$^{0.5}$/cm^2)	1.87E-06	1.87E-06	17.4
	Day 30 – Results			
1	R_{sol} (ohm-cm^2)	3.85E+01	3.85E+01	3.0
2	C_c (F/cm^2)	6.62E-07	6.62E-07	2.5
3	R_{po} (ohm-cm^2)	2.64E+02	2.64E+02	13.1
4	Q-Yo (S-sec^n /cm^2)	1.20E-04	1.20E-04	5.0
5	Q-n (0<n<1)	4.17E-01	4.17E-01	3.5
6	R_{ct} (ohm-cm^2)	3.83E+04	3.83E+04	28.6
8	W (S-sec$^{0.5}$/cm^2)	3.34E-03	3.33E-04	42.7

An increase in coating capacitance and a sharp decline in the coating resistance (R_{po}) indicates that the dielectric grease could not provide adequate resistance to corrosive electrolyte after 30 days of accelerated testing.

3.1.1.9 Copper-Deox-IT – NaCl-$CaCl_2$ blend

Figures A 15 and A 16 in Appendix A show the Nyquist and Bode plots for Cu-DO-A after 1 day (1 hour) and 30 days of testing, respectively. The corresponding values of key parameters obtained from the modeling of raw data of Cu-DO-A after 1 hour and 30 days are given in Table 3.10.

Table 3.10 Coating-related parameters obtained after modeling raw EIS data for Cu-DO-A-1Hr and day 30

The exposed area of coupon Cu-DO-A | 6.45 cm^2		**Chi-Squared (x^2) – day 1 plots** | 8.58×10^{-4} **Chi-Squared (x^2) – day 30 plots** | 9.57×10^{-5}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	1.45E+01	1.45E+01	3.4
2	C_c (F/cm^2)	9.04E-07	9.04E-07	9.6
3	R_{po} (ohm-cm^2)	5.12E+04	5.12E+04	20.6
4	Q-Yo (S-sec^n /cm^2)	9.02E-06	9.02E-06	5.6
5	Q-n (0<n<1)	7.29E-01	7.29E-01	2.3
6	R_{ct} (ohm-cm^2)	2.16E+04	2.16E+04	6.9
7	W (S-$sec^{0.5}$/cm^2)	1.35E-04	1.35E-04	26.3
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	1.93E+01	1.93E+01	1.2
2	C_c (F/cm^2)	6.30E-07	6.30E-07	1.3
3	R_{po} (ohm-cm^2)	9.39E+03	9.39E+04	7.6
4	Q-Yo (S-sec^n /cm^2)	6.63E-06	6.63E-06	1.8
5	Q-n (0<n<1)	6.43E-01	6.43E-01	0.7
6	R_{ct} (ohm-cm^2)	1.00E+05	1.00E+05	6.3
7	W (S-$sec^{0.5}$/cm^2)	4.28E-05	4.28E-05	22.7

The results after 30 days of EIS testing for Deox-IT in the blend salt brine indicate that the metal surface was protected well by Deox-IT over 30 days. Though there was some color change around the edges of the coupons at the end of testing that can be observed in Figure C 7, Appendix C, however, the coating pore resistance did not drop at all and the charge transfer resistance rather increased. The coating capacitance also did not show any increase, which indicates no water uptake in the film. Deox-IT provides good protection for copper in the blend salt brine.

3.1.2 EIS modeling results – Data for $MgCl_2$-Beet blend

Although EIS tests were performed for days 0, 1, 5, 10, 15, 20, 25, and 30 for the $MgCl_2$-Beet blend as well, only the data from day 0 or 1 and day 30 are added to this section. The remaining data can be provided electronically upon request.

3.1.2.1 Steel-Lubra Seal – $MgCl_2$-Beet blend

Day 1 – Results

Figures A 17 and A 18 in Appendix A show the Nyquist and Bode plots for S-LS-A after 1 day and 30 days of testing, respectively. The corresponding values of key parameters obtained from the modeling of raw data of S-LS-A-1D and S-LS-A-30 days are given in Table 3.11.

Table 3.11 Coating-related parameters obtained after modeling raw EIS data for S-LS-A-day 1 and day 30

Exposed area of coupon S-LS-A | 9.677 cm^2		**Chi-Squared (x^2) – day 1 plots |** 5.9 x 10^{-4} **Chi-Squared (x^2) – day 30 plots |** 2.77 x 10^{-4}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	1.64E+03	1.64E+03	7.6
2	C_c (F/cm^2)	8.96E-09	8.96E-09	15.3
3	R_{po} (ohm-cm^2)	2.00E+03	2.00E+03	4.7
4	Q-Yo (S-sec^n /cm^2)	6.18E-07	6.18E-07	2.8
5	Q-n (0<n<1)	8.00E-01	7.20E-01	0.6
6	R_{ct} (ohm-cm^2)	1.22E+06	1.22E+06	1.2
7	C_{dl} (F/cm^2)	1.26E-06	1.26E-06	16.9
8	R_p (ohm-cm^2)	2.83E+03	2.83E+04	22.9
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	1.40E+02	1.40E+02	3.5
2	C_c (F/cm^2)	2.13E-07	2.13E-07	15.2
3	R_{po} (ohm-cm^2)	7.61E+02	7.61E+01	15.7
4	Q-Yo (S-sec^n /cm^2)	4.26E-05	4.26E-05	1.2
5	Q-n (0<n<1)	5.41E-01	5.41E-01	0.6
6	R_{ct} (ohm-cm^2)	7.63E+04	7.63E+04	2.2
7	C_{dl} (F/cm^2)	2.33E-05	2.33E-05	7.3
8	R_p (ohm-cm^2)	1.26E+03	1.26E+03	6.7

The Bode magnitude plot indicates a break between 0.1 and 1 Hz, which represents the time constant, shown as a semicircle in the Nyquist plot. Though the impedance values are high (nearly 1 mega ohm-cm2), the coating itself may have flaws on the surface, because the Nyquist plot depicts a coating that is failing. Moreover, the phase angle plot suggests a coating that is deteriorating with the angle reasonably good (62^{o}) only for a while and then drops significantly. The data shows usual values for C_c (8.9 nF/cm^2) and C_{dl} (1.2 µF/cm^2) and a reasonably good R $_{po}$, after one day into the EIS testing.

After 30 days of testing, S-LS in the $MgCl_2$-Beet blend shows a significant drop in performance as the overall impedance from the Bode and Nyquist plots decreases ten times. The phase angle also dropped, and the data shows that the C_c increased 100 times, indicating that a considerable amount of water uptake occurred during the 30 days of intermittent immersion of S-LS. Overall, the coating resistance and the polarization resistance both declined, while the double-layer capacitance increased. All of this indicates that the electrolyte penetrated through the coating and formed corrosion products as an interface between the metal and electrolyte.

3.1.2.2 Steel-Fluid Film – $MgCl_2$-Beet blend

Day 1 – Results

Figures A 19 and A 20 in Appendix A show the Nyquist and Bode plots for S-FF-A-1D and S-FF-A-30 days, respectively. The corresponding values of key parameters obtained from the modeling of raw data of S-FF-A after 1 day and 30 days are given in Table 3.12.

Table 3.12 Coating-related parameters obtained after modeling raw EIS data for S-FF-A-day 1 and day 30

Exposed area of coupon S-FF-A | 9.677 cm^2	**Chi-Squared (x^2) – day 1 plots |** 4.68 x 10^{-4} **Chi-Squared (x^2) – day 30 plots |** 1.69 x 10^{-4}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	2.69E+03	2.69E+03	15.9
2	C_c (F/cm^2)	1.62E-08	1.62E-08	9.2
3	R_{po} (ohm-cm^2)	2.53E+06	2.53E+05	17.8
4	Q-Yo (S-sec^n /cm^2)	1.66E-07	1.66E-07	7.2
5	Q-n ($0<n<1$)	5.52E-01	5.52E-01	1.4
6	R_{ct} (ohm-cm^2)	2.97E+06	2.97E+06	1.6
7	C_{dl} (F/cm^2)	4.46E-08	4.46E-08	21.2
8	R_p (ohm-cm^2)	4.32E+05	4.32E+05	12.2
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	3.38E+05	3.38E+04	15.6
2	C_c (F/cm^2)	1.57E-09	1.57E-09	13.6
3	R_{po} (ohm-cm^2)	4.58E+05	4.58E+05	16.0
4	Q-Yo (S-sec^n /cm^2)	8.21E-09	8.21E-09	10.2
5	Q-n ($0<n<1$)	5.80E-01	5.80E-01	1.8
6	R_{ct} (ohm-cm^2)	5.82E+06	5.82E+06	2.5
7	C_{dl} (F/cm^2)	1.09E-07	1.09E-07	28.5
8	R_p (ohm-cm^2)	7.06E+05	7.06E+05	22.0

Fluid Film has shown remarkable results in EIS testing in this blend salt brine. Beet juice is a corrosion inhibitor that helped retard the corrosion and potentially slowed down the deterioration of the lubricant. But even on its own Fluid Film is an excellent protectant of steel. Apart from a slight increase in double-layer capacitance, all other parameters after 30 days of testing show no significant difference in performance from day 1 to day 30. The lubricant has yielded the highest phase angle and impedance values when compared with all other results for all products in both blends. The Bode plots after one day show a good phase angle (56°) but it dropped down to only 20°. Impedance is very high (over a mega ohms-cm^2) after a day of immersion for S-FF in the blend salt brine. After 30 days, the highest phase angle value is 48° and dropped to zero, indicating deteriorating protection. The impedance value of |Z| is still very high, but the shape of the Nyquist plot tells that the coating is failing and developing a low pore resistance.

3.1.2.3 Steel-Aquapon – $MgCl_2$-Beet blend

Figures A 21 and A 22 in Appendix A show the Nyquist and Bode plots for S-AP-C-1D and S-AP-C- 30 days, respectively. The corresponding values of key parameters obtained from modeling raw data of S-AP-C after 1 day and 30 days are given in Table 3.13.

Table 3.13 Coating-related parameters obtained after modeling raw EIS data for S-AP-C-day 1 and day 30

Exposed area of coupon S-AP-C | 9.677 cm^2	**Chi-Squared (x^2) – day 1 plots |** 1.824 x 10^{-4} **Chi-Squared (x^2) – day 30 plots |** 3.163 x 10^{-4}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	7.87E+01	7.87E+01	8.0
2	C_c (F/cm^2)	1.28E-07	1.28E-07	17.1
3	R_{po} (ohm-cm^2)	8.87E+01	8.87E+01	8.6
4	Q-Yo (S-sec^n /cm^2)	1.82E-05	1.81E-05	4.7
5	Q-n (0<n<1)	8.00E-01	6.25E-01	1.1
6	R_{ct} (ohm-cm^2)	3.00E+03	3.00E+03	3.2
7	L (Henri-cm^2)	4.18E+03	4.18E+03	12.8
8	R_m (ohm-cm^2)	8.87E+02	8.87E+02	5.9
9	C_{dl} (F/cm^2)	9.48E-05	9.48E-05	11.5
10	R_p (ohm-cm^2)	1.25E+04	1.25E+03	6.3
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	9.80E+01	9.80E+01	10.1
2	Q-Yo (S-sec^n /cm^2)	1.00E-05	1.00E-05	4.3
3	Q-n (0<n<1)	8.00E-01	5.13E-01	1.1
4	R_{po} (ohm-cm^2)	1.81E+04	1.81E+04	4.4
5	Q-Yo (S-sec^n /cm^2)	1.18E-04	1.18E-04	8.5
6	Q-n (0<n<1)	8.00E-01	5.70E-01	7.7
7	R_{ct} or R_p (ohm-cm^2)	3.71E+03	3.71E+04	12.6

Based on the data after 30 days of testing, Aquapon (zinc-rich epoxy primer) provides excellent protection for steel against corrosive media. Both R_{po} and R_{ct} for S-AP increased after 30 days of testing. This is a typical behavior of a galvanized layer that further improves its resistance against corrosion after in contact with water, due to its self-healing properties. The modeled plots after 1 day indicate a low phase angle of 42° that dropped to zero and the |Z| is also low (only 10 K Ohms). The Nyquist plot is a semi-circle indicating poor coating health. However, after 30 days, the Nyquist plot curve and the impedance value in the Bode plot (Fig. A 22) both indicate an improvement in the coating's health, which is expected of the galvanized layer.

3.1.2.4 Stainless Steel-Lubra Seal – $MgCl_2$-Beet blend

Day 1 – Results

Figures A 23 and A 24 Appendix A show the Nyquist and Bode plots for SS-LS-B after 1 day and 30 days of testing, respectively. The corresponding values of key parameters obtained from modeling raw data of SS-LS-B-1D and SS-LS-B-30 days are given in Table 3.14.

Table 3.14 Coating-related parameters obtained after modeling raw EIS data for SS-LS-B-day 1 and day 30

Exposed area of coupon SS-LS-B | 9.67 cm^2	**Chi-Squared (x^2) – day 1 plots** | 2.16×10^{-3} **Chi-Squared (x^2) – day 30 plots** | 5.06×10^{-3}

Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	4.64E+03	4.64E+03	7.3
2	C_c (F/cm^2)	5.73E-09	5.73E-09	17.3
3	R_{ct} (ohm-cm^2)	2.04E+05	2.04E+06	60.2
4	Q-Yo (S-sec^n /cm^2)	1.09E-06	1.09E-06	8.4
5	Q-n (0<n<1)	6.10E-01	6.10E-01	2.6
6	R_{po} (ohm-cm^2)	4.98E+03	4.98E+03	9.7
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	2.54E+03	2.54E+03	9.2
2	C_c (F/cm^2)	1.05E-08	1.05E-08	16.7
3	R_{po} (ohm-cm^2)	3.84E+03	3.84E+03	11.3
4	Q-Yo (S-sec^n /cm^2)	1.46E-06	1.46E-06	5.6
5	Q-n (0<n<1)	6.29E-01	6.29E-01	1.9
6	R_{ct} (ohm-cm^2)	1.62E+05	1.62E+05	18.6
7	C_{dl} (F/cm^2)	3.10E-07	3.10E-07	17.9
8	R_p (ohm-cm^2)	3.21E+07	3.21E+07	132.6

Lubra Seal when applied on stainless steel show excellent resistance against corrosive salt solution. Though the coating itself showed very low impedance from the beginning, the fact that the pore resistance did not drop and R_{ct} also only showed a small decline (from 2 mega ohm to 0.16 mega ohm) makes this sealant a good choice for stainless steel. A small increase in C_c tells that some water uptake occurred in

30 days. Nevertheless, stainless steel may very well remain protected without any coating on it due to its passivity phenomenon. Overall, any coating on stainless steel would show good resistance against corrosion.

Modeled plots after a day of testing show a high impedance of 1 mega-ohm and a good phase angle of 50^o which is not dropping. Nyquist plot shows a straight line of slope of more than 70^o, which is an indication of excellent coating health and that it is fully intact. After 30 days the phase angle further improves (60^o) and is not dropping as well. Impedance is still in mega ohms, whereas the Nyquist plot is also still a straight line with a slope closer to 70^o. Modeled plots therefore indicate the SS-LS combination has not deteriorated at all and rather improved its resistance, perhaps due to the stainless steel passivity characteristic.

3.1.2.5 Stainless Steel-Armour Seal – $MgCl_2$-Beet blend

Figures A 25 and A 26 in Appendix A show the Nyquist and Bode plots for SS-AS-B-1D and SS-AS-B-30 days. Various process parameters obtained from modeling raw data of SS-AS-B-1D and SS-AS-B-30 days are given in Table 3.15.

Table 3.15 Coating-related parameters obtained after modeling raw EIS data for SS-AS-B-day 1 and day 30

Exposed area of coupon SS-AS-B | 9.67 cm^2

Chi-Squared (x^2) – day 1 plots | 1.41×10^{-4}
Chi-Squared (x^2) – day 30 plots | 3.49×10^{-4}

Index	Parameters	Start value	End value	Relative standard error %
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	3.88E+01	3.88E+01	1.0
2	C_c (F/cm^2)	2.23E-06	2.23E-06	4.3
3	R_{ct} (ohm-cm^2)	1.03E+05	1.03E+05	31.2
4	Q-Yo (S-sec^n /cm^2)	6.34E-05	6.34E-05	3.3
5	Q-n (0<n<1)	8.00E-01	6.98E-01	0.5
6	R_{po} (ohm-cm^2)	3.16E+01	3.16E+01	5.7
7	C_{dl} (F/cm^2)	6.36E-05	6.36E-05	5.7
8	R_p (ohm-cm^2)	1.89E+06	1.89E+06	16.2
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	2.57E+01	2.57E+01	1.9
2	C_c (F/cm^2)	2.47E-06	2.47E-06	8.4
3	R_{po} (ohm-cm^2)	2.07E+02	2.07E+01	17.3
4	Q-Yo (S-sec^n /cm^2)	1.07E-04	1.07E-04	12.2
5	Q-n (0<n<1)	6.41E-01	6.41E-01	2.3
6	R_{ct} (ohm-cm^2)	8.25E+04	8.24E+04	195.4
7	C_{dl} (F/cm^2)	8.43E-05	8.43E-05	21.4
8	R_p (ohm-cm^2)	4.54E+05	4.54E+05	19.6

Armour Seal did not show very good pore resistance from day one of the test, which is why the coating capacitance was also high on day one. However, over the test period of 30 days, it showed good resistance against the corrosive media as the values of R_{po}, $R_{c\,t}$, and C_{dl} did not change. For stainless steel, R_p is usually high and for the SS-AS system, it did not drop much. Keep in mind that R_p (polarization resistance) tells about the corrosion occurring on the metal surface and it would only decrease significantly when the corrosive ions through the electrolyte penetrate through the coating reaching the metal, causing its corrosion. Therefore, for stainless steel, this should be high unless the passive film is compromised.

The Bode and Nyquist plots after a day show a high |Z| value reaching 1 mega-ohm and the phase angle (74^o) is even better than SS-LS. The Nyquist plot shows a straight line with a slope (~80^o) even higher than its counterpart in SS-LS. After 30 days the Nyquist plot is still a relatively straight line with a very high slope, though it is curving a little suggesting that SS-AS developed a low pore resistance. The impedance remained high (approaching a mega-ohm) and the phase angle remained at 74^o. Overall, the modeled plots of SS-AS indicate that Armour Seal showed better performance than Lubra Seal (SS-LS).

3.1.2.6 Aluminum-Armour Seal – $MgCl_2$-Beet blend

Figures A 27 and A 28 in Appendix A show the Nyquist and Bode plots for AL-AS-B-1D and Al-AS-B-30 days, respectively. The corresponding values of key parameters obtained from modeling raw data of AL-AS-B after 1 day and 30 days of testing are given in Table 3.16.

Table 3.16 Coating-related parameters obtained after modeling raw EIS data for Al-AS-B-day 1 and day 30

Exposed area of coupon AL-AS-B | 9.67 cm^2		**Chi-Squared (x^2) – day 1 plots |** 1.16 x 10^{-3} **Chi-Squared (x^2) – day 30 plots |** 3.29 x 10^{-2}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	5.58E+01	5.58E+01	4.0
2	C_c (F/cm^2)	2.77E-06	3.58E-06	15.4
3	R_{po} (ohm-cm^2)	5.64E+04	3.47E+03	30.2
4	Q-Yo (S-sec^n /cm^2)	2.85E-06	2.85E-06	5.0
5	Q-n (0<n<1)	8.15E-01	8.15E-01	0.6
6	$R_{c\,t}$ (ohm-cm^2)	5.79E+05	5.79E+05	2.0
7	C_{dl} (F/cm^2)	3.58E-06	2.77E-06	8.9
8	R_p (ohm-cm^2)	3.47E+05	5.64E+04	14.2
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	2.96E+01	2.96E+01	9.0
2	C_c (F/cm^2)	3.43E-06	3.43E-06	11.0
3	$R_{c\,t}$ (ohm-cm^2)	2.44E+04	2.44E+04	6.7
4	Q-Yo (S-sec^n /cm^2)	6.96E-06	6.96E-06	23.0
5	Q-n (0<n<1)	9.01E-01	9.01E-01	5.8

Exposed area of coupon AL-AS-B | 9.67 cm^2		**Chi-Squared (x^2) – day 1 plots** | **Chi-Squared (x^2) – day 30 plots** |		1.16 x 10^{-3} 3.29 x 10^{-2}
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
6	R_{po} (ohm-cm^2)	3.59E+03	3.60E+02	41.3

Once again, the AL-AS system showed good corrosion resistance against chloride salt, evident from not a significant decrease in R_{po} and $R_{c\,t}$. This comes from the fact that aluminum itself has the anodizing characteristic that protects it from corrosion attack even when not coated. Though the pore resistance of Armour Seal is generally low, the lack of change in C_c indicates that water uptake was very low in 30 days of the test period. Overall, the Al-AS combination degraded but not to a great extent. The modeled plots after a day show a very good value for phase angle (74^{o}), but it dropped down to 10o. Impedance is high and is approaching one mega-ohm. The Nyquist plot for Al-AS after a day, unlike SS-AS, is showing a semi-circle development, indicative of the coating's degradation. After 30 days, the Bode plot shows the phase angle remained high but again it dropped to zero. Impedance has dropped significantly from 1 mega-ohm to 40 K-ohms, which indicates that the coating developed a low pore resistance. The Nyquist plot also indicates the deterioration of the coating after 30 days and that the Al-AS system is failing.

3.1.2.7 Aluminum-Fluid Film – $MgCl_2$-Beet blend

Day 1 – Results

Figures A 29 and A 30 in Appendix A show the Nyquist and Bode plots for Al-FF-A-1D and Al-FF-A-30 days, respectively. The corresponding values of key parameters obtained from modeling raw data of Al-FF-A-1D Al-FF-A-30 days are given in Table 3.17.

Table 3.17 Coating-related parameters obtained after modeling raw EIS data for Al-FF-A-day 1 and day 30

Exposed area of coupon Al-FF-A | 9.67 cm^2		**Chi-Squared (x^2) – day 1 plots** | **Chi-Squared (x^2) – day 30 plots** |		2.27 x 10^{-3} 3.6 x 10^{-3}
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	1.41E+08	1.41E+04	> 100 %
2	C_c (F/cm^2)	1.13E-11	1.13E-11	1.6
3	R_{po} (ohm-cm^2)	7.40E+08	7.40E+08	29.5
4	C_{dl} (F/cm^2)	5.99E-12	5.99E-12	85.7
5	$R_{c\,t}$ or R_p (ohm-cm^2)	5.13E+08	5.13E+08	42.3
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	1.07E+06	1.07E+05	31.5
2	C_c (F/cm^2)	1.99E-10	1.99E-10	15.0

Exposed area of coupon Al-FF-A | 9.67 cm^2		**Chi-Squared (x^2) – day 1 plots |** **Chi-Squared (x^2) – day 30 plots |**		2.27 x 10^{-3} 3.6 x 10^{-3}
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
3	R_{po} (ohm-cm^2)	3.98E+06	3.98E+06	6.5
4	Q-Yo (S-sec^n /cm^2)	1.45E-08	1.45E-08	2.8
5	Q-n (0<n<1)	6.58E-01	6.58E-01	2.2
6	R_{ct} (ohm-cm^2)	2.67E+08	2.67E+08	7.2
7	C_{dl} (F/cm^2)	3.00E-11	3.00E-11	4.5
8	R_p (ohm-cm^2)	2.57E+06	2.57E+06	7.5

The very low C_{dl} values for Fluid Film suggest that its ability to stop water from being penetrated through its surface is outstanding. This is why perhaps it protects the metal surface so well in salt-laden environments. Normally the C_{dl} value is in µF/cm^2, but for this lubricant, the values are as low as for coating capacitance – in nano F/cm^2. A very high pore resistance (740 mega ohms) was noticed on the first day of testing for Al-FF, which dropped to 4 mega ohms after 30 days of testing. Once again, the lack of change in R_{ct} indicates that no layer between the metal surface and the electrolyte formed because water could not work its way through the coating. However, some increase in C_c (from 10^{-11} to 10^{-10} F/cm^2) suggests that some water uptake did occur.

The modeled Nyquist and Bode plots after day 1 indicate that Fluid Film is an outstanding barrier for aqueous media, because of its highest phase angle value of 90^o (though dropped down to zero) and a very high impedance value of one giga-ohm. This high |Z| value was only seen with Fluid Film, indicating a very high pore resistance. The Nyquist plot however shows a semi-circle that occurs due to the poor health of the coating. After 30 days the coating showed signs of deterioration and its health declined significantly, as the impedance value in the Bode plot dropped to only 40 K-ohms and the highest phase angle also reached a lower value of 74^o before it dropped to zero. The Nyquist plot again showed a semi-circle.

3.1.2.8 Copper-Dielectric grease – $MgCl_2$-Beet blend

Day 1 – Results

Figures A 31 and A 32 in Appendix A show the Nyquist and Bode plots for Cu-DG-A-1D and Cu-DG-A-30 days, respectively. The corresponding values of key parameters obtained from modeling raw data of Cu-DG-A-1D and 30 days are given in Table 3.18.

Table 3.18 Coating-related parameters obtained after modeling raw EIS data for Cu-DG-A-day 1 and day 30

Exposed area of coupon Cu-DG-A | 9.677 cm^2	**Chi-Squared (x^2) – day 1 plots** | 3.49 x 10^{-4} **Chi-Squared (x^2) – day 30 plots** | 5.98 x 10^{-4}

Index	Parameters	Start value	End value	Relative standard error %
Day 1 – Results				
1	R_{sol} (ohm-cm^2)	1.64E+03	1.64E+03	10.3
2	C_c (F/cm^2)	2.56E-04	2.56E-08	9.1
3	R_{po} (ohm-cm^2)	4.86E+03	4.86E+03	4.1
4	Q-Yo (S-sec^n /cm^2)	4.19E-06	4.19E-06	0.9
5	Q-n (0<n<1)	8.00E-01	5.30E-01	0.7
6	$R_{c\,t}$ (ohm-cm^2)	1.13E+06	1.14E+06	3.4
7	C_{dl} (F/cm^2)	5.56E-09	5.56E-09	11.9
8	R_p (ohm-cm^2)	3.05E+03	3.05E+03	5.1
Day 30 – Results				
1	R_{sol} (ohm-cm^2)	4.56E+01	4.56E+01	3.3
2	C_c (F/cm^2)	1.37E-06	1.37E-06	13.3
3	R_{po} (ohm-cm^2)	2.13E+04	2.13E+02	27.2
4	Q-Yo (S-sec^n /cm^2)	2.24E-05	2.24E-05	1.2
5	Q-n (0<n<1)	8.00E-01	6.49E-01	0.6
6	$R_{c\,t}$ (ohm-cm^2)	2.92E+05	2.92E+05	3.5
7	C_{dl} (F/cm^2)	1.39E-06	1.39E-06	13.5
8	R_p (ohm-cm^2)	2.73E+01	2.73E+01	26.1

From the results of EIS, the performance of dielectric grease when applied to copper was not satisfactory in the $MgCl_2$-Beet blend. For lubricants, C_{dl} values after day one are very low, usually ranging from 10 – 50 µF/cm^2, but here it is in nF/Cm^2. This is not surprising as lubricants like dielectric grease, deox-IT, and Fluid Film can provide an excellent barrier for water to penetrate through. In this case, however, it only lasts for a while, and at the end of the test period, a sharp increase in both C_{dl} and C_c indicates significant water absorption and double-layer formation between the metal-electrolyte interface. The coating resistance dropped and so did the polarization resistance, meaning the dielectric grease failed to protect the copper in corrosive conditions. The modeled plots show a good Nyquist curve (a relatively straight line with a slope of ~50^o) and the phase angle in Bode plots reaches a very good value of 65o, dropped down, but again approached an angle of 65^o. The impedance curve in the Bode plot for capacitance magnitude (|C|, in case of a dielectric system) remained a straight line, with no breaks. After 30 days, the |C| value remained the same, and the highest phase angle value remained high (65^o) though it dropped to 20^o and then rose again to 58^o. Nyquist curve also remained a straight line with a slope angle of nearly 50^o. Overall, the Cu-DG combination performed well in the $MgCl_2$-Beet blend under accelerated corrosion conditions.

3.1.2.9 Copper-Deox-IT – MgCl$_2$-Beet blend

Day 1 – Results

Figures A 33 and A 34 in Appendix A show the Nyquist and Bode plots for Cu-DO-B-1D and 30 days, respectively. The corresponding values of key parameters obtained from modeling raw data of Cu-DO-B-1D and 30 days are given in Table 3.19.

Table 3.19 Coating-related parameters obtained after modeling raw EIS data for Cu-DO-B-1D and day 30

Exposed area of coupon Cu-DO-B | 9.677 cm^2		**Chi-Squared (x^2) – day 1 plots |** 6.85 x 10^{-4} **Chi-Squared (x^2) – day 30 plots |** 3.53 x 10^{-4}		
Index	**Parameters**	**Start value**	**End value**	**Relative standard error %**
		Day 1 – Results		
1	R_{sol} (ohm-cm^2)	4.83E+02	4.83E+02	13.4
2	C_c (S-sec^n /cm^2)	1.12E-08	1.12E-08	6.5
3	R_{po} (0<n<1)	2.27E+03	2.27E+03	5.7
4	Q-Yo (ohm-cm^2)	5.24E-06	5.24E-06	1.1
5	Q-n (F/cm^2)	8.00E-01	5.93E-01	0.7
6	R_{ct} (ohm-cm^2)	1.38E+06	1.38E+06	4.9
7	C_{dl} (F/cm^2)	1.83E-07	1.83E-07	25.4
8	R_p (ohm-cm^2)	1.22E+03	1.22E+03	12.4
		Day 30 – Results		
1	R_{sol} (ohm-cm^2)	1.58E+01	1.58E+01	1.3
2	C_c (F/cm^2)	7.98E-06	7.98E-06	3.7
3	R_{po} (ohm-cm^2)	2.06E+02	2.06E+02	10.0
4	Q-Yo (S-sec^n /cm^2)	3.27E-05	3.27E-05	1.6
5	Q-n (0<n<1)	7.71E-01	7.71E-01	0.6
6	R_{ct} (ohm-cm^2)	3.05E+04	3.05E+04	1.0
7	C_{dl} (F/cm^2)	1.38E-05	1.38E-05	8.2
8	R_p (ohm-cm^2)	8.01E+00	8.01E+00	16.0

The pore or coating's resistance decreased after 30 days for Deox IT in the MgCl$_2$-Beet blend. The polarization resistance (R_p) decreased sharply after 30 days indicating corrosion occurring at the metal surface, which means that the Doex-IT failed to protect the copper in the MgCl$_2$-Beet blend in this accelerated corrosion test. The charge transfer also increased between the metal and electrolyte as the R_{ct} dropped significantly, which is also supported by an increase in C_{dl} after 30 days of testing. Water uptake occurred, which is evident from an increase in the coating's capacitance value. Overall, this lubricant is not an effective protectant of copper in corrosive conditions for longer periods (e.g., 30 days).

After day 1, the modeled plots show the highest phase angle of 45^o that dropped down to 34^o, whereas the impedance value is approaching a high value of 1 mega-ohm-cm^2. The Nyquist curve is a straight line with a slope of a little more than 45^o. After 30 days, the Nyquist curve was a semi-circle, and |Z| in the

Bode plot decreased to ~40 Kohms-cm^2. The highest value of phase angle improved to 74^o, though it dropped below 10^o. In summary, the Deox-IT performance was not satisfactory in the $MgCl_2$-Beet blend.

3.2 Salt spray test – Results

3.2.1 Corrosion Rates for S-LS, Cu-DG, and Cu-DO

The corrosion rates for steel-Lubra Seal coupons (A, B, C, and D), at different intervals during the test period, are provided in Table 3.20. Exposure time after 45 cycles was 1056 hours and after 60 cycles was 1416 hours. The details for the weight loss can be provided electronically on demand as an Excel spreadsheet.

Table 3.20 Corrosion rates for S-LS coupons (A, B, C, and E) at various intervals during the testing period

Corrosion Rates for Steel-Lubra Seal (S-LS)

Dec. 4, 2023 (45 Cycles)				
Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Corrosion rate (mpy)	2.165	2.543	1.649	3.050
Dec. 20, 2023 (60 Cycles)**				
Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Corrosion rate (mpy)	8.770	7.732	7.008	9.276

**On test day 31, the S-LS coupons were cleaned and weighed for corrosion rate calculations for the first time*

***Coupons were cleaned and washed with acetone on the last day of testing (Dec 19, 60 cycles) and were placed in the oven for 12 hours before their weights could be taken the next day on Dec 20*

The density for steel was 7.86 g/cm^3 and the area of coupons was 48.39 cm^2 (7.5 in^2). The corrosion rates for Cu-DG coupons (A, B, C, and D) are provided in Table 3.21. The density for copper was used as 8.94 g/cm^3 and the area of coupons was 12.90 cm^2 (2 in^2).

Table 3.21 Corrosion rates for Cu-DG coupons (A, B, C, and D) at the end of the testing

Corrosion Rates for Copper-Dielectric grease (Cu-DG)

Dec. 20, 2023 (61 Cycles)				
Replicates	Cu-DG-A	Cu-DG-B	Cu-DG-C	Cu-DG-D
Corrosion rate (mpy)	2.302	2.851	2.725	3.253

The corrosion rates for Cu-DO coupons (A, B, C, and D) are provided in Table 3.22.

Table 3.22 Corrosion rates for Cu-DO coupons (A, B, C, and D) at the end of the testing

Corrosion Rates for Copper-Dielectric grease (Cu-DG)				
Dec. 20, 2023 (61 Cycles)				
Replicates	Cu-DO-A	Cu-DO-B	Cu-DO-C	Cu-DO-D
Corrosion rate (mpy)	2.154	1.563	1.816	1.352

For determining the rust grade on the S-LS coupons, ASTM D610 - 08 (ASTM International, 2019a) and for rust creepage ASTM D1654 (ASTM International, 2016), were followed, respectively. To measure the percentage area covered by rust on S-LS coupons Image J was utilized, as shown in Figure 3.3, in which the total rusted area in the 3in x 2.5in coupon was calculated as 52.3%. Image J was also followed to calculate the rust creepage after every 10 cycles, for the steel-Lubra Seal coupons.

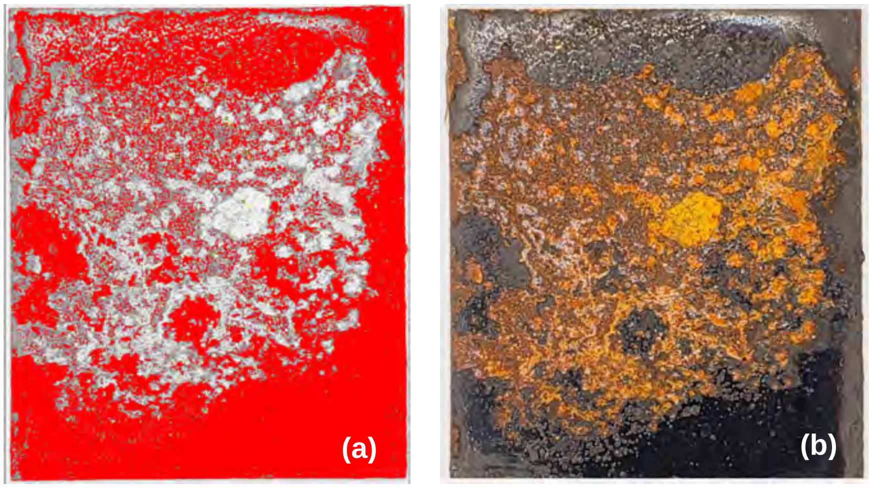


Figure 3.3 Area quantification in Image J for S-LS-B after 43 cycles: (a) Image J 8-bit split channel, Red (b) Original image taken under a lightbox

The results for rust grades for S-LS, and rust creepage at every ten cycles during the test period, are provided in Table 3.23.

Table 3.23 Rust grades and creepage rating for S-LS coupons at every 10-cycle interval during the test period

Rust Grades for Steel-Lubra Seal (S-LS)

10 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade (percent)	3-P (16%)	2-P (18%)	2-P (19%)	2-H (23%)
Creepage Rating	9	9	9	9

20 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade (percent)	2-H (30%)	2-H (28%)	1-H (35%)	1-H (36%)
Creepage Rating	9	8	8	9

30 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade (percent)	1-H (37%)	1-H (41%)	1-H (38%)	1-H (35%)
Creepage Rating	8	5	7	6

40 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade (percent)	1-H (50%)	1-H (51%)	None-G (52%)	None-G (69%)
Creepage Rating	6	2	1	2

50 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade	*None-G (65%)	*None-G (69%)	*None-G (79%)	*None-G (86%)
Creepage Rating	**0	0	0	0

60 Cycles

Replicates	S-LS-A	S-LS-B	S-LS-C	S-LS-E
Rust grade	*None-G (60.6%)	*None-G (61.8%)	*None-G (84%)	*None-G (82.6%)
Creepage Rating	0	0	0	0

**None indicates a rust grade which is for rusting coverage of higher than 50%* (ASTM International, 2019a)

***A creepage rating of 0 is for creepage of 16mm or more*

3.3 Adhesion Test – Results

The adhesion strength results in pounds per square inch (psi) and the types of failure for the hard coatings used in this study are given in Table 3.24.

Table 3.24 Adhesion strength results along with the type of failures that occurred for hard coatings

Aluminum-Armour Seal

Coupons	Al-AS-1	Al-AS-2	Al-AS-3	Al-AS-4	Al-AS-5
Strength (psi)	430	433	101	531	451
Type of failure	Adhesive	Adhesive	Priming issues	Adhesive	Adhesive

Steel-Lubra Seal

Coupons	S-LS-1	S-LS-2	S-LS-3	S-LS-4	S-LS-5
Strength (psi)	104	797	1025	-	-
Type of failure	Priming issues	Glue Failure	Glue Failure	Not performed	Not performed

Steel-Aquapon

Coupons	S-AP-1	S-AP-2	S-AP-3	S-AP-4	S-AP-5
Strength (psi)	1923	706	1209	1711	1510
Type of failure	Cohesive	Cohesive	Cohesive	Cohesive	Cohesive

Stainless Steel-Armour Seal

Coupons	SS-AS-1	SS-AS-2	SS-AS-3	SS-AS-4	SS-AS-5
Strength (psi)	691	414	453	461	547
Type of failure	Cohesive	Cohesive	Cohesive	Cohesive	Cohesive

Stainless Steel-Lubra Seal

Coupons	SS-LS-1	SS-LS-2	SS-LS-3	SS-LS-4	SS-LS-5
Strength (Psi)	654	578	542	677	706
Type of failure	Glue Failure	Glue Failure	Glue Failure	Glue Failure	Glue Failure

This test was not suitable for soft coatings such as Fluid Film, dielectric grease, and Deox IT.

Based on the adhesion strength results, Aquapon by PPG Industries, Inc. showed the highest bond strength when applied to Steel. Armour Seal when applied on stainless steel showed a little higher strength than on aluminum coupons. For Lubra Seal, no concrete results were obtained as the glue failure occurred for all of the coupons with Lubra Seal on them.

3.4 Pencil hardness test – Results

Results from the pencil hardness test are given below in Table 3.25.

Table 3.25 Pencil hardness test results for Al-AS, S-LS, S-AP, SS-AS, and SS-LS

Coupons	**Hardness Scale (with 4.9N device)**		
Al-AS	Gouge	Scratch	Mark
	All	N/A	N/A
S-LS	Gouge	Scratch	Mark
	All	N/A	N/A
S-AP	Gouge	Scratch	Mark
	None	5H (with additional 250g)	B
SS-AS	Gouge	Scratch	Mark
	All	N/A	N/A
SS-LS	Gouge	Scratch	Mark
	All	N/A	N/A

This test was not suitable for soft coatings such as Fluid Film, dielectric grease, and Deox IT.

Both Armour Seal and Lubra Seal were too soft to resist even the softest pencil (6B), which easily cut through the coated surfaces of S-LS, SS-LS, SS-AS, and Al-AS coupons down to the substrate material. Therefore, the results indicate ‘All’ for gouge for the coupons with sealants. Since the surfaces were gouged down by all the leads, results for a scratch and a mark were not applicable. However, for the Aquapon (epoxy primer), the hardest lead could only leave a mark on the coupons, when the regular weight (device’s weight) of 500g (4.9N) was used. With an added weight of 250g on the top of the device, both 6H and 5H produced a scratch on the surface of the Aquapon on steel. Mark was produced without added weight by leads from 6H to B (hardest to softest).

Therefore, for S-AP, the Vickers hardness test was also performed, to have another type of hardness test result for this coating.

3.5 Vickers hardness test – Results

The results from the Vickers hardness test for Steel-Aquapon are given in Table 3.26.

Table 3.26 Vickers hardness results for steel-Aquapon coupons

	Steel-Aquapon		
Coupons	S-AP-1	S-AP-2	S-AP-3
Vickers Hardness (HV)	157.4	166.6	159.4

The results indicate typical hardness values for a zin-rich coating on steel. Only three coupons were used because two of them were already utilized in the pencil hardness test.

3.6 Coating thickness measurements

3.6.1 Using a microscope camera

Steel-Aquapon (S-AP)

Table 3.27 Coating thickness measurements for Aquapon® (zinc-rich epoxy primer) applied on steel

Coating Thickness for Steel-Aquapon (S-AP)						
No. of Readings	1	2	3	4	5	Average
Thickness (mm)	0.14	0.13	0.16	0.13	0.14	0.14 mm
Thickness (inch)	0.01	0.01	0.01	0.01	0.01	0.01 inch
Thickness (microns)	143.04	127.15	155.49	127.15	137.74	138.11 µm
Thickness (mils)	5.63	5.01	6.12	5.01	5.42	5.44 mils

Steel-Lubra Seal (S-LS)

Table 3.28 Coating thickness measurements for Lubra Seal® applied on steel

Coating Thickness for Steel-Lubra Seal (S-LS)						
No. of Readings	1	2	3	4	5	Average
Thickness (mm)	0.08	0.07	0.05	0.04	0.05	0.06 mm
Thickness (inch)	0.00	0.00	0.00	0.00	0.00	0.00 inch
Thickness (microns)	81.23	65.34	47.68	37.08	54.74	57.21 microns
Thickness (mils)	3.20	2.57	1.88	1.46	2.16	2.25 mils

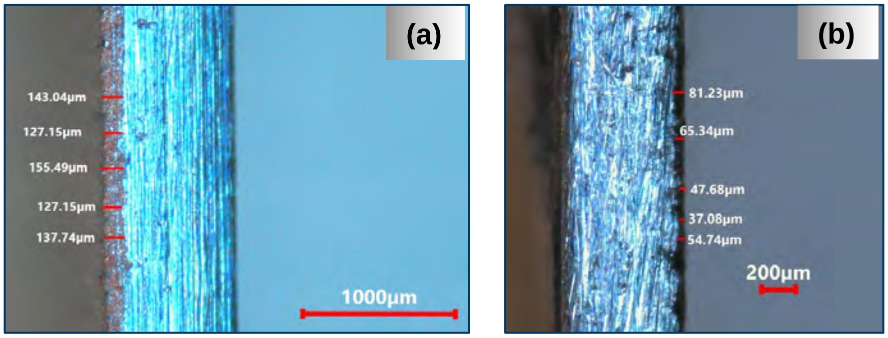


Figure 3.4 Coating thickness measurements using a microscope camera (a) S-AP (b) S-LS

Stainless steel-Armour Seal (SS-AS)

Table 3.29 Coating thickness measurements for Armour Seal® applied on stainless steel

Coating Thickness for Steel-Aquapon (SS-AS)						
No. of Readings	1	2	3	4	5	Average
Thickness (mm)	0.13	0.13	0.14	0.13	0.13	0.13
Thickness (inch)	0.01	0.01	0.01	0.01	0.01	0.01
Thickness (microns)	130.00	130.00	140.00	130.00	130.00	132.00
Thickness (mils)	5.12	5.12	5.51	5.12	5.12	5.20

Stainless steel-Lubra Seal (SS-LS)

Table 3.30 Coating thickness measurements for Lubra Seal® applied on stainless steel

Coating Thickness for Steel-Lubra Seal (SS-LS)						
No. of Readings	1	2	3	4	5	Average
Thickness (mm)	0.05	0.04	0.04	0.03	0.04	0.04
Thickness (inch)	0.00	0.00	0.00	0.00	0.00	0.00
Thickness (microns)	52.98	42.38	37.08	33.55	37.08	40.61
Thickness (mils)	2.09	1.67	1.46	1.32	1.46	1.60

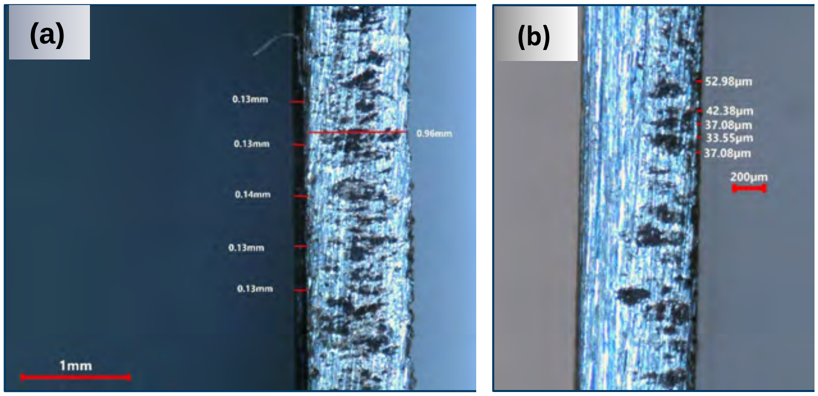


Figure 3.5 Coating thickness measurements using a microscope camera (a) SS-AS (b) SS-LS

Aluminum-Armour Seal (Al-AS)

Table 3.31 Coating thickness measurements for Armour Seal® applied on aluminum

Coating Thickness for Aluminum-Armour Seal (Al-AS)						
No. of Readings	1	2	3	4	5	Average
Thickness (mm)	0.22	0.23	0.19	0.22	0.18	0.21
Thickness (inch)	0.01	0.01	0.01	0.01	0.01	0.01
Thickness (microns)	220.00	230.00	190.00	220.00	180.00	208.00
Thickness (mils)	8.66	9.06	7.48	8.66	7.09	8.19

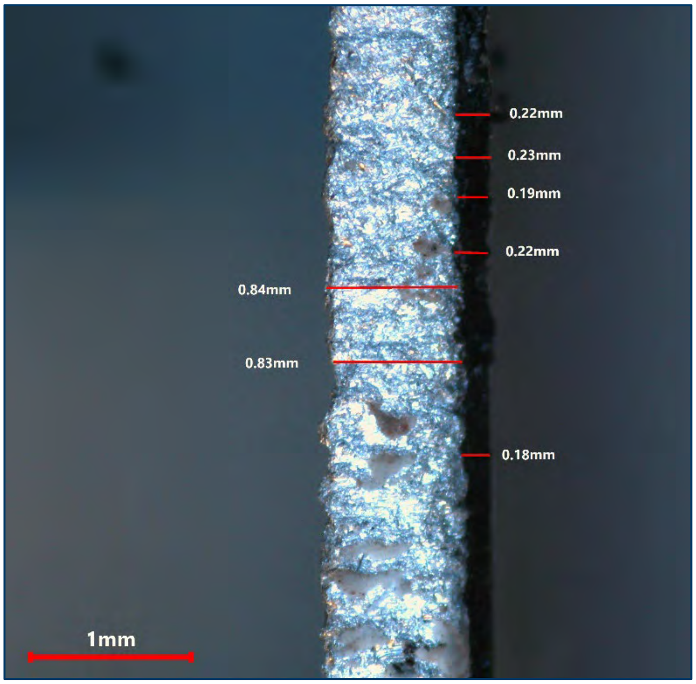


Figure 3.6 Coating thickness measurements using a microscope camera for Al-AS

NOTE:

There were no coating thickness requirements provided in the TDS of Lubra Seal. Lubra Seal is a very thing product, compared to Armour Seal. For Armour Seal the range was very wide from 15 mils to 60 mils, depending on where it is applied and if the metal surface is rusted or not and if sound dampening properties are needed or not. For Aquapon, the recommended dry film thickness was 3 to 5 mils (75 – 125 microns), depending on the metal-coating system.

Chapter 4: Discussion and Conclusions

In this chapter, we discuss the results from aforementioned tests of coatings, and evaluate the performance of each coating.

4.1 EIS test results – Discussion

4.1.1 Performance of coatings in NaCl-CaCl$_2$ salt blend

Based on the parameters obtained, specifically those related to coatings, after modeling the raw data from EIS testing, the relative performance of the selected coating-metal combinations in the NaCl-CaCl$_2$ blend salt brine is illustrated in Figure 4.1, in terms of pore resistance (R_{po}).

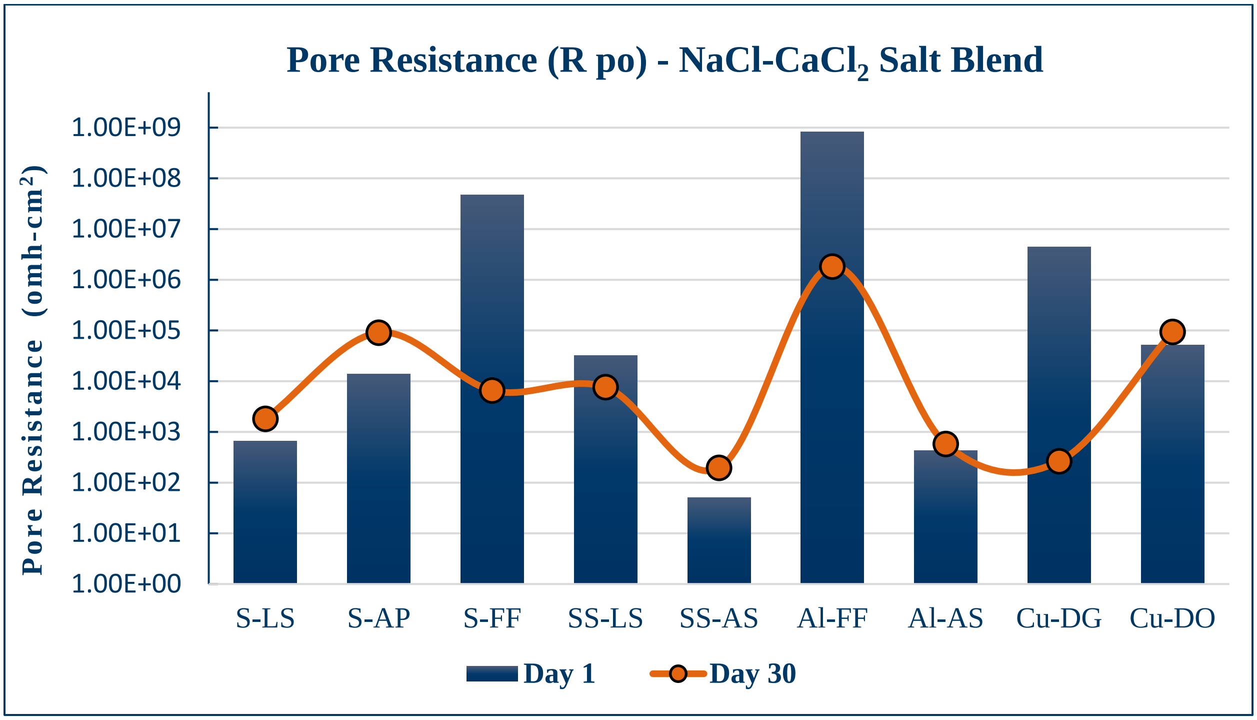


Figure 4.1 Pore resistances for day 1 and day 30 for all combinations tested in NaCl-CaCl$_2$ blend salt brine

In Figure 4.1, dark blue blocks indicate the R_{po} for day 1 and the orange line with markers on it represents the R_{po} on day 30 for all the metal-coating combinations. It is evident that Fluid Film showed a very high R_{po} (coating or pore resistance) on day 1, but it also significantly dropped for steel and even for Al-FF. For Steel S-FF-30 D, R_{po} dropped lower than the R_{po} of S-AP-30D. Also, for steel protected with galvanizing (S-AP) the resistance increased, due to the self-healing characteristic of steel-zinc bond in water. Therefore, for steel, Aquapon (zinc-rich epoxy primer) proved to be the best protection for long-term use. Note that the increase in R_{po} for S-LS indicates metallic corrosion products block the pores of the coating causing an increase in R_{po}, which was evident from a drop in both the charge resistance and R_p. For stainless steel, Lubra Seal is better than Armour Seal in NaCl-CaCl$_2$ blend salt brine. Fluid Film is an excellent protection for Aluminum compared to Armour Seal, and dielectric grease is better for copper than Deox IT for short-term use, but Deox IT would be a better choice for long-term use. This is because the R $_{po}$ for the Cu-DG combo was higher on day 1 than Cu-DO, but after 30 days R_{po} for Cu-DO ended up higher.

4.1.2 Performance of coatings in $MgCl_2$-Beet blend

For the same coatings when tested in the MgCl2-Beet blend, Figure 4.2 shows their performance based on the pore resistance or the coating resistance values from day 1 and day 30.

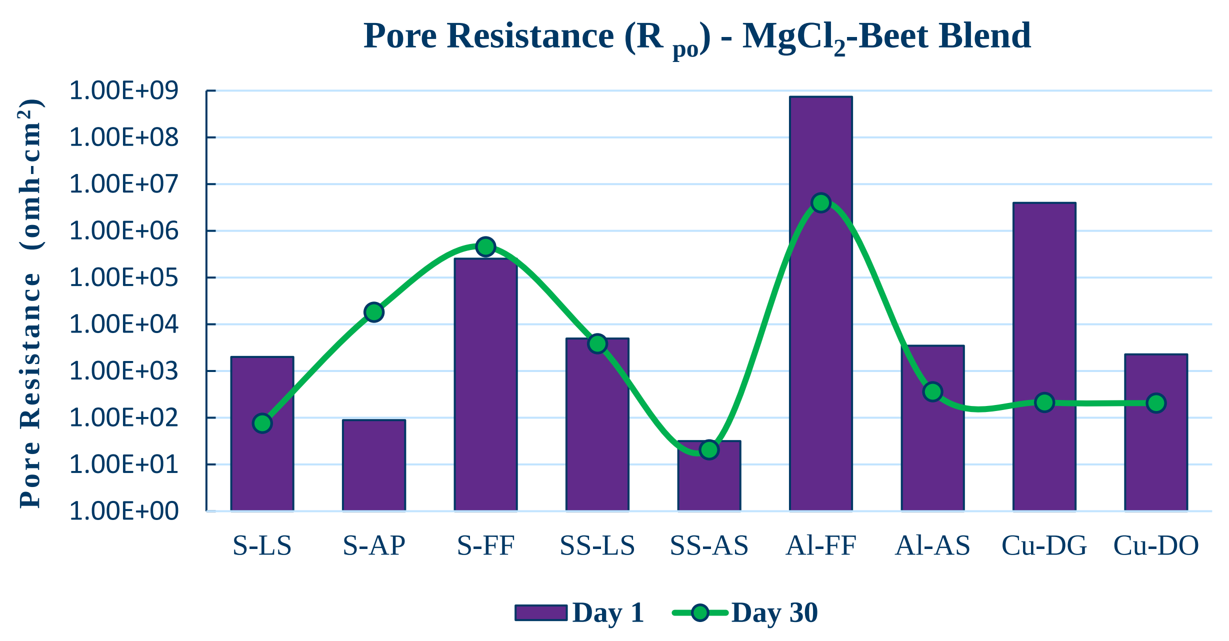


Figure 4.2 Pore resistances from day 1 and day 30 for all combinations tested in $MgCl_2$-Beet blend

Figure 4.2 shows pore resistance (coating resistance) in magenta blocks and the green line with markers on it represents the R po for the day 30 for all metal-coating combinations tested in the $MgCl_2$-Beet blend. Once again, Fluid Film stands out for its resistance against corrosive aqueous media. With added corrosion inhibitor it could also be used for long run for steels in the presence of $MgCl_2$ salt. The second highest R_{po} on day 30 for all material-coating combinations is shown by S-AP, followed by SS-LS. Note that for this blend with added corrosion inhibitor, S-LS showed normal behavior and the R po decreased after 30 days of testing, perhaps due to less corrosion occurring at the metal interface and not blocking the pores. S-AP showed similar trends on both blends due to the self-healing characteristic of Zn-rich primer on steel in aqueous media. For non-ferrous metals/alloys, Fluid Film outperformed Armour Seal in protecting aluminum, whereas dielectric grease showed slightly better performance than Deox IT for protecting copper due to higher R po in the early stages of the test.

4.1.3 Coating rankings – Based on EIS results

Table 4.1 shows the ranks of the tested coatings based on their overall performance in both blends, where 1 indicates the best performer. Fluid Film and zinc-rich epoxy primer (galvanizing) are the top two products tested for EIS in blend salt brines with and without corrosion inhibitors.

Table 4.1 Coating rankings based on EIS test results for both blend salt brines

For NaCl-$CaCl_2$ Salt Blend			
Steel			
Rank	1	2	3
Combinations	S-AP	S-FF	S-LS
Stainless Steel			
Rank	1	2	
Combinations	SS-LS	SS-AS	
Aluminum			
Rank	1	2	
Combinations	Al-FF	Al-AS	
Copper			
Rank	1	2	
Combinations	Cu-DO	Cu-DG	
For $MgCl_2$-Beet Blend			
Steel			
Rank	1	2	3
Combinations	S-FF	S-AP	S-LS
Stainless Steel			
Rank	1	2	
Combinations	SS-LS	SS-AS	
Aluminum			
Rank	1	2	
Combinations	Al-FF	Al-AS	
Copper			
Rank	1	2	
Combinations	Cu-DG	Cu-DO	

4.2 Salt spray test (SAE J2334) – Discussion

For the salt spray test results, it is not possible to rank each coating individually. However, based on the photos taken at regular intervals, it was evident that three combinations failed the salt spray test – S-LS, Cu-DG, and Cu-DO. Two of these did not have any scribing done because the coatings were soft (dielectric grease and Deox-IT). For steel, Lubra Seal failed to protect it in an accelerated corrosion attack, projected for 5 years of real-life salt-laden conditions. However, Fluid Film remained successful in steel protection and showed astonishing results (evident from photos in Figures C 8 to C 24, Appendix C) after the completion of 60 cycles of the SAE J2334 test. There was no rust forming on steel coupons lubricated with Fluid Film after 60 days of testing. Those coupons are still not rusting (drying at room temperature and relative humidity of 35±10%), which is remarkable because steel picks up flash rust very quickly. Similarly, aluminum lubricated with Fluid Film has not shown any signs of corrosion still to this day and passed the 60 cycles for the SAE J2334 test.

Steel coated with Aquapon (zinc-rich epoxy primer) also passed the salt spray test, though some salt deposits were prominent on its surface. Armour Seal also passed the test for both stainless steel and aluminum. Therefore, performance against corrosion is not distinguishable between S-FF, S-AP, Al-AS, Al-FF, SS-AS, and SS-LS, because they all passed the test with no signs of corrosion on them.

Corrosion rates for S-LS, Cu-DO, and Cu-DG are already presented in the results section. Table 4.2 shows metal-coating combinations tested for salt spray that passed and failed the test.

Table 4.2 Metal-combinations that failed and passed the salt spray test

SAE J2334 – Cyclic Salt Spray Test					
Passed Combinations					
S-AP	S-FF	SS-LS	SS-AS	Al-FF	Al-AS
Failed Combinations					
S-LS		Cu-DO		Cu-DG	

4.3 Adhesion test results – Discussion

Based on the adhesion test results for hard coatings (Lubra Seal, Armour Seal, and Aquapon), Aquapon showed the highest adhesion strength results. No concrete results for Lubra Seal were obtained due to glue failure. The average adhesion strength for Al-AS was 461 psi, for S-AP was 1412 psi, and for SS-AS was 513 psi. Armour Seal showed 11% higher adhesion strength when applied on stainless steel compared to aluminum.

4.4 Hardness test results – Discussion

4.4.1 Pencil hardness test

The pencil hardness test did not yield any concrete results for hard coatings. The sealants tested were both too soft to resist even the softest lead (6B) scratching their surfaces. The softest lead pencil produced gouges on the surfaces of Lubra and Armour seals, which are deeper than a scratch. Because there were no concrete results for both Lubra Seal and Armour Seal coupons, it can be deduced from the pencil hardness test that both sealants are too soft to be regarded as a proper coating on metals. Their application perhaps is not to protect the metal surface from indentations or scratches but to solely protect it from salt-based deicers and to avoid corrosion as long as they remain on the surface of the metal. However, these sealants can easily be scratched or cut and maybe worn out quickly by solid and hard particles of snow and ice or gravel on the road; and are therefore not very suitable for long-term use. They should also be monitored regularly for any scratched areas, where corrosion may occur, especially when applied on steel.

For the Steel-Aquapon combination, the coating turned out to be too hard to be scratched without adding further weight on the testing device, and still only a scratch was produced by the hardest lead. Therefore, the Vickers hardness test was done for it.

4.4.2 Vickers hardness test

Micro Vickers hardness test was done for Steel coated with Aquapon. The average Vickers hardness for S-AP was reported as 161 HV, which is the usual hardness for a galvanized surface. Therefore, the zinc-rich epoxy paint (primer) passed the hardness test as well

4.5 Concluding remarks and comparison chart

4.5.1 Concluding remarks

Lightweight vs heavy products

Coatings when applied to materials can also increase the weight of the material. In the case of steel, zinc-rich epoxy primer (Aquapon®) includes a metal (zinc) to be added to the paint as part of the 3-component paint formulation, for up to 90% by weight. On the other hand, the lubricant (Fluid Film®) is lightweight compared to Aquapon and even Armour Seal. It also successfully passed the salt spray test just as Armour Seal and Aquapon. Lubra Seal, though more lightweight than Armour Seal, failed to protect steel, but was good for stainless steel, perhaps due to the material self-protecting characteristic known as passivity.

Lifespan and costs of applied products

Though Fluid Film may be lightweight and could also protect steel, stainless steel, and aluminum in the long run; it can be washed away easily in heavy rainfall or during the washing of vehicles. However, Aquapon and the sealants may stay on the material even after any heavy rainfall or washing event. Moreover, the abrasion resistance and resistance to scratch and indentation of Aquapon is much higher than any other tested product. This will reduce the cost of re-application, but the cost of the first application for Aquapon® will be higher than any other material tested. A 1-gal kit of Aquapon costs $225, which is 400% higher than Fluid Film in the same quantity. Aquapon® would also require special surface preparation methods, whereas Fluid Film and sealants can be applied over rusted surfaces as well. Since Aquapon® provides galvanizing to steel, it further extends its lifespan due to its self-healing characteristic. In conclusion, for long-term use, galvanizing or similar protection for steel is the best choice. However, lightweight, inexpensive, and versatile Fluid Film® is the overall best product in this research study.

Recommended washing and drying practices

Washing vehicles with the right type of water is a very important aspect of controlling the corrosion of metals. Steel will rust more in tap water (with a higher mineral content) compared to distilled water. Dissolved oxygen in water and its hardness level play an important role in corrosion mitigation of metals/alloys as well. Water may be treated to achieve the right properties that would be less corrosive to materials. It is also important to keep the vehicles dry when they are not in use (after washing). Any puddle or water droplets could aggravate localized corrosion attacks, as discussed in Chapter 2.

4.5.2 Comparison charts for tested products

Table 4.3 Comparison chart for the products (paints/sealants, lubricants) tested, specifically oriented to material preparation and application method

COMPARISON CHART FOR TESTED PRODUCTS (Material Preparation & Product Application)					
PRODUCTS	Cleaning Required	Surface Preparation	Mode of Application	Re-application Window	Suitable Materials
Fluid Film®	No. If a lot of flash rust is present, it would be better to remove it. Can be applied over flash rust to stop its further spreading. Dry surfaces are recommended.	No special NACE requirement for surface preparation.	Airless sprayer for bulk application. On smaller areas, can spray would work too.	SAE J2334 showed if surfaces are not washed thoroughly, it can stay on them for more than a year. Should be applied after every washing but may not be re-applied after rain exposure.	Steels, Stainless-steels, Aluminum alloys
Aquapon ® (zinc-rich epoxy primer)	Yes. It is important to clean all the flash rust before applying it on steel. The surface must be dry.	Yes. Follow the TDS, to make sure proper surface profile is achieved by sandblasting.	Pressure pot with a stirrer to constantly agitate the mixture of epoxy, paint, and zinc dust, while spraying it. A conventional spray gun (e.g., Lynx 100 series L100C) is required.	SAE J2334 and EIS both showed this product if applied correctly can stay for 3 to 5 years, protecting the surface from rust.	Steels

COMPARISON CHART FOR TESTED PRODUCTS
(Material Preparation & Product Application)

PRODUCTS	Cleaning Required	Surface Preparation	Mode of Application	Re-application Window	Suitable Materials
Lubra Seal®	Yes. Follow the TDS for Spreader and Chassis. Surfaces exposed to salt should be washed with a neutralizer, cleaned, and must be dried.	No special surface profile preparation is required. However, for steel, it would be better to clean any excessive flash rust.	RHOMAR's Heavy Duty Public Works Applicator is recommended. Though pistol grip spray gun can be used as well.	Follow the TDS for Spreader and Chassis, which indicates re-application might be required on several different occasions. As per the test results, at least a bi-annual application might be required.	Steel (only for 6 months), and stainless steel. Though might also be useful on Aluminum alloys.
Armour Seal®	Yes. Follow the TDS. Surfaces exposed to salt should be washed with a neutralizer, cleaned, and must be dried.	No special surface profile preparation is required. However, for steel, it would be better to clean any excessive flash rust. Can be applied on rusted surfaces.	Rhomar's Pistol-Grip spray gun. The product also comes in Schutz quart bottles which makes it convenient to apply with a pistol-grip spray gun.	SAE J2334 and EIS accelerated tests showed the product if applied correctly can stay on surfaces for 3 to 5 years.	Aluminum alloys and stainless steel.

COMPARISON CHART FOR TESTED PRODUCTS (Material Preparation & Product Application)

PRODUCTS	Cleaning Required	Surface Preparation	Mode of Application	Re-application Window	Suitable Materials
Di-electric grease	Yes. It is recommended to remove any corrosion products from copper. Follow the TDS. Surfaces must be cleaned from dirt, oil, etc., and dried.	No special surface profile preparation is required. Applying on a clean and dried copper surface will do the job.	For this study, a paintbrush was used which worked well. A clean microfiber cloth may also be used to apply and coat the grease. Follow the TDS.	Regular inspection must be done of areas where the grease was applied. If the terminals or wire connections are exposed again, make sure to clean them and re-apply whenever necessary.	Copper
Deox-IT®	Yes. It is recommended to remove any corrosion products from copper. Follow the TDS. Surfaces must be cleaned from dirt, oil, etc., and dried.	No special surface profile preparation is required. Applying on a clean and dried copper surface will do the job.	For this study, the can packaging came with a nozzle applicator to spray the product on surfaces, which worked well. Follow the TDS for the Gold G-series.	Regular inspection must be done of areas where the grease was applied. If the terminals or wire connections are exposed again, make sure to clean them and re-apply whenever necessary.	Copper and gold.

Table 4.4 Comparison chart for the products (paints/sealants, lubricants) tested, specifically oriented to suitable environments

COMPARISON CHART FOR TESTED PRODUCTS
(Suitable Environments)

PRODUCTS	Feasible Environments	Abrasion Resistance	Resistance to Scratch and Indentation	Adhesion Strength
Fluid Film®	Will resist corrosion in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions. Suitable for temperatures up to 60° C.	In abrasive environments, the protective film might come off easily, but eventually, it would spread out to unprotected areas.	It is a lubricant with negligible hardness. Although a scratch or indentation when made will remove the protective film, the lubricant may spread out again to the affected areas.	Lubricants do not have adhesion strengths, but Fluid Film has good sticking properties, and it did not come off the coupons during the SAE J2334 test.
Aquapon ® (zinc-rich epoxy primer)	Will resist corrosion in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions. Suitable for temperatures up to 60° C.	It has good abrasion resistance and can be used safely in environments where sand, snow, and gravel may impact on surfaces. Regular inspection is always necessary.	The finished product has a very good hardness on steel and therefore can resist scratches and indentation. Even if the scratch is made, its self-healing characteristic would protect steel from rusting.	Of all the tested products, Aquapon had the highest adhesion strength on steel. This is why it can last longer. The minimum re-application window for it would be 3 years.

COMPARISON CHART FOR TESTED PRODUCTS
(Suitable Environments)

PRODUCTS	Feasible Environments	Abrasion Resistance	Resistance to Scratch and Indentation	Adhesion Strength
Lubra Seal®	May resist corrosion in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions, for only up to 6 months. May not be suitable for temperatures above 30° C.	Sealants are generally not good against abrasives and are not suitable in such environments for longer runs. Regular inspection is a must when it is used in abrasive conditions.	Lubra Seal had negligible hardness and is therefore not suitable in environments where it can be scratched and receive indentation.	The adhesion strength on steel and stainless steel is the bare minimum for this sealant. It will come off easily and should be regularly inspected and re-applied whenever necessary.
Armour Seal®	Will resist corrosion in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions, for up to at least 3 years. Suitable for temperatures up to 60° C.	Sealants are generally not good against abrasives and are not suitable in such environments for longer runs. Regular inspection is a must when it is used in abrasive conditions.	Armour Seal has negligible hardness and is therefore not suitable in environments where it can be scratched and receive indentation.	The adhesion strength of Armour Seal was poor on both aluminum and stainless. However, it was better for aluminum. It will come off easily and should be regularly inspected and re-applied whenever necessary.

COMPARISON CHART FOR TESTED PRODUCTS (Suitable Environments)

PRODUCTS	Feasible Environments	Abrasion Resistance	Resistance to Scratch and Indentation	Adhesion Strength
Di-electric grease	May resist corrosion and oxidation in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions, for only up to 6 months. May not be suitable for temperatures above 30° C.	Di-electric grease is not supposed to have abrasion resistance. It may, however, spread out to the affected area after some time. Regular inspection and timely re-application is necessary.	Di-electric grease is not supposed to have resistance to scratch or indentation. It may, however, spread out to the affected area after some time. Regular inspection and timely re-application is necessary.	Di-electric grease is not supposed to have any adhesion strength; however, it may stay on the applied material for up to 6 months before re-application is needed.
Deox-IT®	May resist corrosion and oxidation in salt-laden wet environments containing chloride, calcium, sodium, and magnesium ions, for only up to 6 months. May not be suitable for temperatures above 30° C.	Deox-IT is not supposed to have abrasion resistance. According to TDS, it will, however, spread out to the affected area after some time, only if it is not dried out already. Regular inspection and timely re-application are necessary.	Deox-IT is not supposed to have resistance to scratch or indentation. It may, however, spread out to the affected area after some time, if it is not dried out already. Regular inspection and timely re-application are necessary.	Deox-IT is not supposed to have any adhesion strength; however, it may stay on the applied material for up to 6 months before re-application is needed.

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Appendix A: EIS Test – Modeled Plots

Modeled Plots – NaCl-$CaCl_2$ Bend

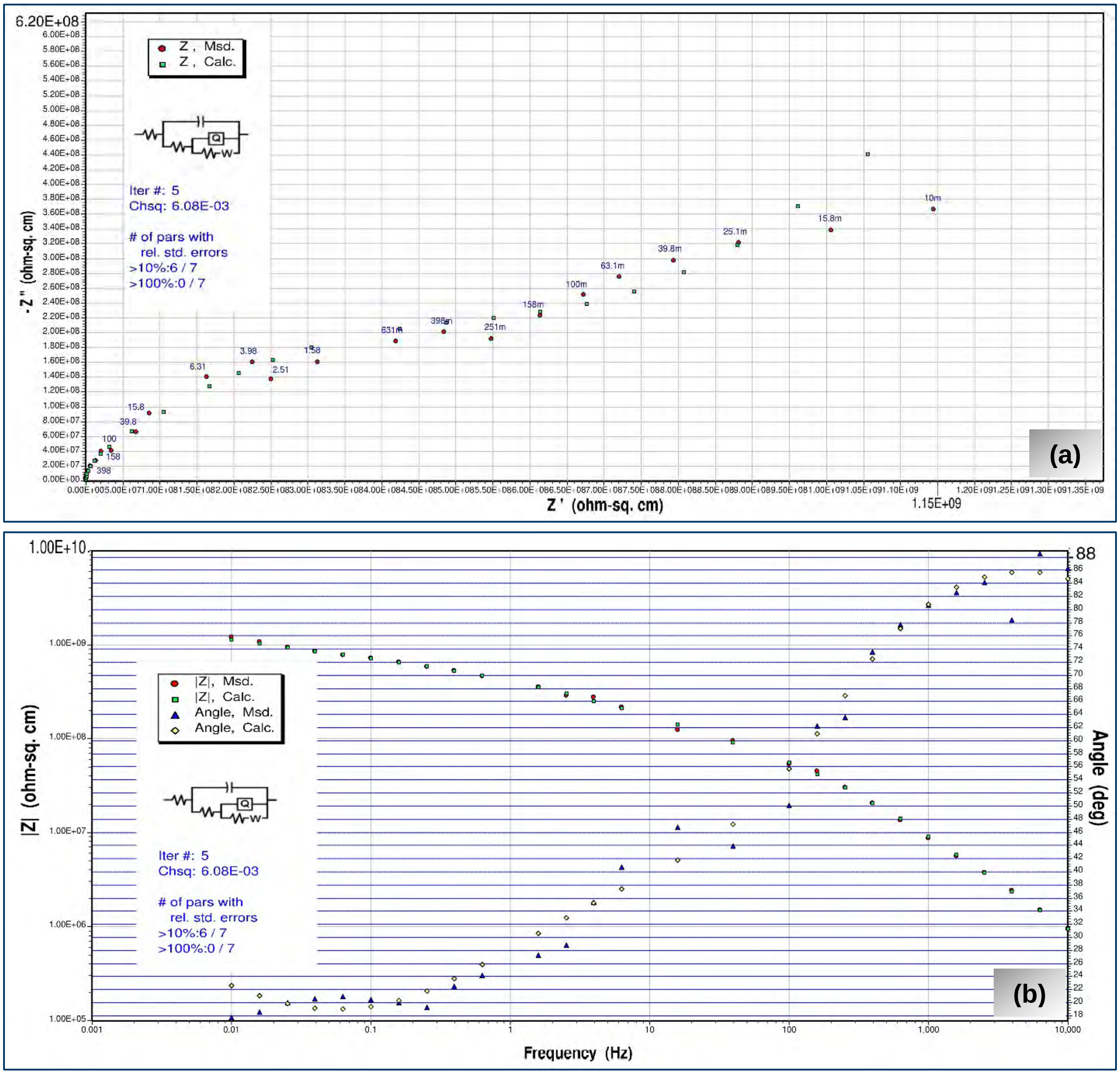


Figure A 1 EIS modeled plots for S-FF-B-day 1, in NaCl-$CaCl_2$ blend (a) Nyquist plot (b) Bode plots

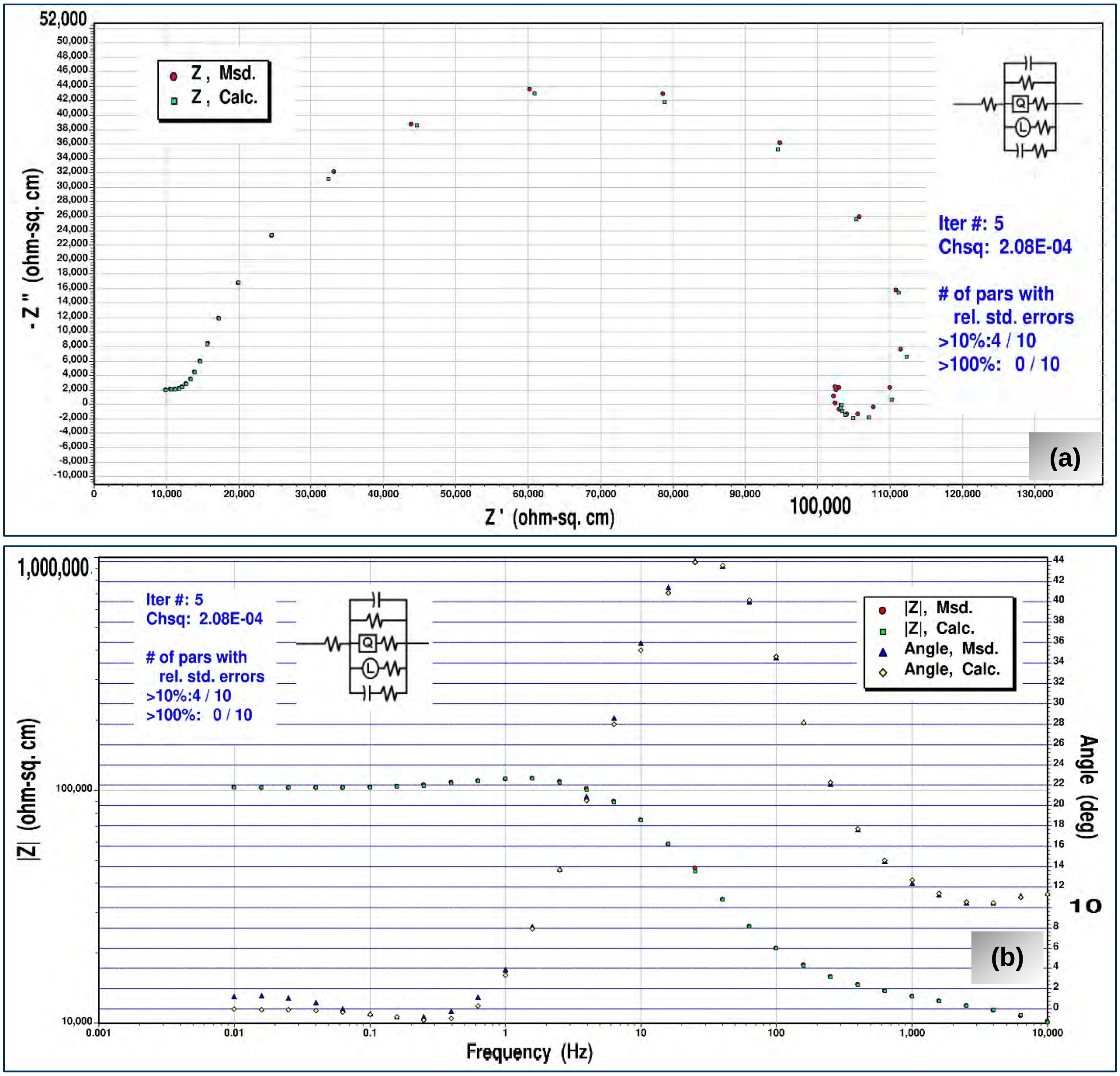


Figure A 2 EIS modeled plots for S-FF-B-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

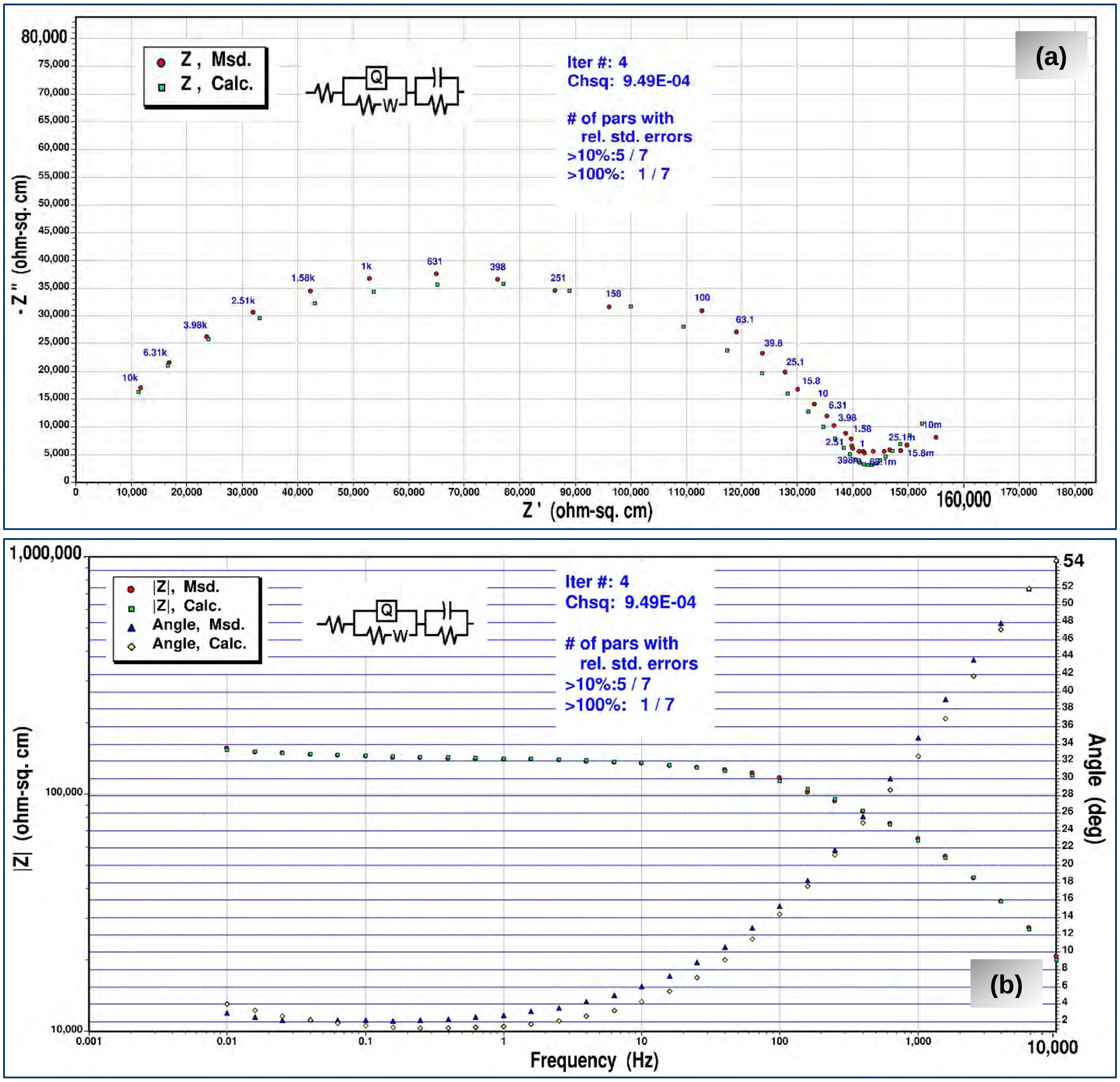


Figure A 3 EIS modeled plots for S-AP-C-day 1, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

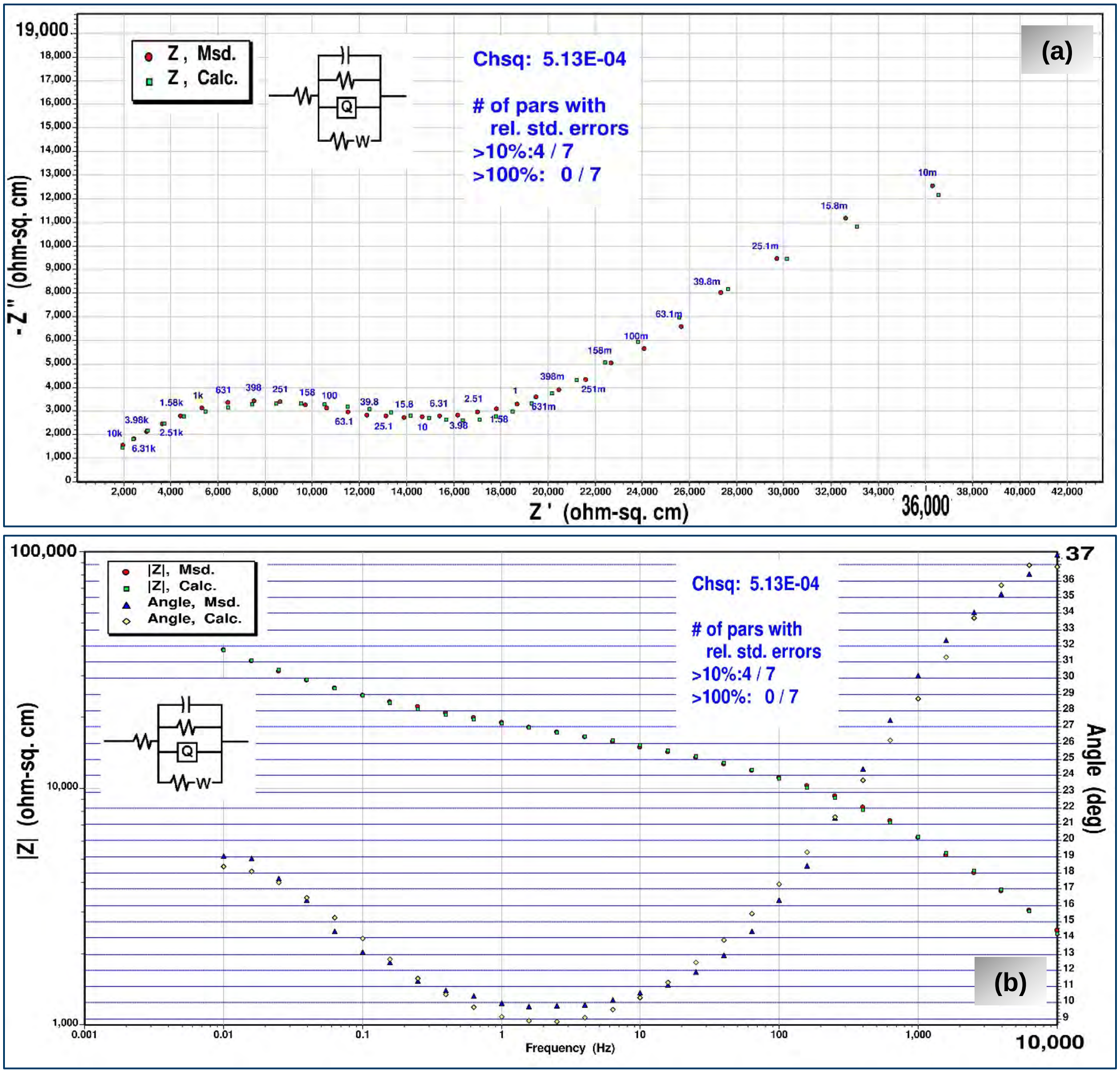


Figure A 4 EIS modeled plots for S-AP-C-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

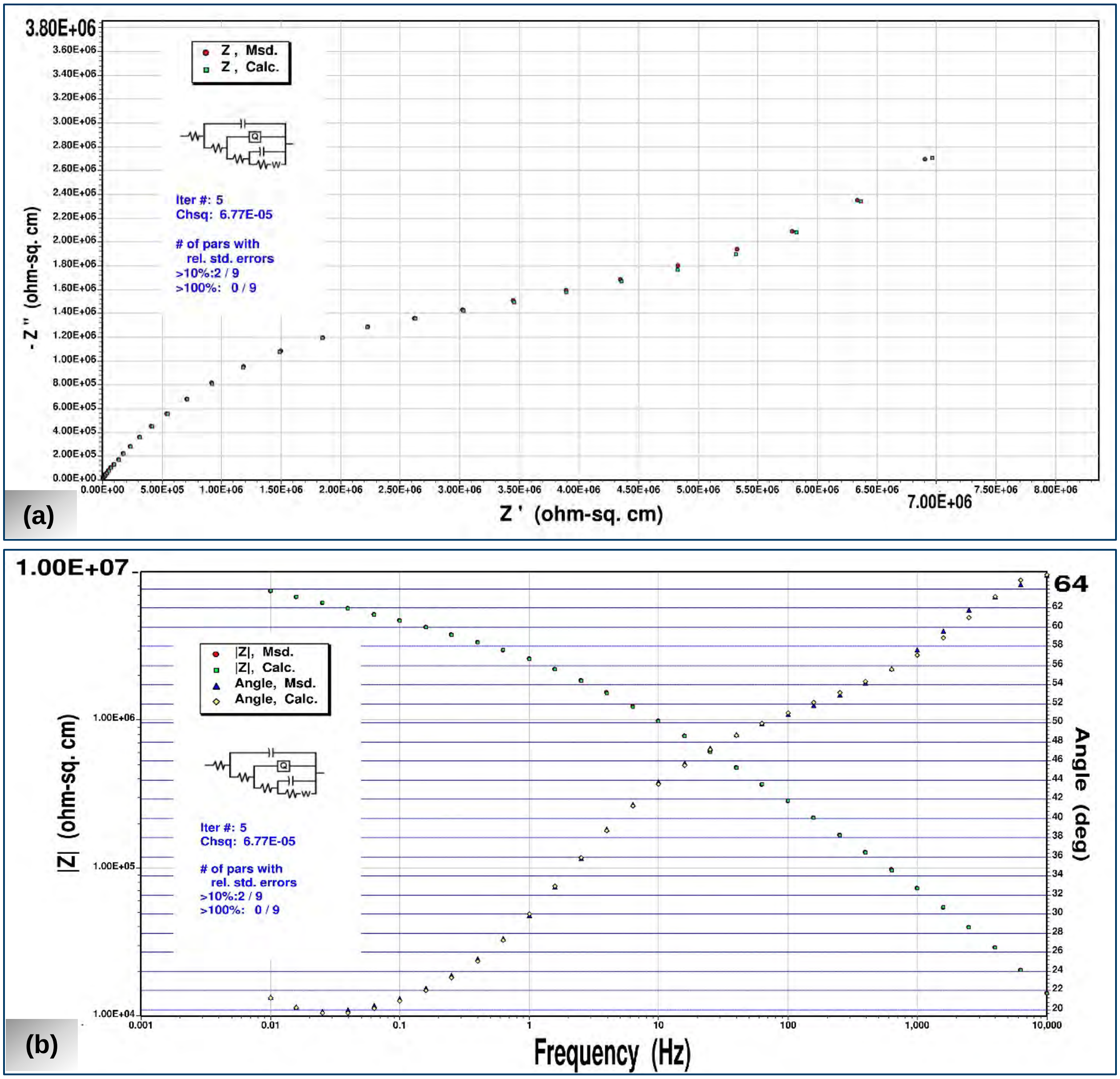


Figure A 5 EIS modeled plots for SS-LS-A-day 1, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

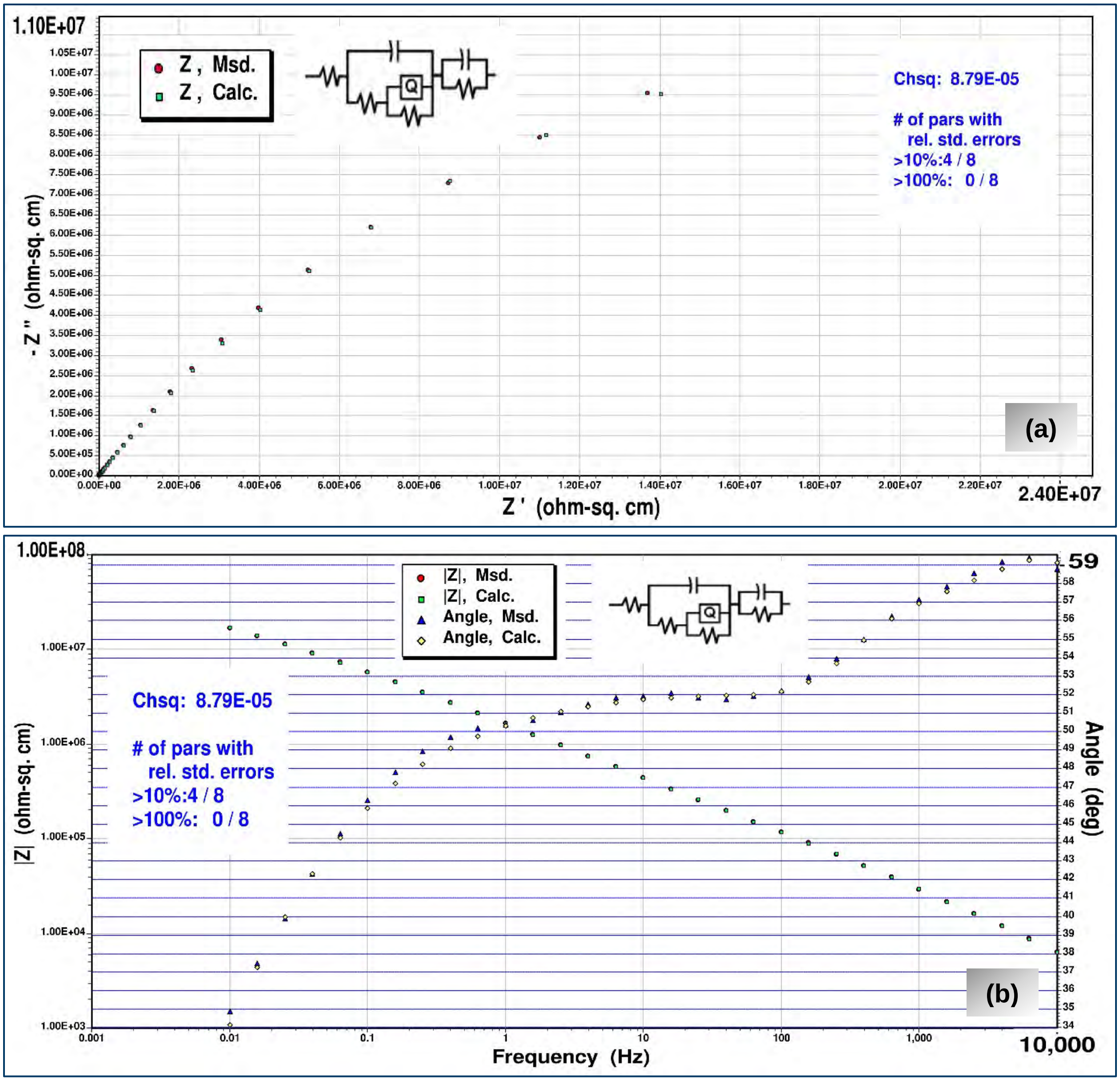


Figure A 6 EIS modeled plots for SS-LS-A-day 30, in NaCl-$CaCl_2$ blend (a) Nyquist plot (b) Bode plots

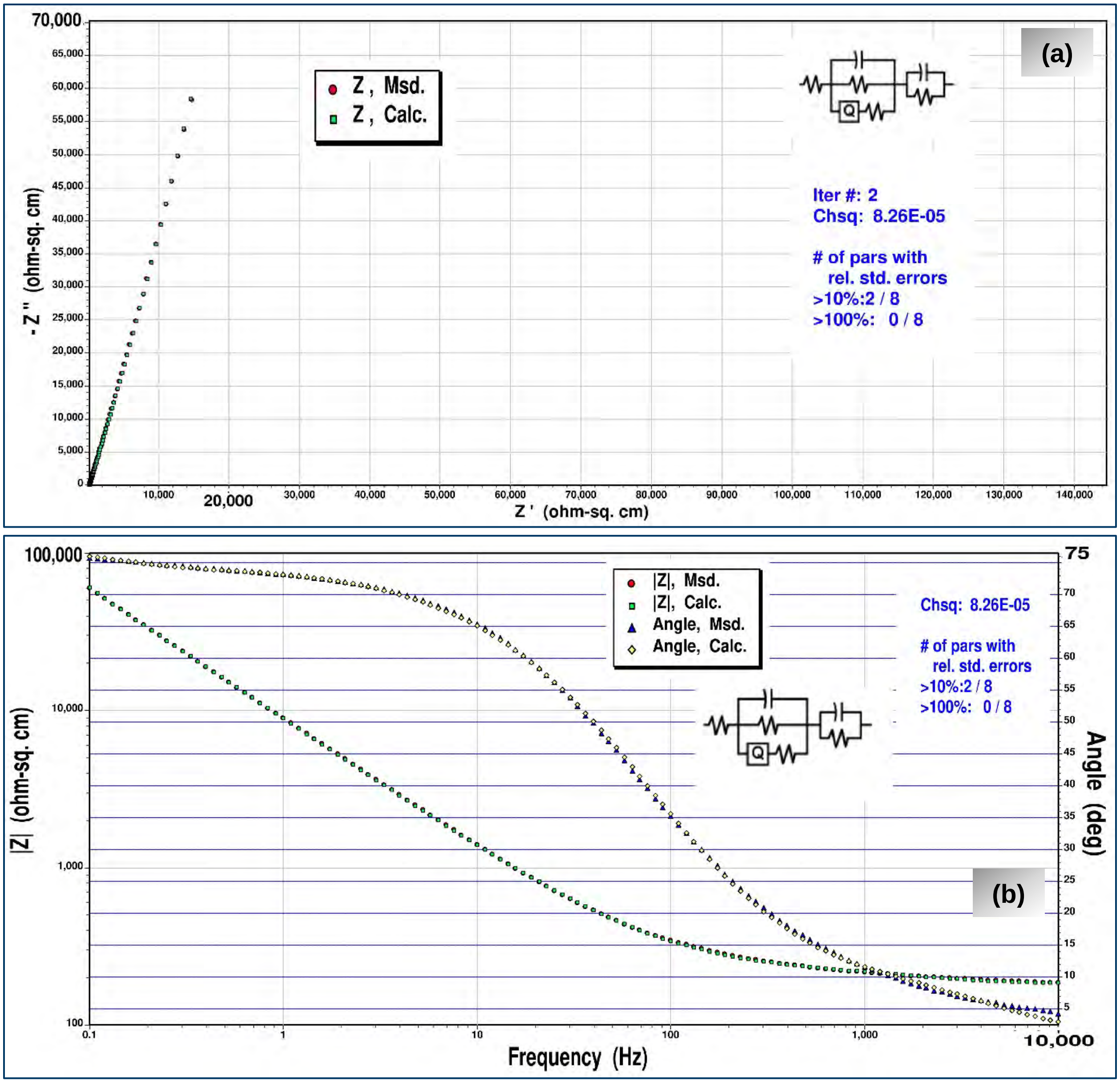


Figure A 7 EIS modeled plots for SS-AS-C-day 2, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

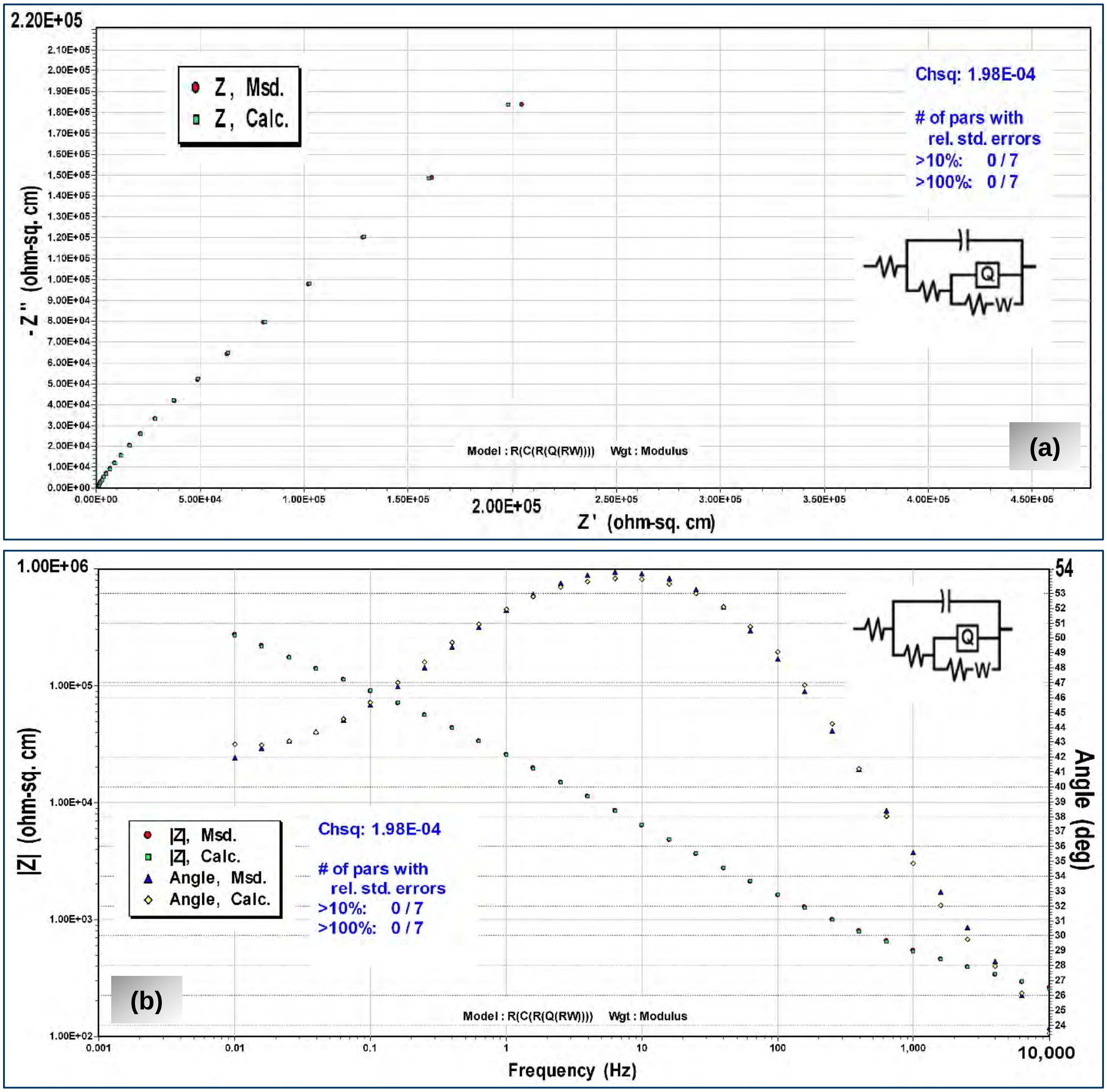


Figure A 8 EIS modeled plots for SS-AS-C-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

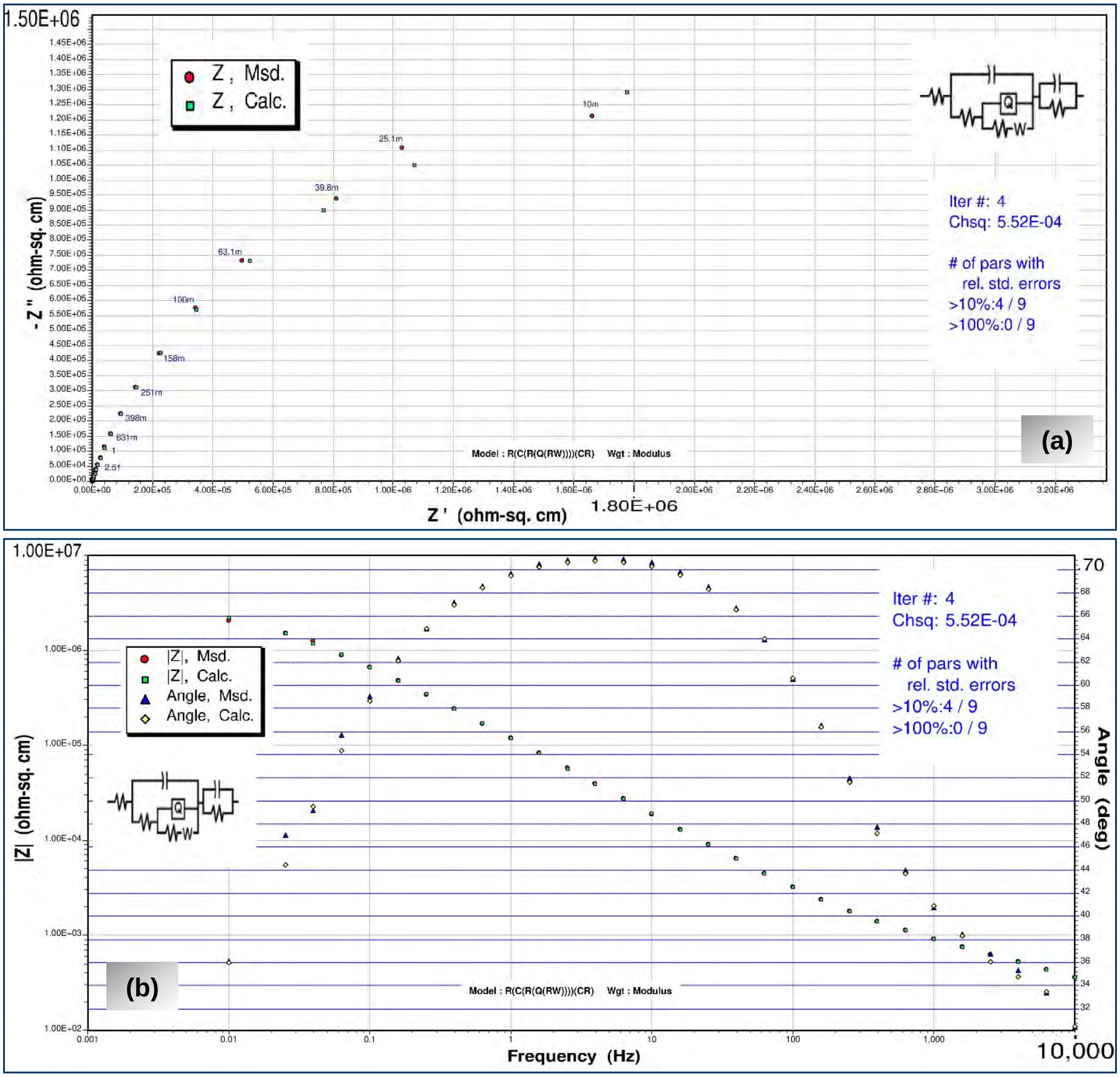


Figure A 9 EIS modeled plots for Al-AS-B-2 hr, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

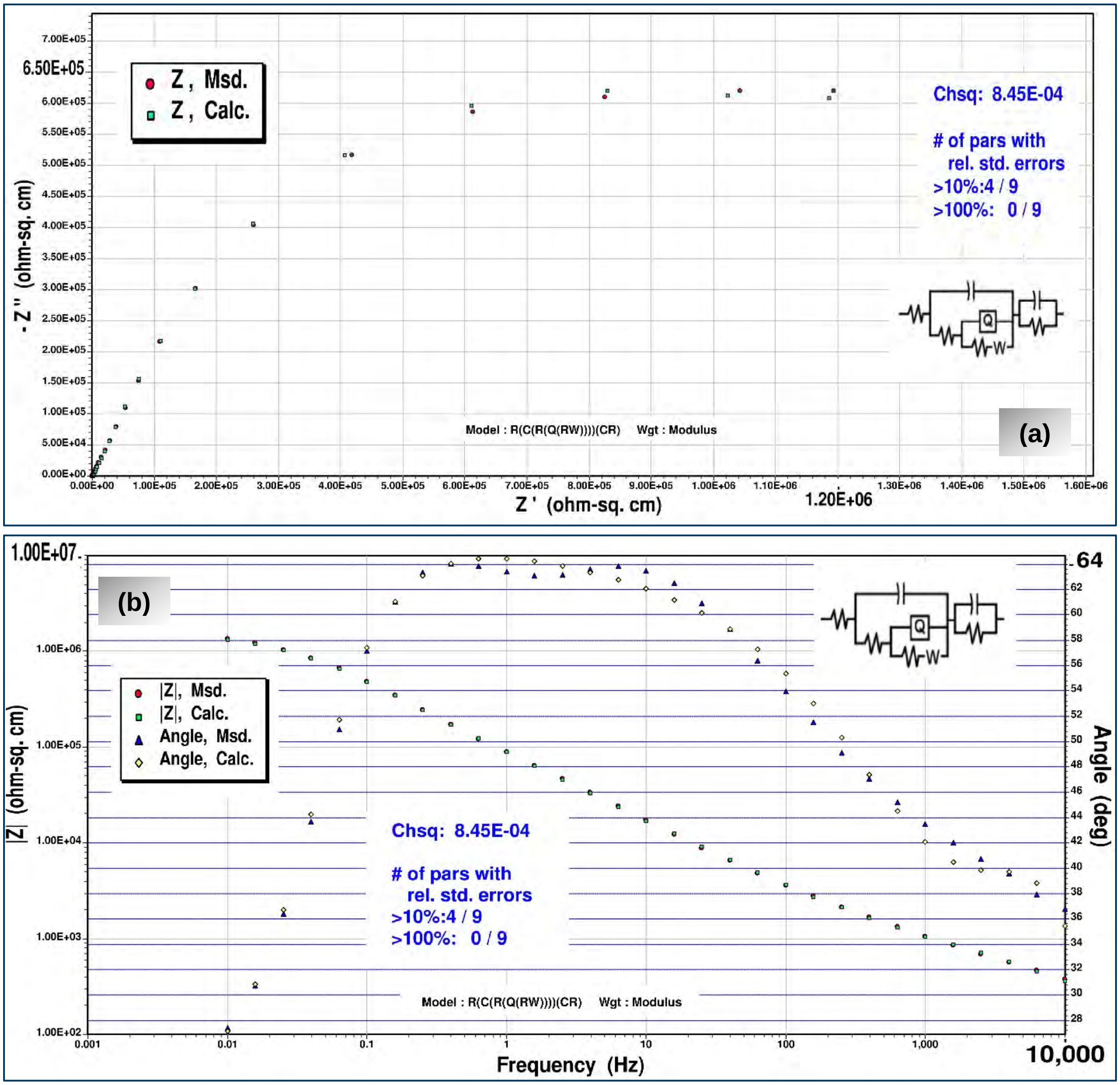


Figure A 10 EIS modeled plots for Al-AS-B-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

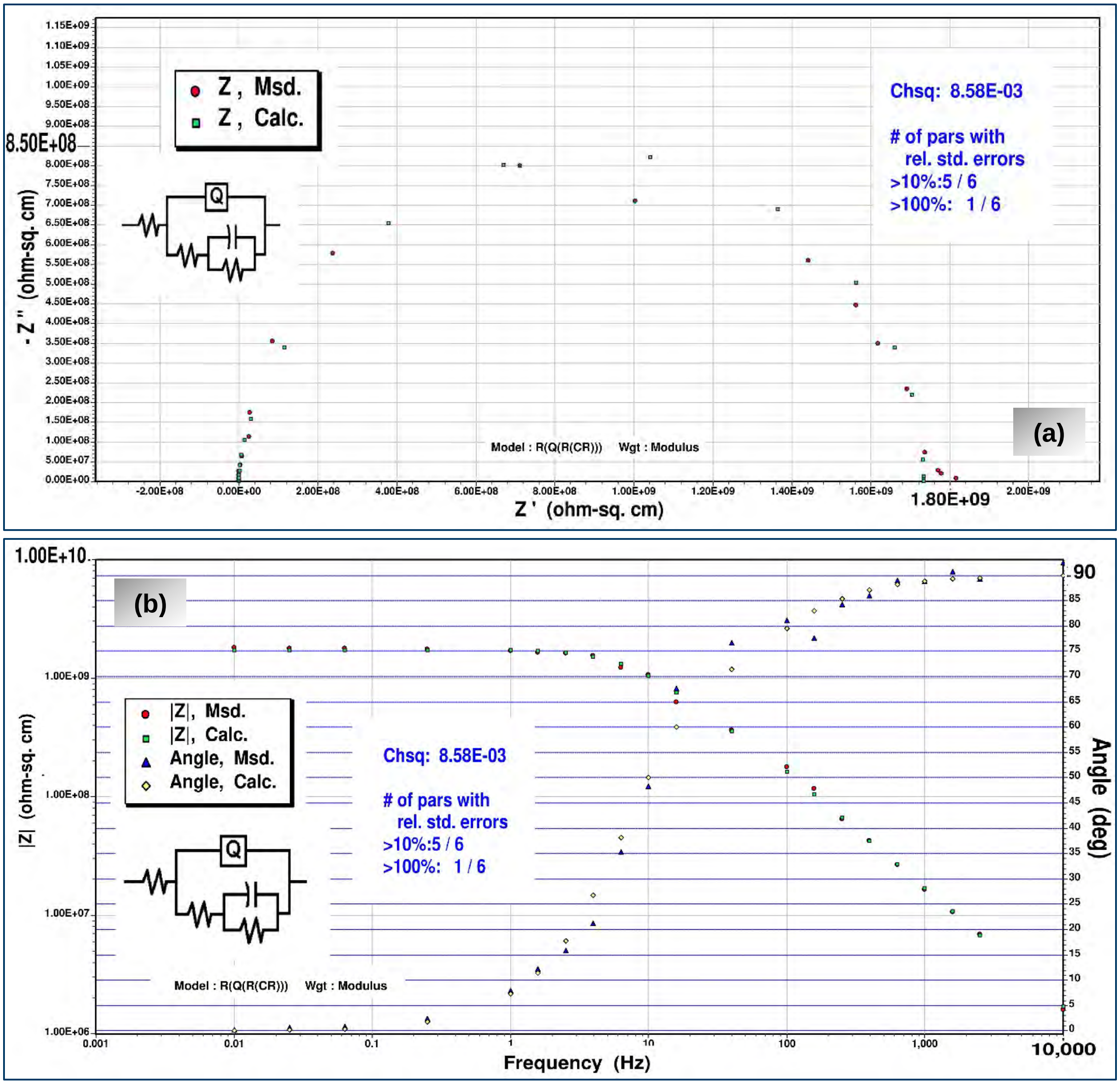


Figure A 11 EIS modeled plots for Al-FF-A-day 5, in NaCl-$CaCl_2$ blend (a) Nyquist plot (b) Bode plots

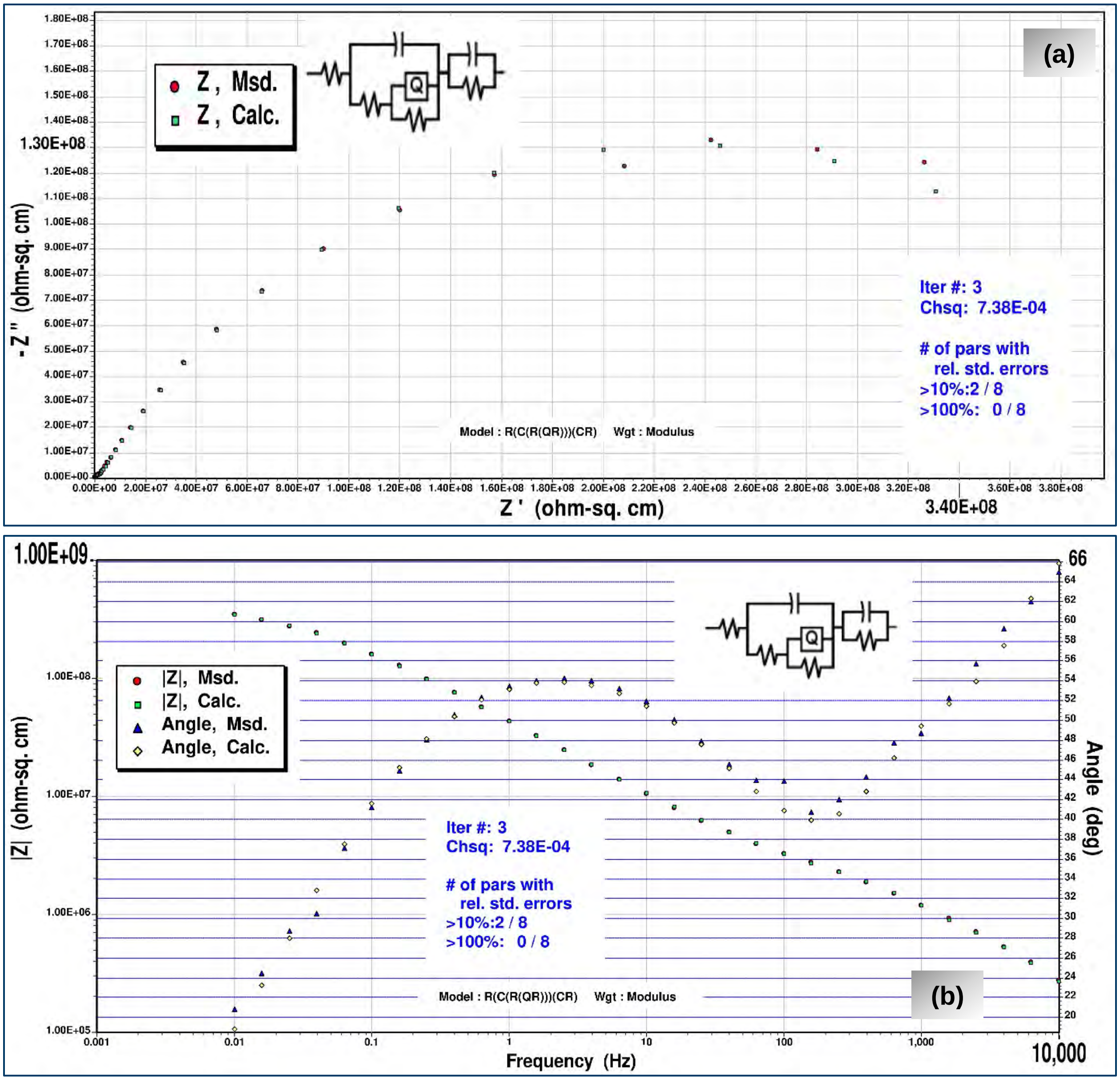


Figure A 12 EIS modeled plots for Al-FF-A-day 30, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

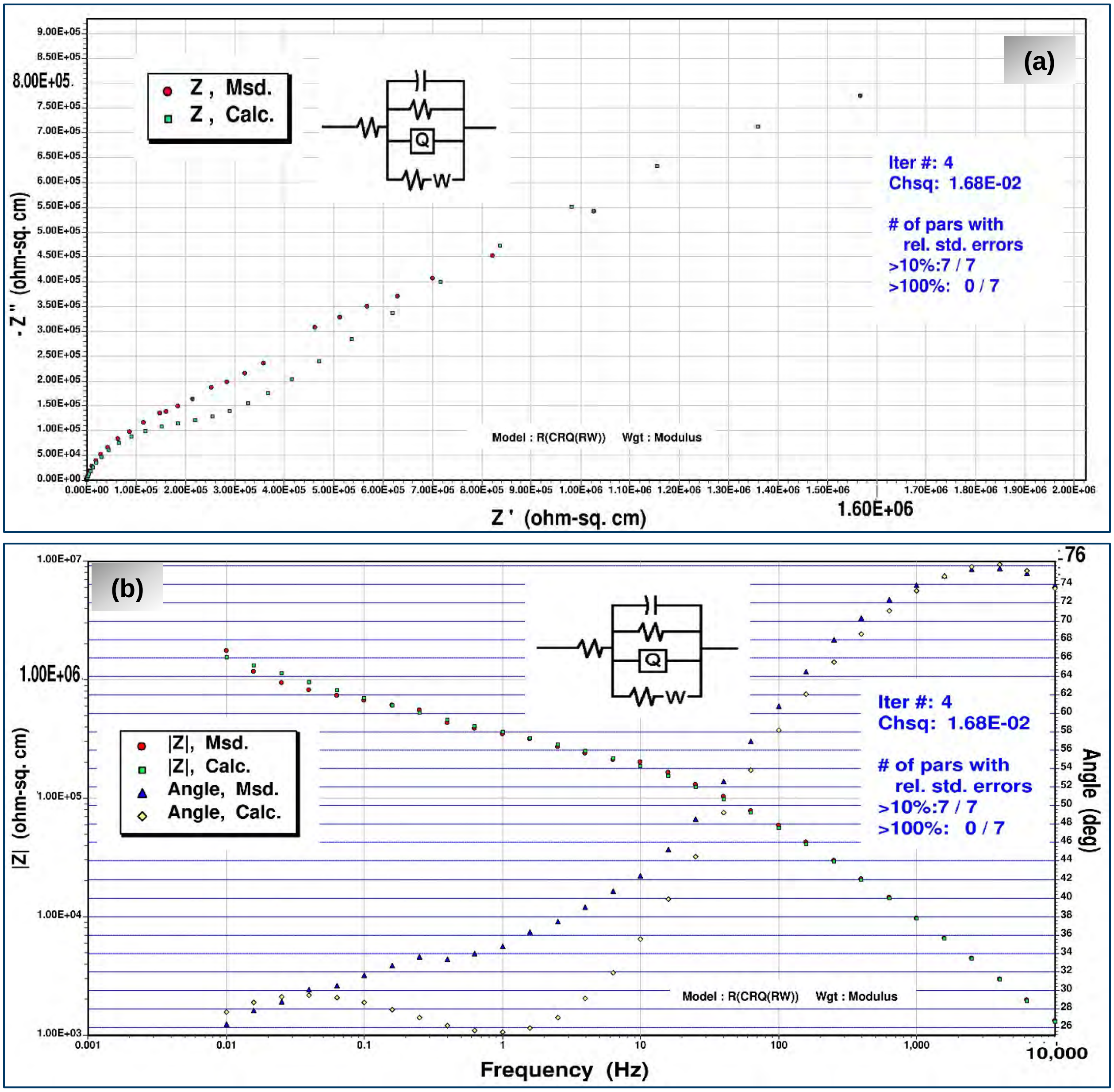


Figure A 13 EIS modeled plots for Cu-DG-B-1Hr, in NaCl-$CaCl_2$ blend (a) Nyquist plot (b) Bode plots

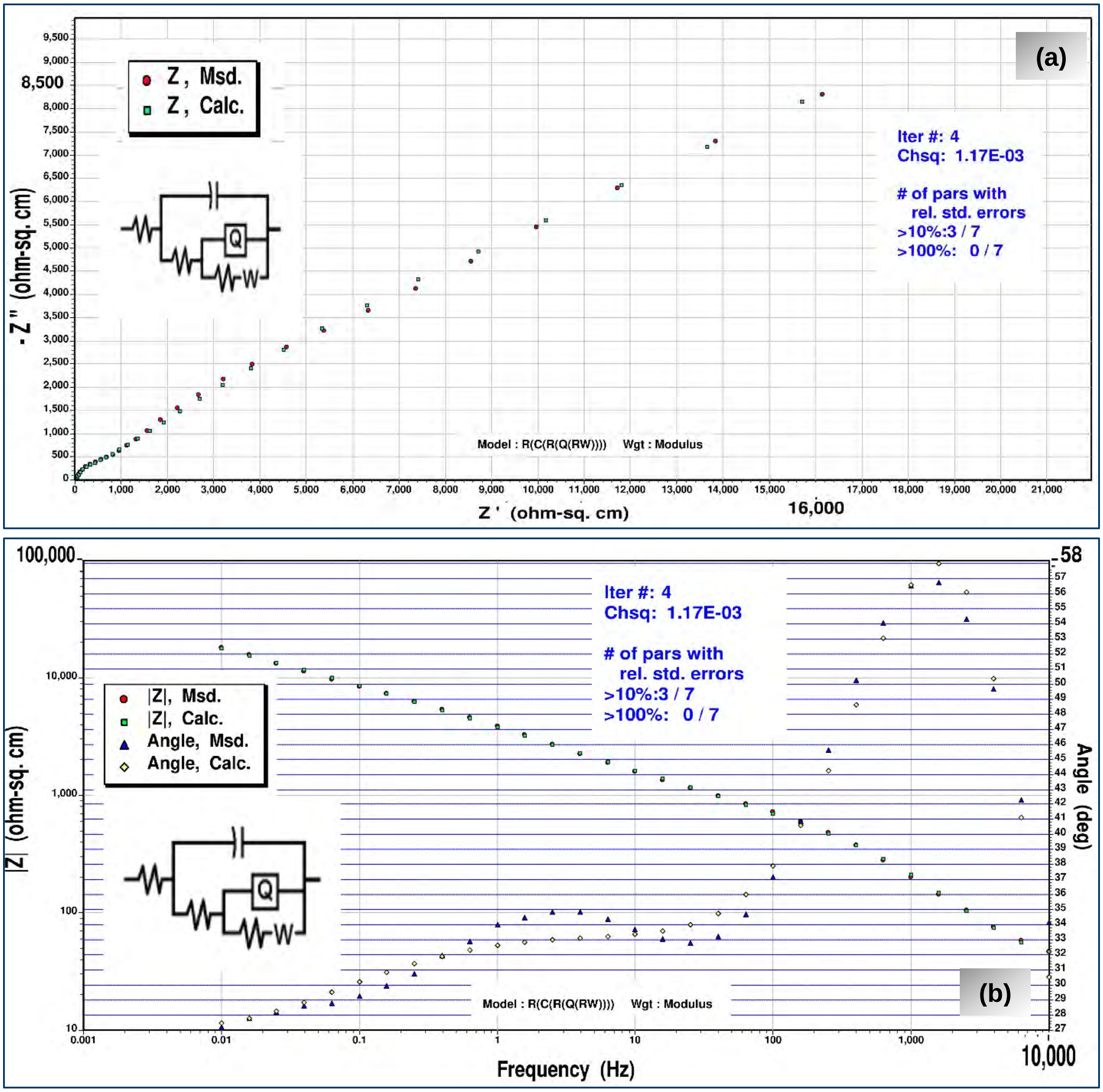


Figure A 14 EIS modeled plots for Cu-DG-B-30D, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

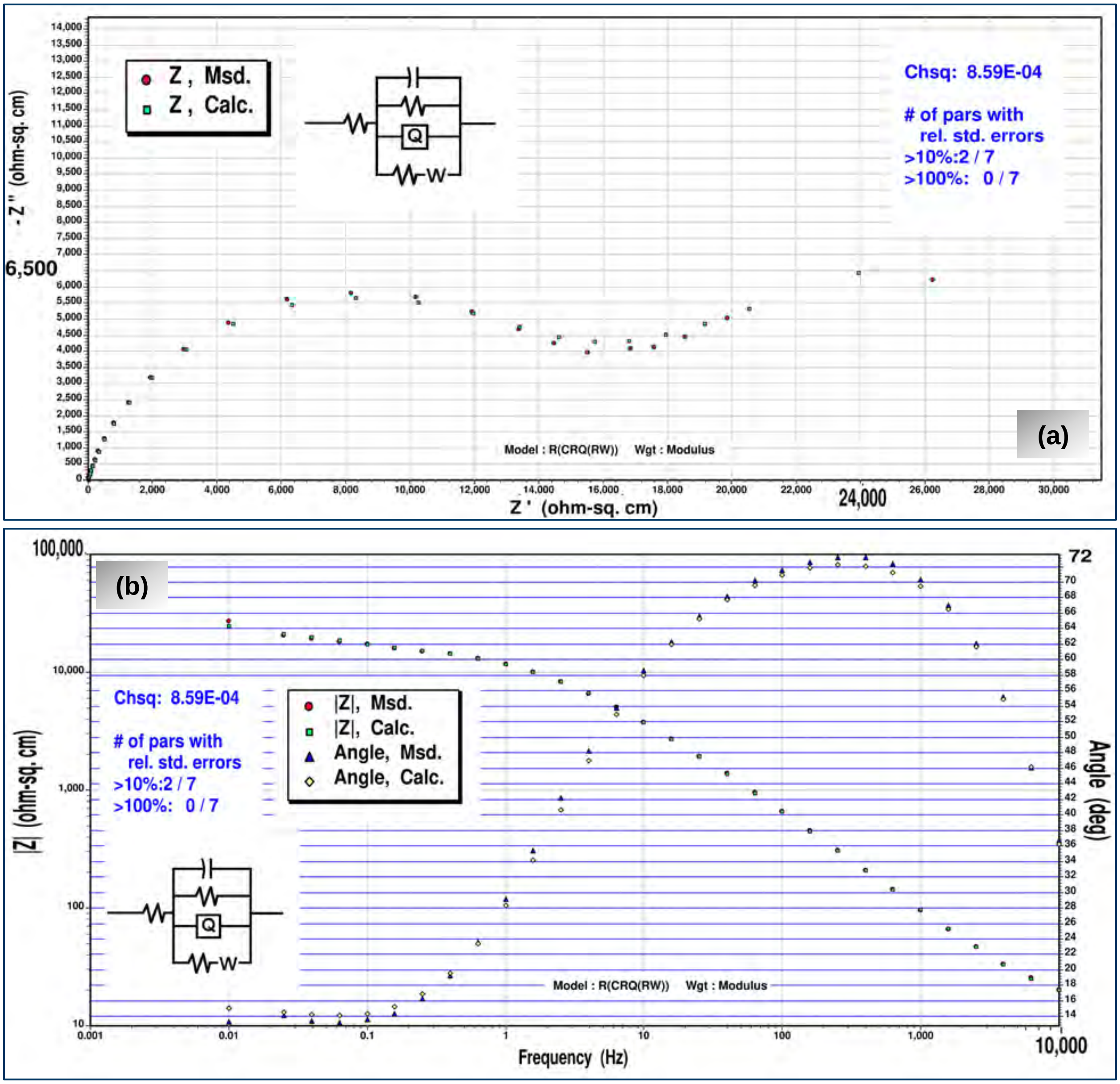


Figure A 15 EIS modeled plots for Cu-DO-A-1 hr, in NaCl-CaCl$_2$ blend (a) Nyquist plot (b) Bode plots

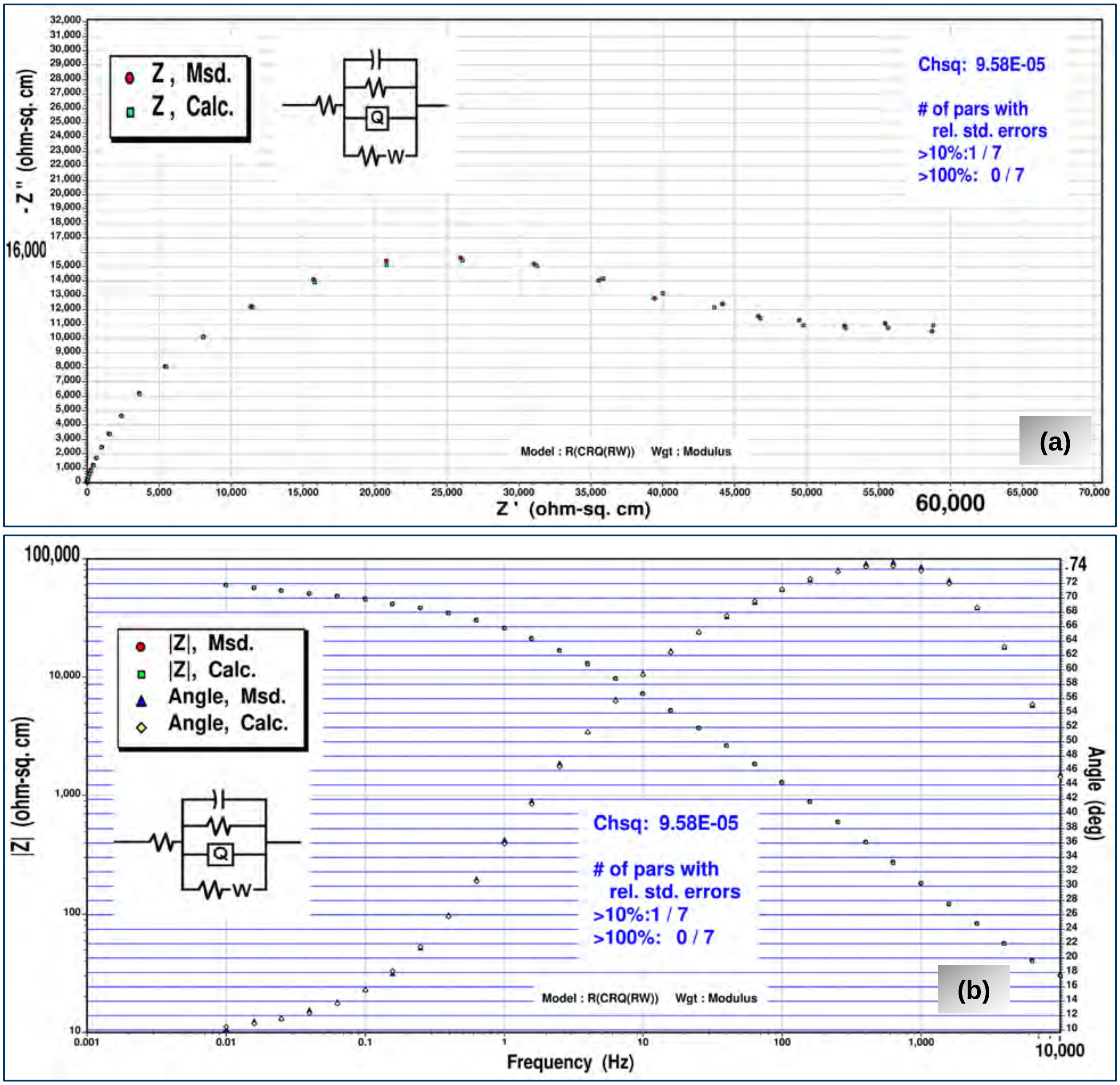


Figure A 16 EIS modeled plots for Cu-DO-A-30 D, in NaCl-$CaCl_2$ blend (a) Nyquist plot (b) Bode plots

Modeled Plots – $MgCl_2$-Beet Blend

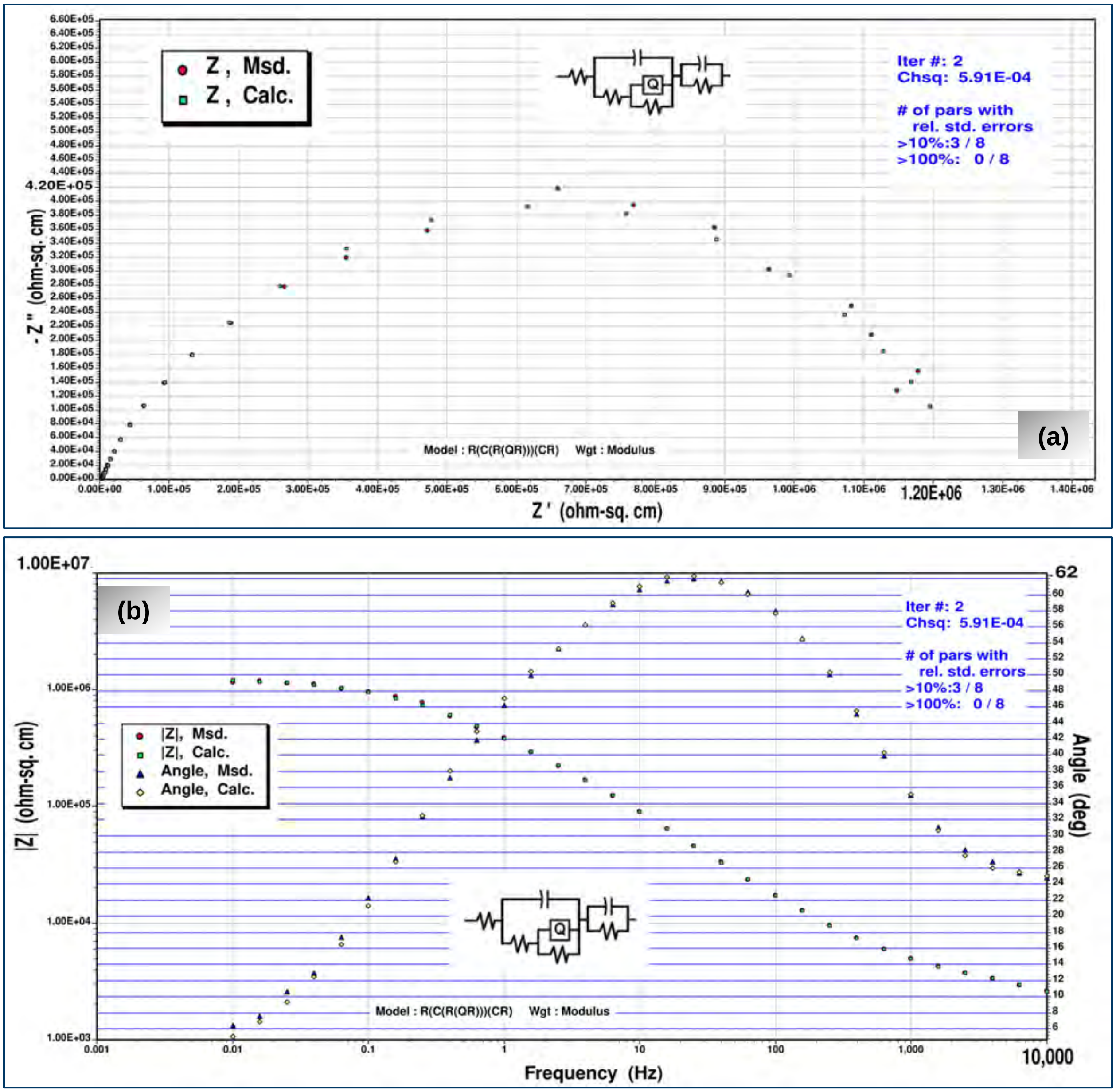


Figure A 17 EIS modeled plots for S-LS-A-day 1, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

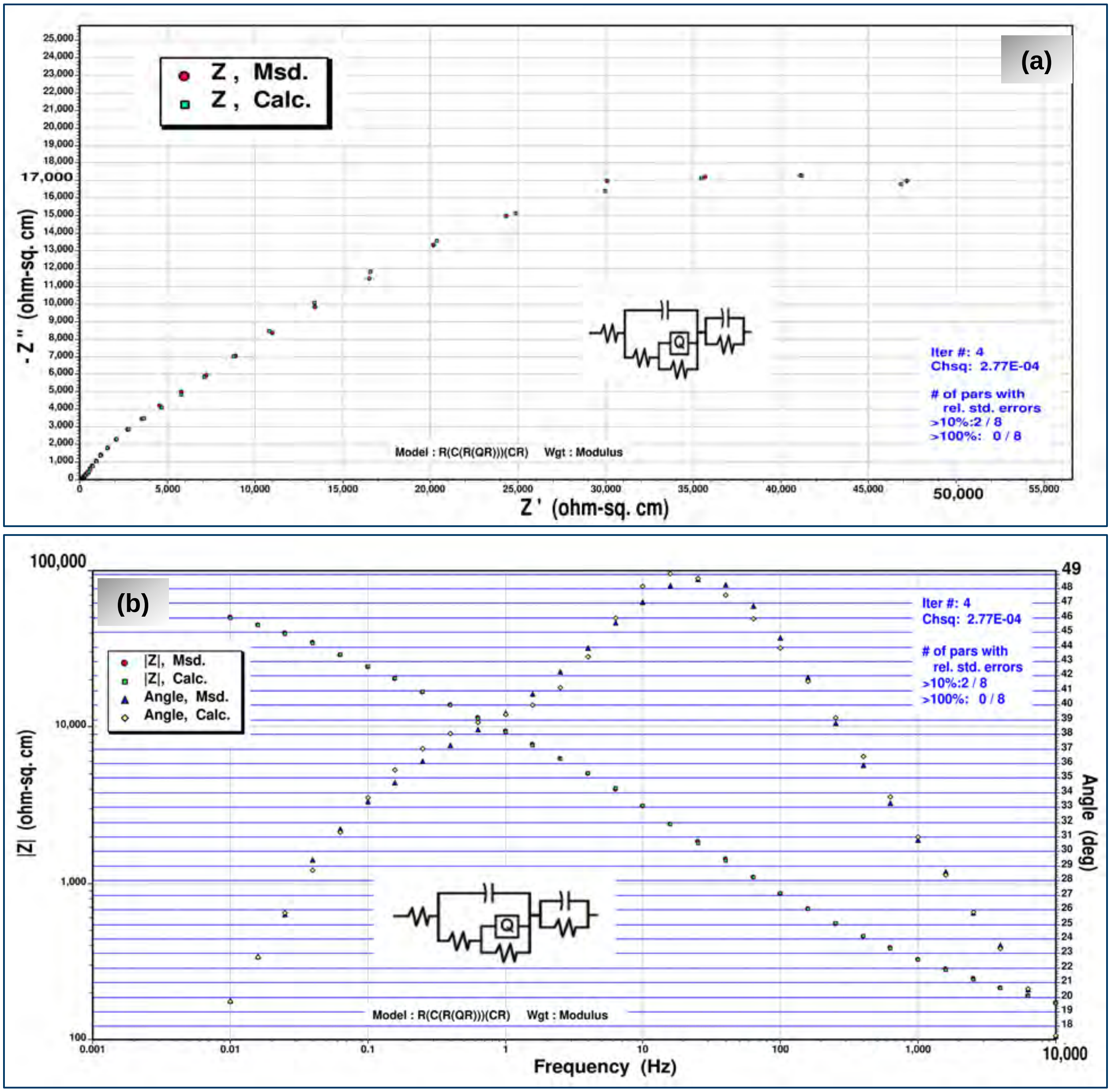


Figure A 18 EIS modeled plots for S-LS-A-day 30, in MgCl$_2$-Beet blend (a) Nyquist plot (b) Bode plots

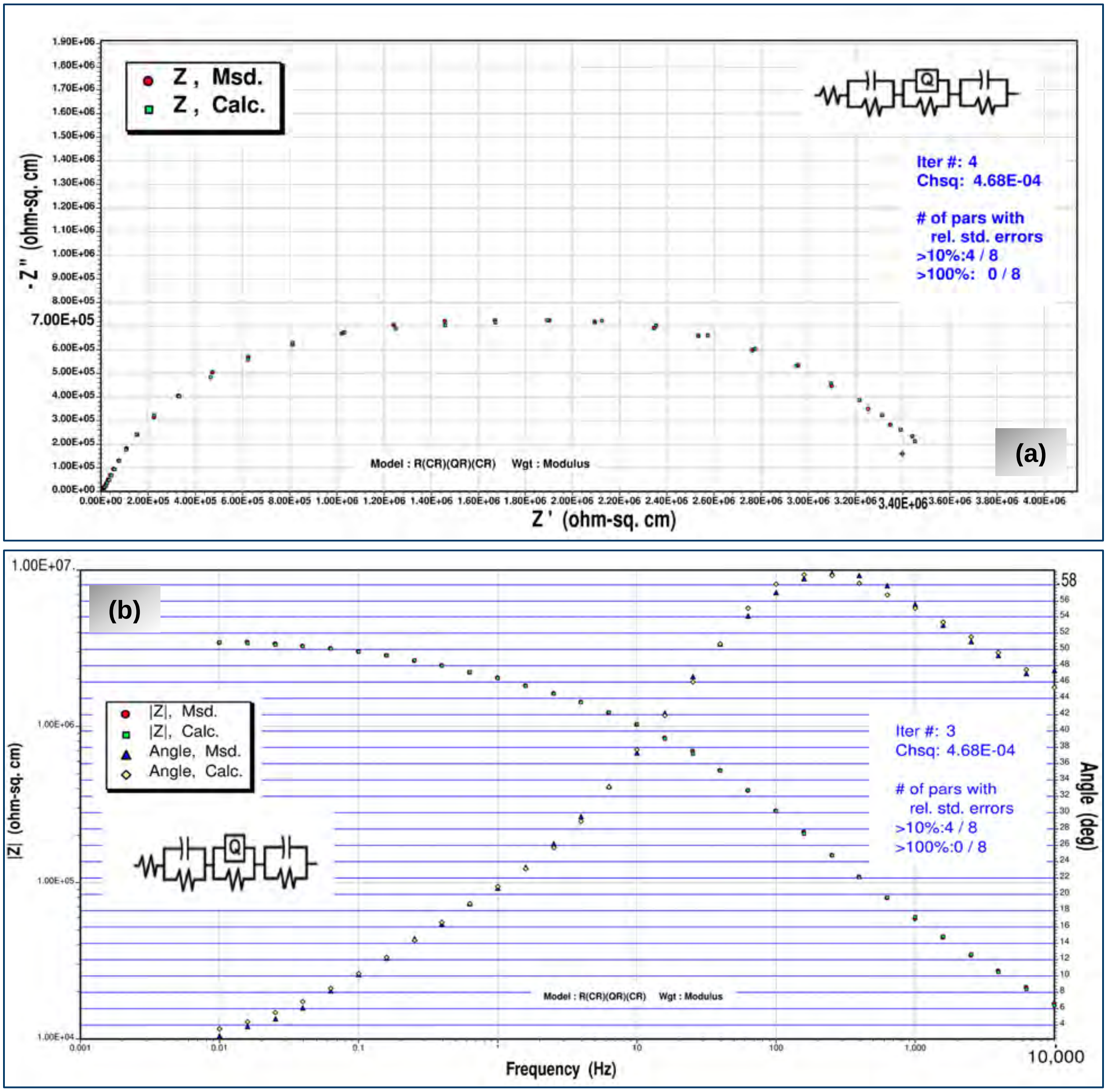


Figure A 19 EIS modeled plots for S-FF-A-day 1, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

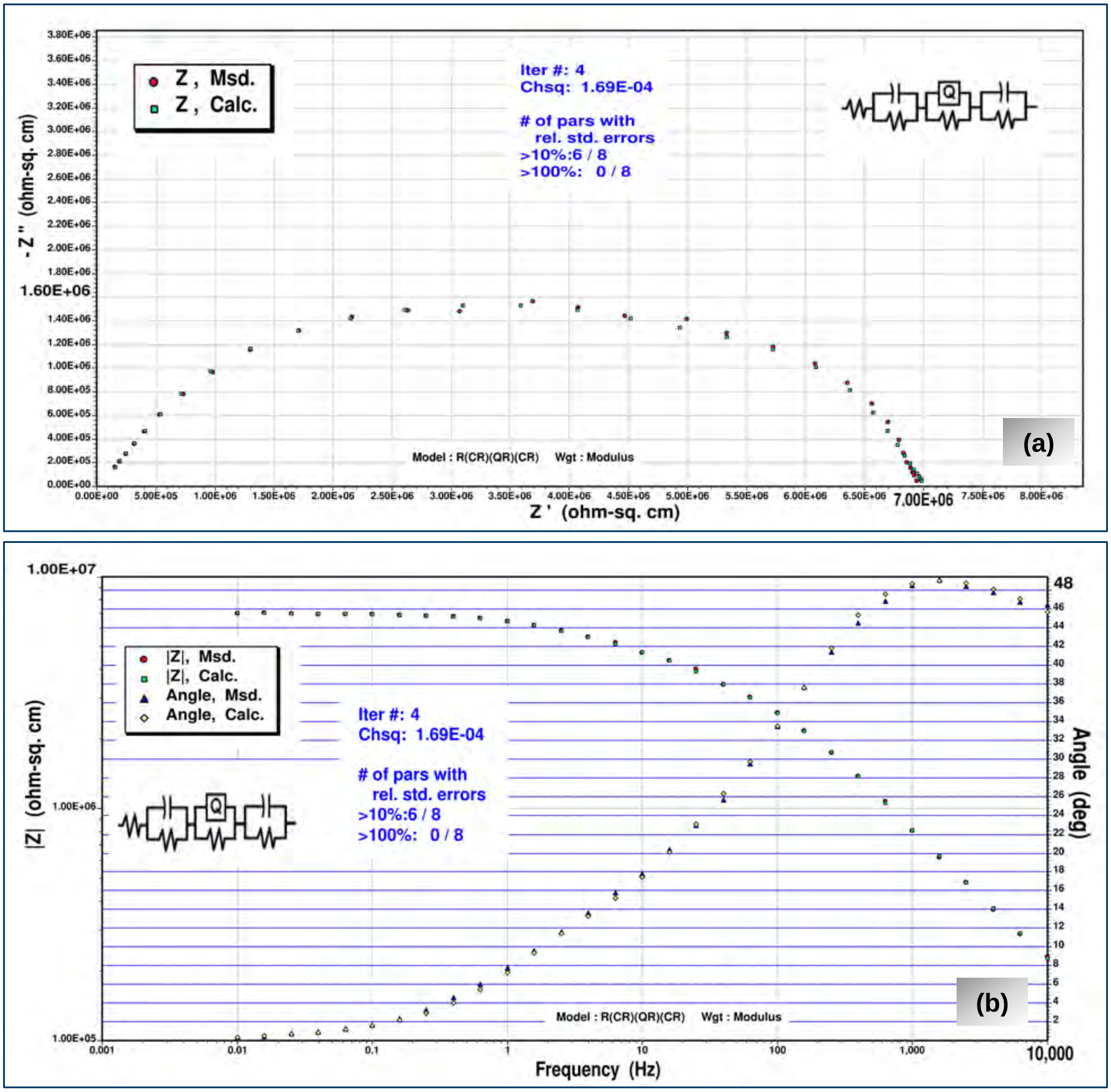
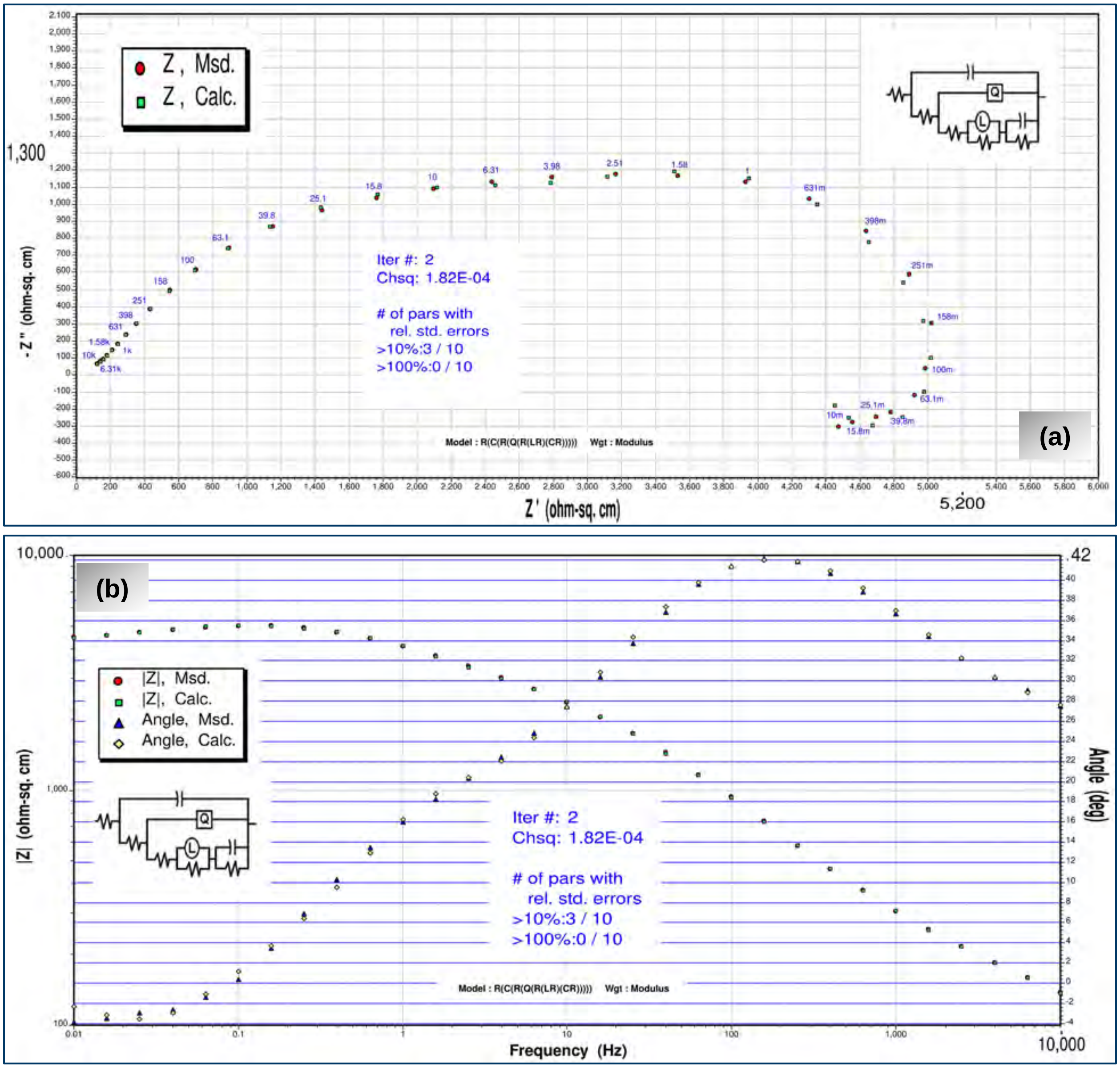


Figure A 20 EIS modeled plots for S-FF-A-day 30, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots



Iter #: 2
Chsq: 1.82E-04

# of pars with
rel. std. errors
>10%:3 / 10
>100%:0 / 10

Model : R(C(R(Q(R(LR)(CR))))) Wgt : Modulus

Figure A 21 EIS modeled plots for S-AP-C-day 1, in MgCl$_2$-Beet blend (a) Nyquist plot (b) Bode plots

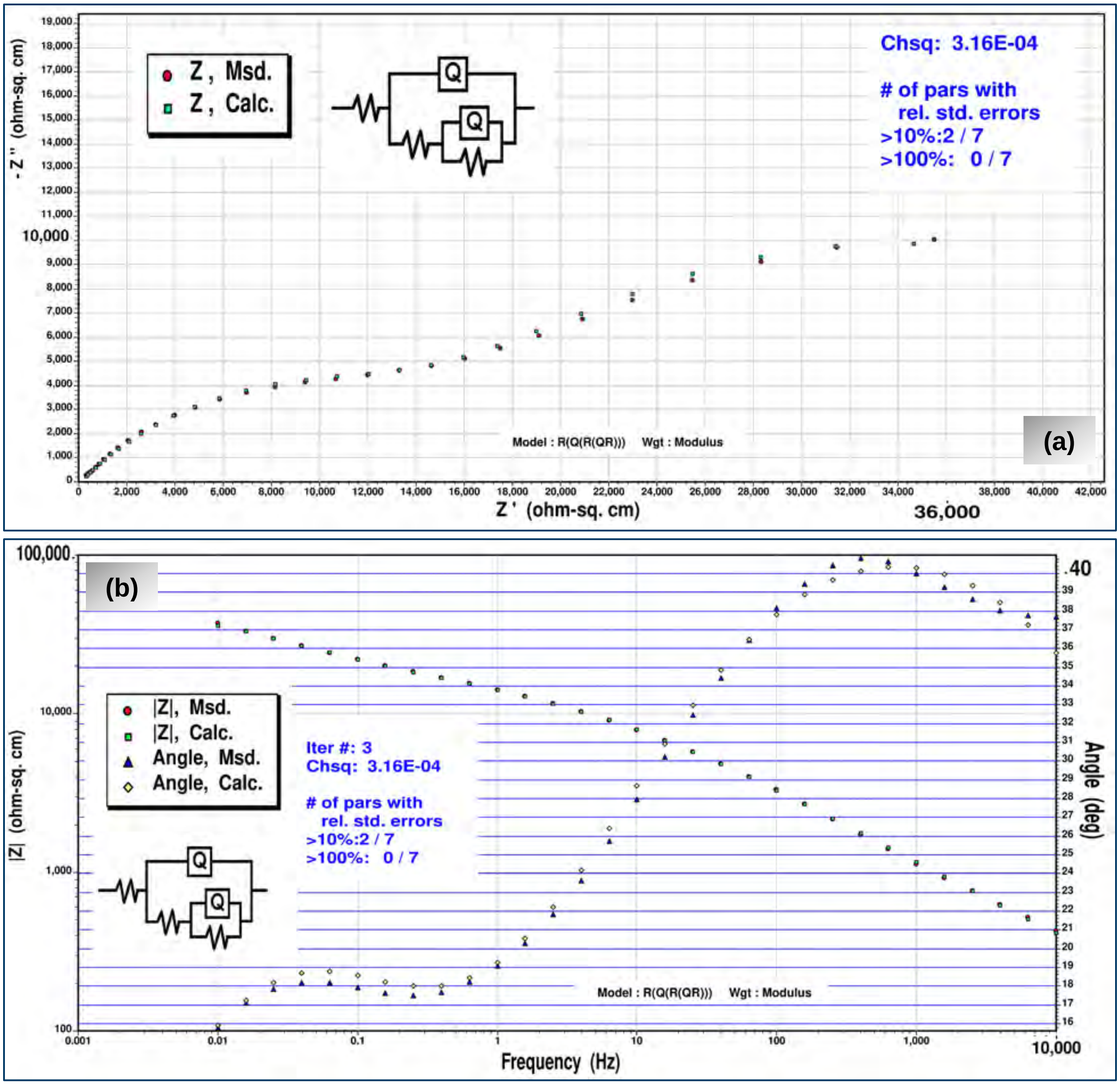


Figure A 22 EIS modeled plots for S-AP-C-day 30, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

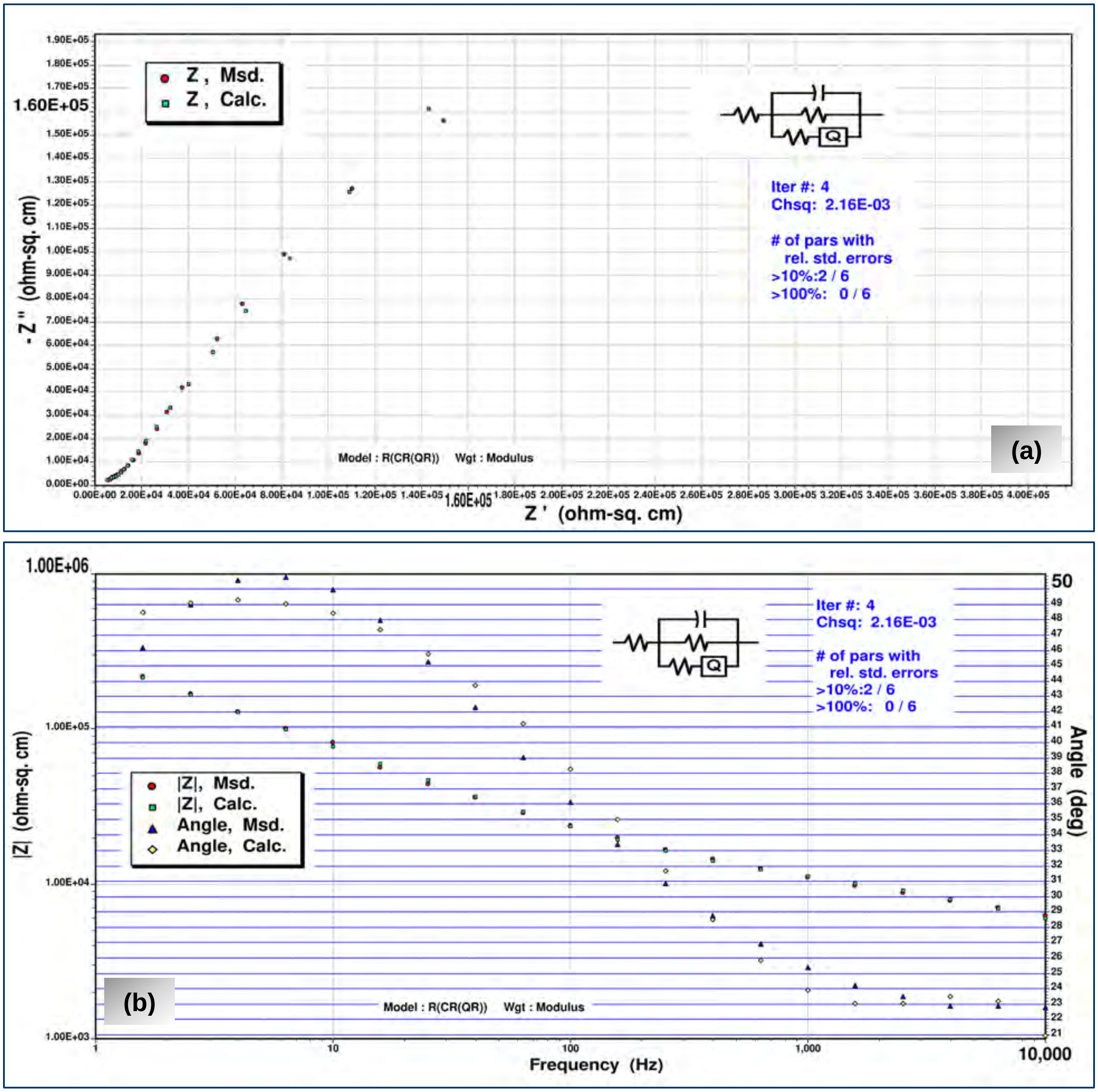


Figure A 23 EIS modeled plots for SS-LS-B-day 1, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

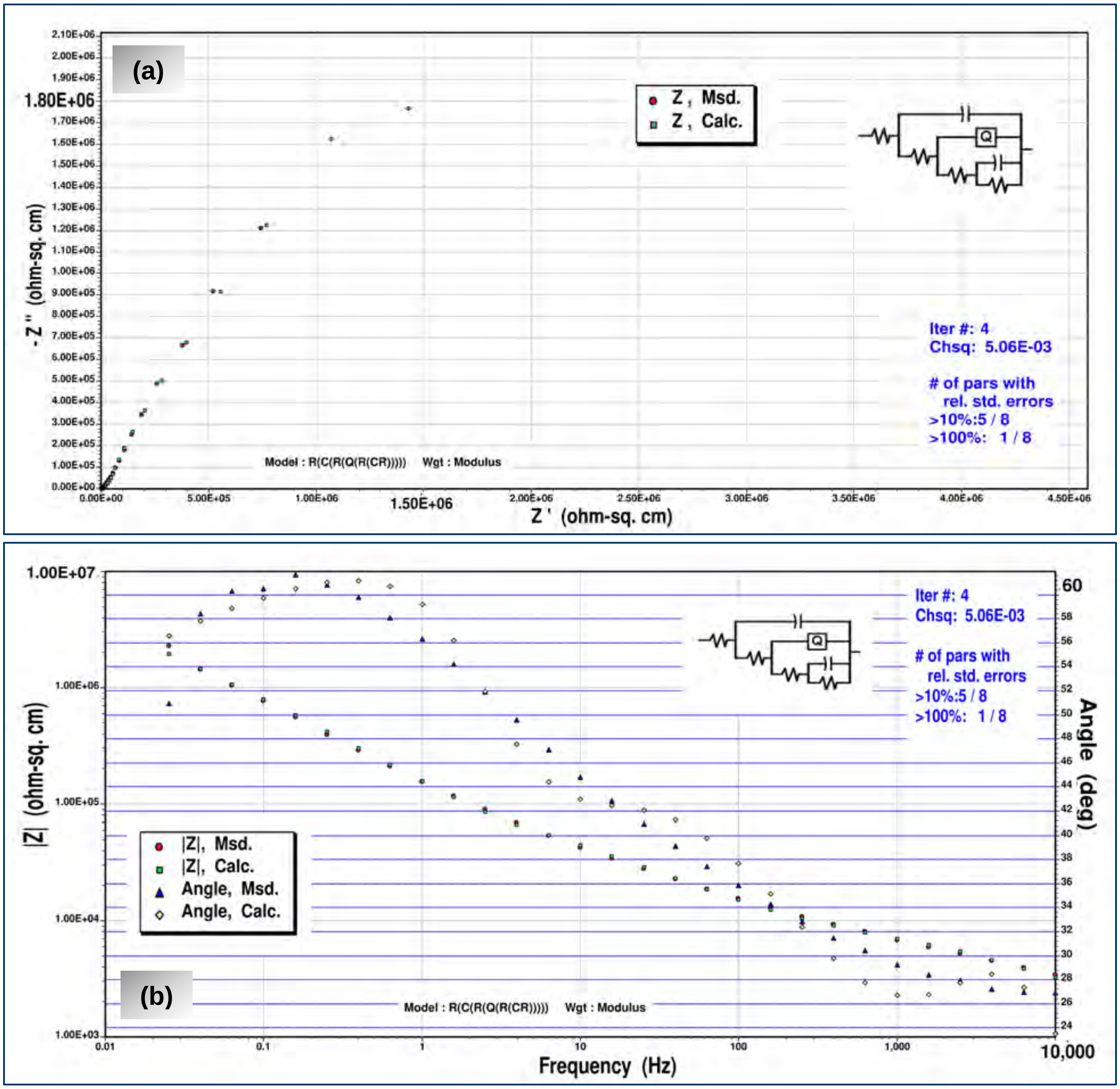


Figure A 24 EIS modeled plots for SS-LS-B-day 30, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

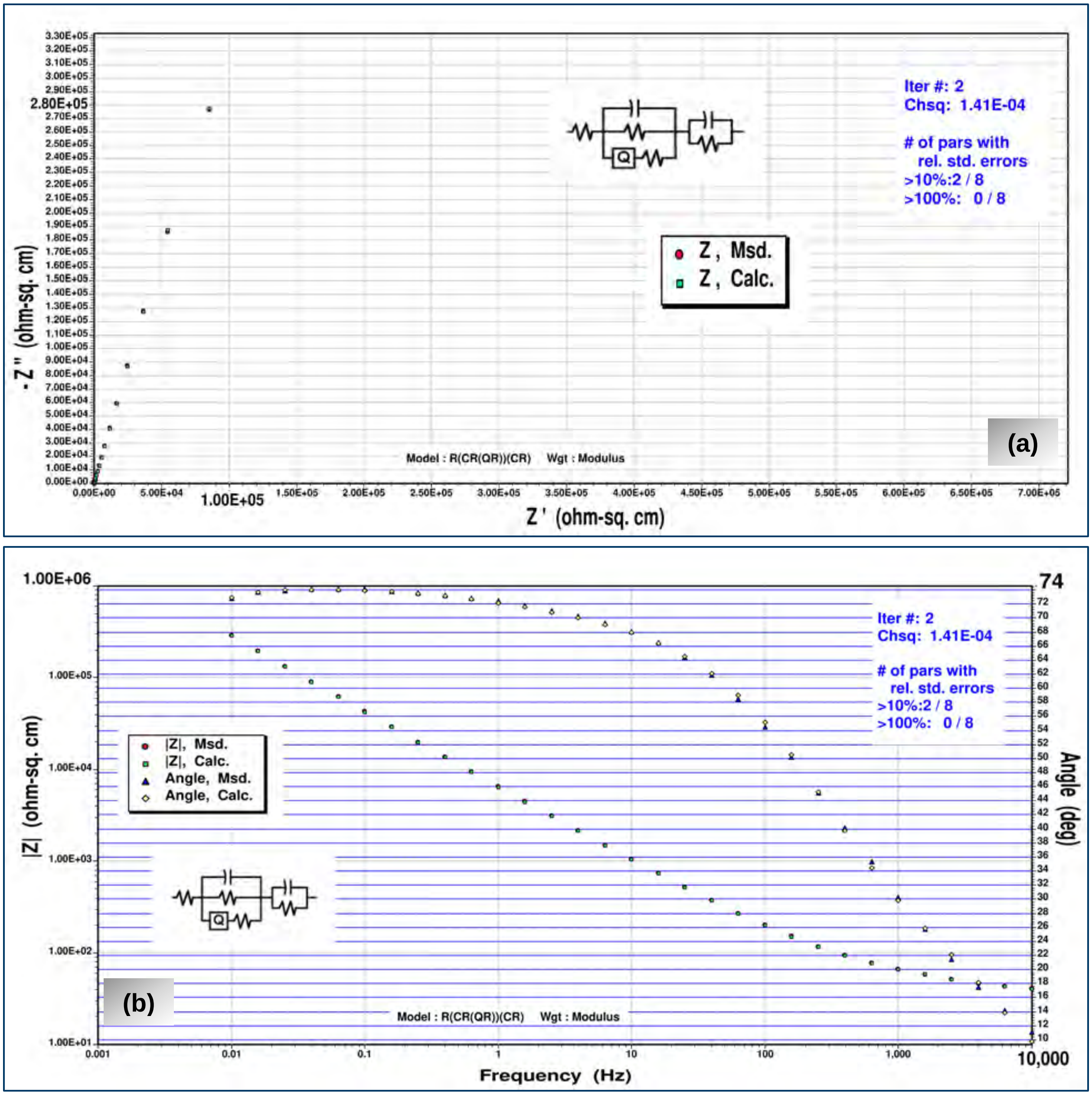


Figure A 25 EIS modeled plots for SS-AS-B-day 2, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

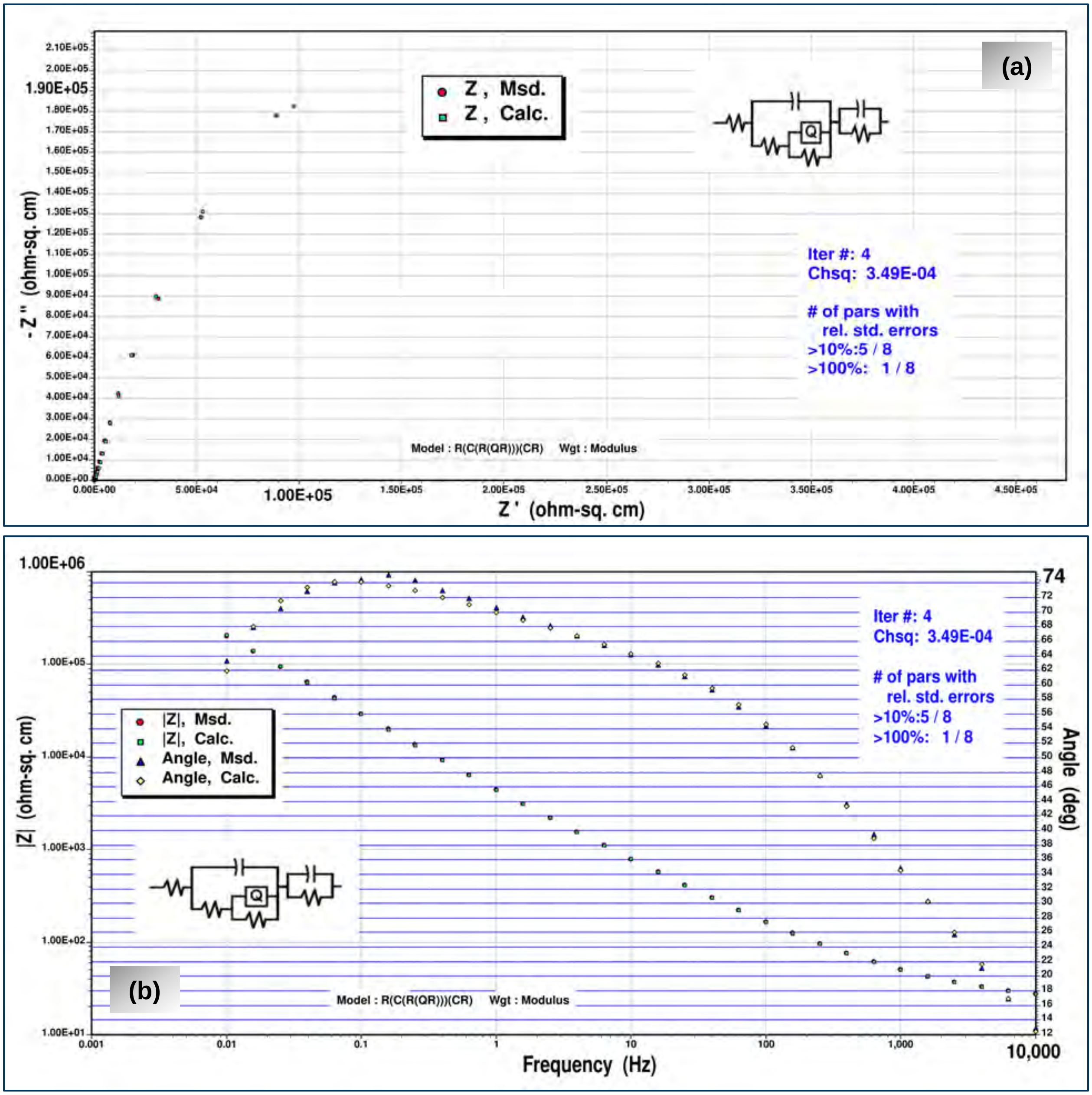


Figure A 26 EIS modeled plots for SS-AS-B-day 30, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

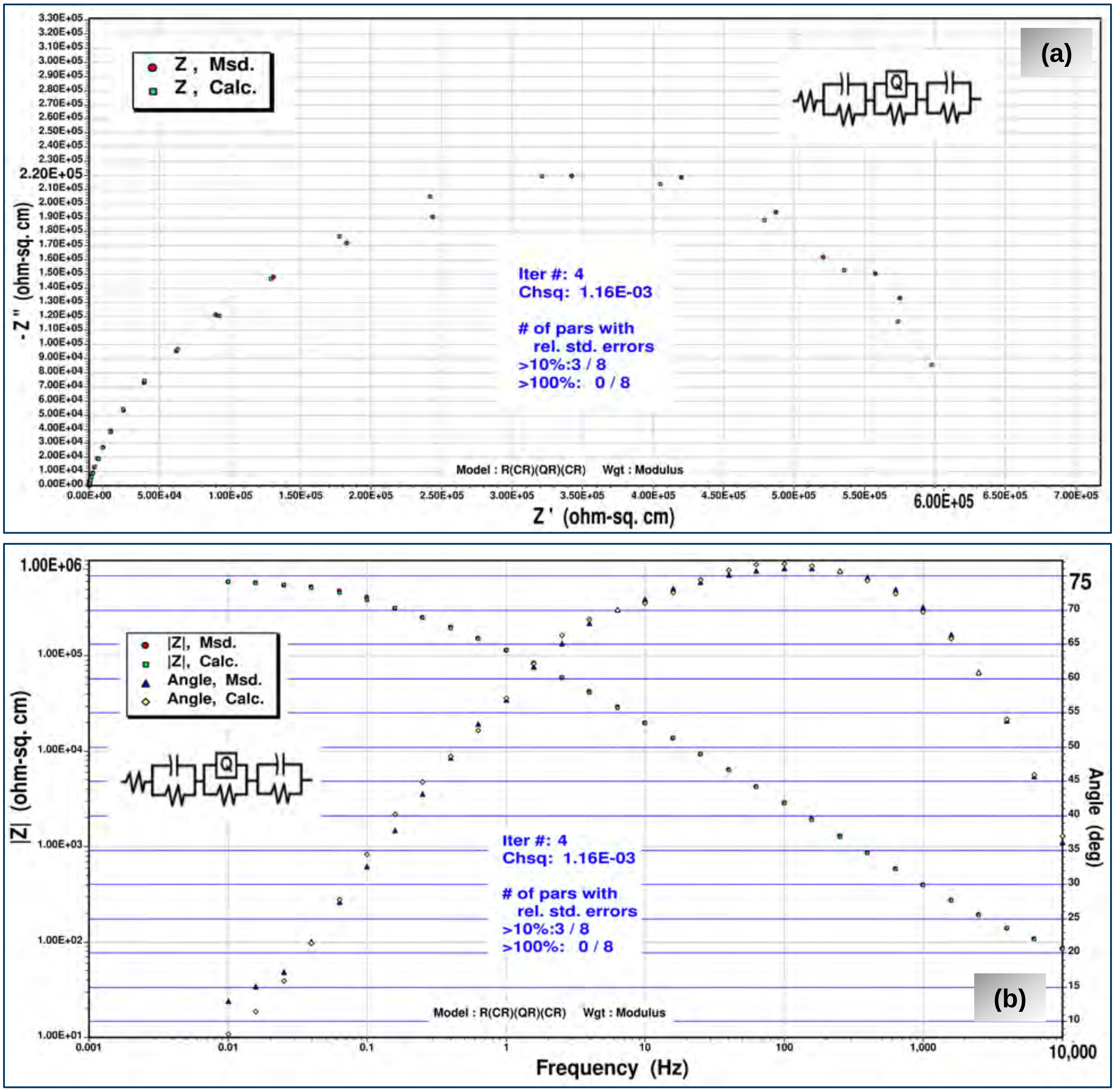


Figure A 27 EIS modeled plots for Al-AS-B-1 day, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

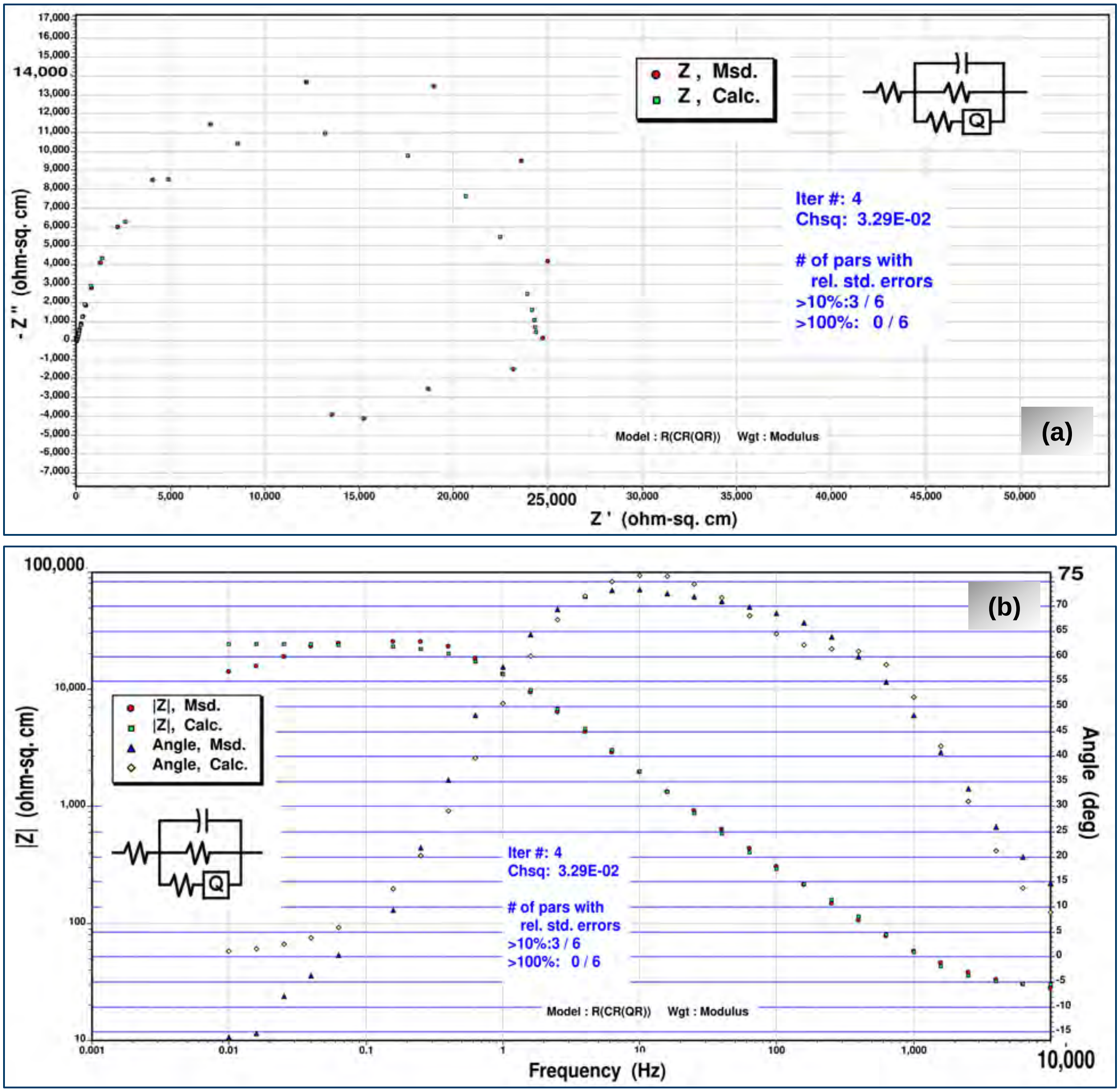


Figure A 28 EIS modeled plots for Al-AS-B-day 30, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

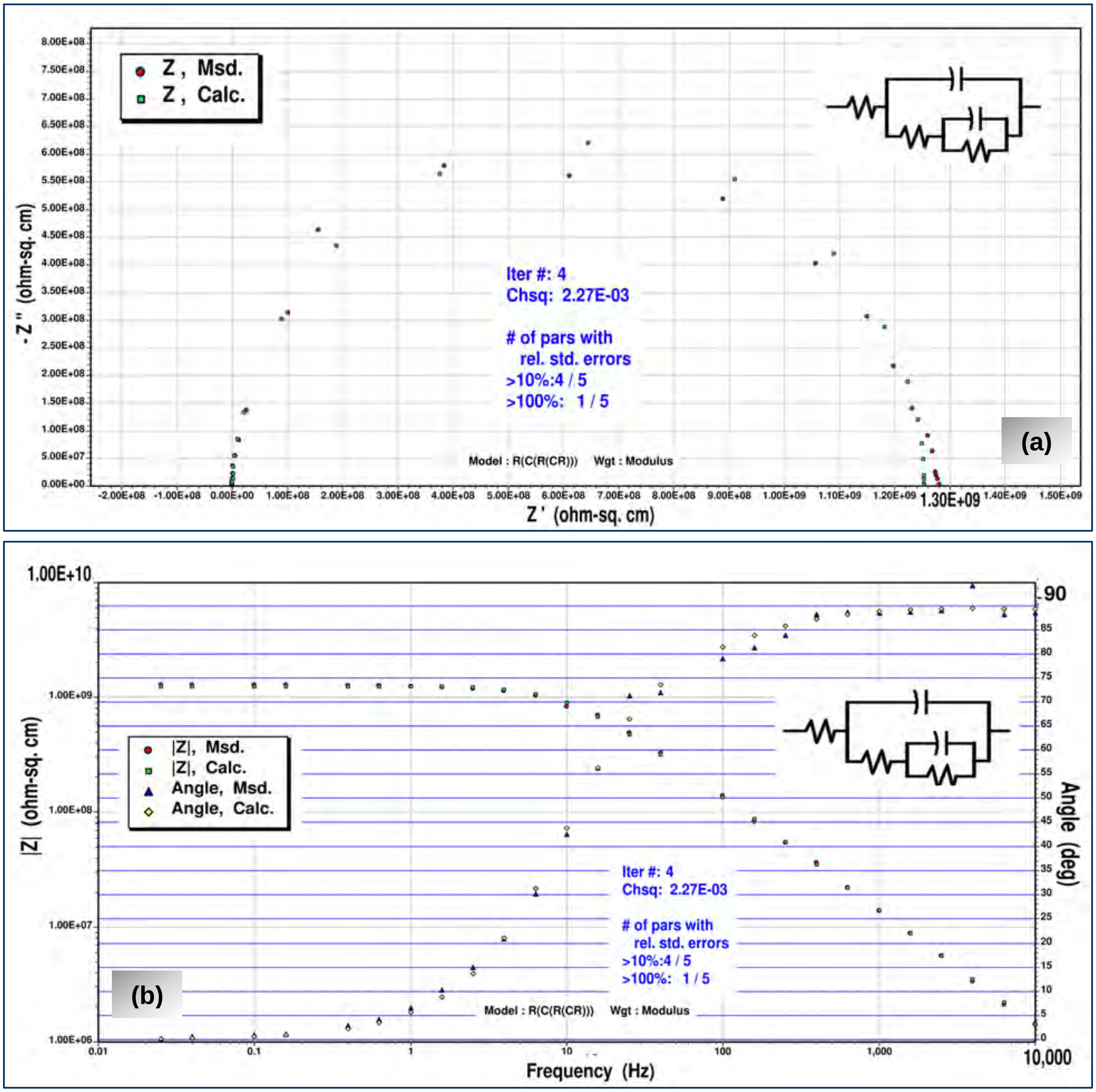


Figure A 29 EIS modeled plots for Al-FF-A-day 1, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

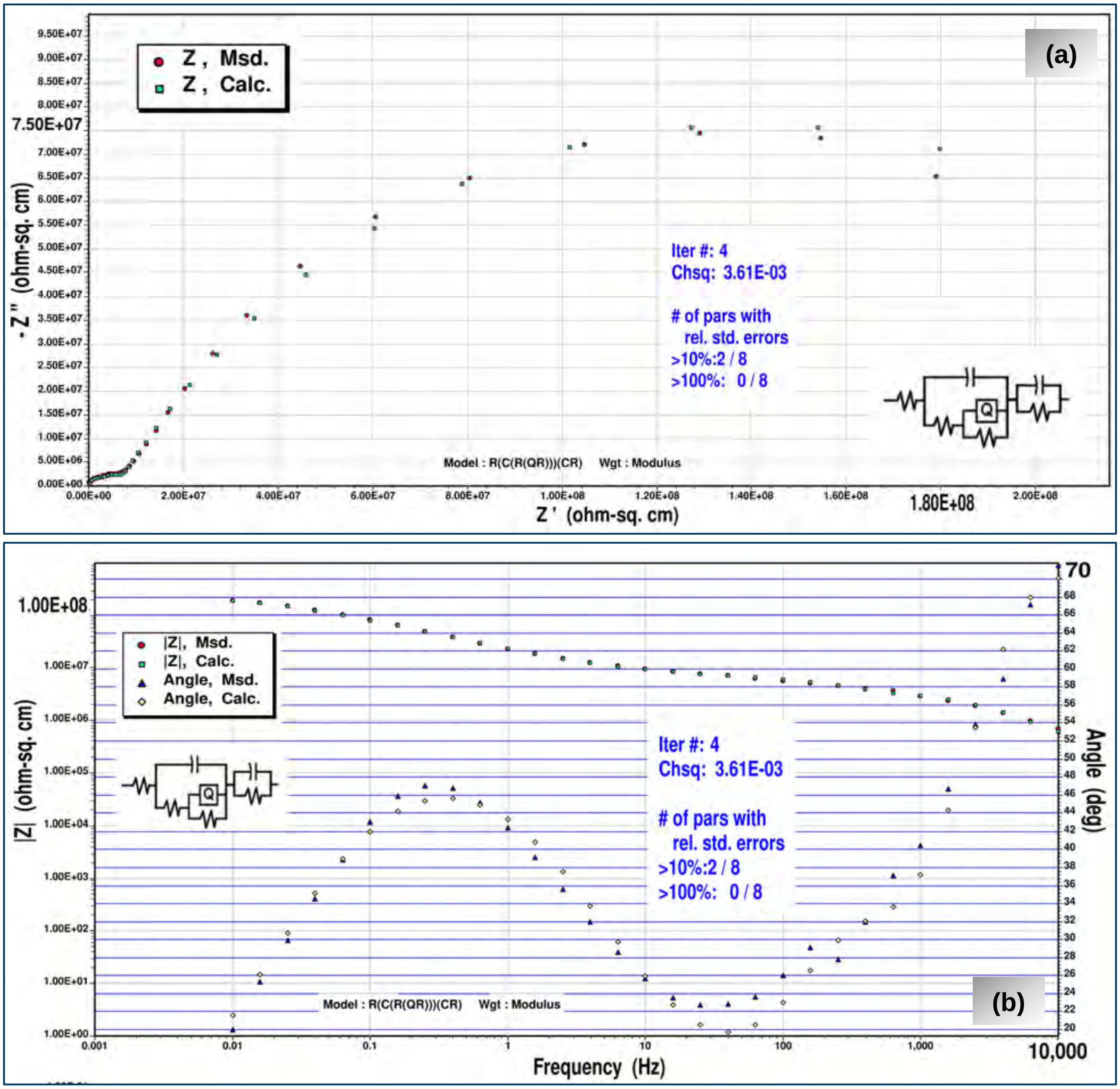


Figure A 30 EIS modeled plots for Al-FF-A-day 30, in MgCl$_2$-Beet blend (a) Nyquist plot (b) Bode plots

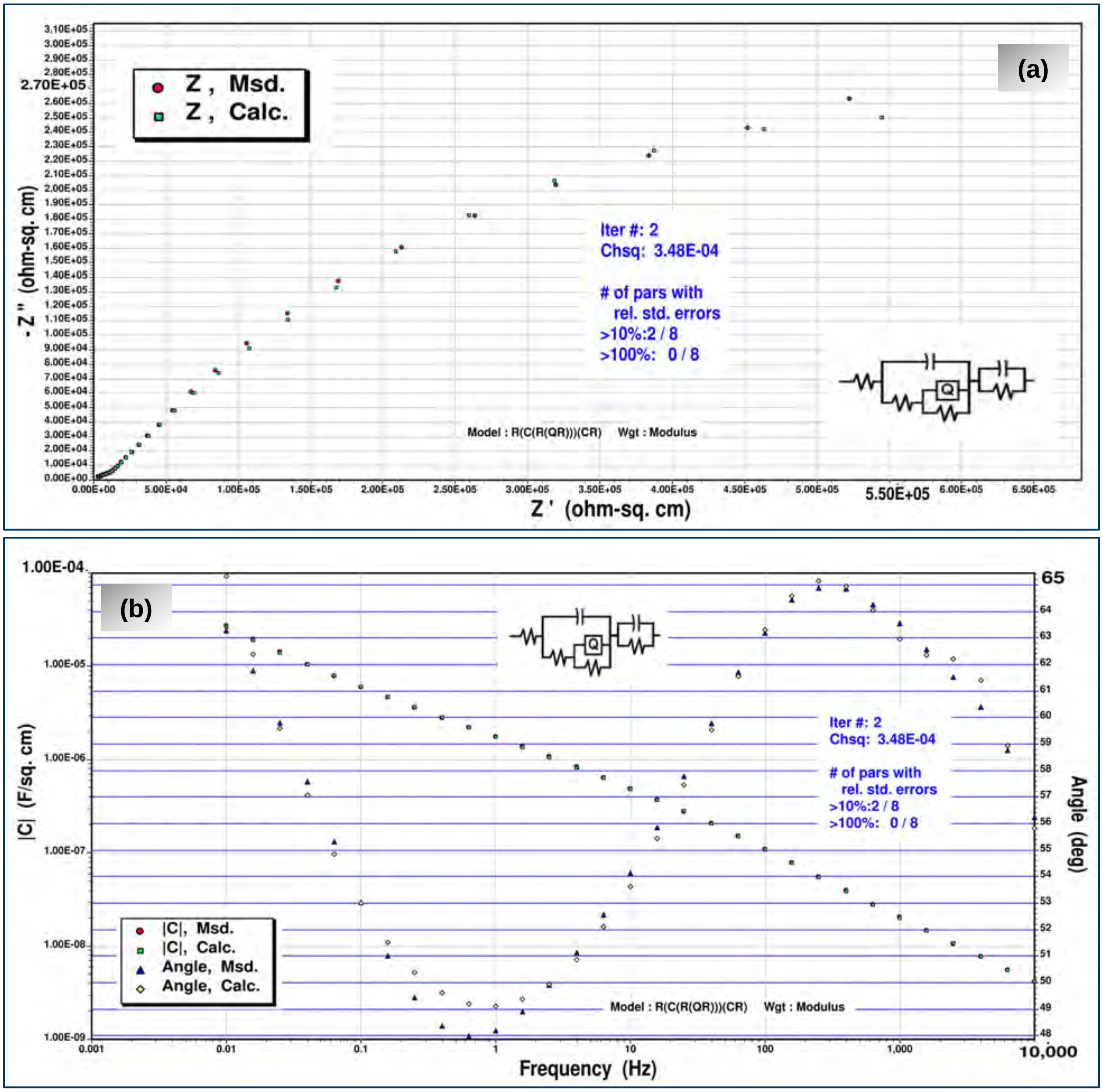


Figure A 31 EIS modeled plots for Cu-DG-A-day 1, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

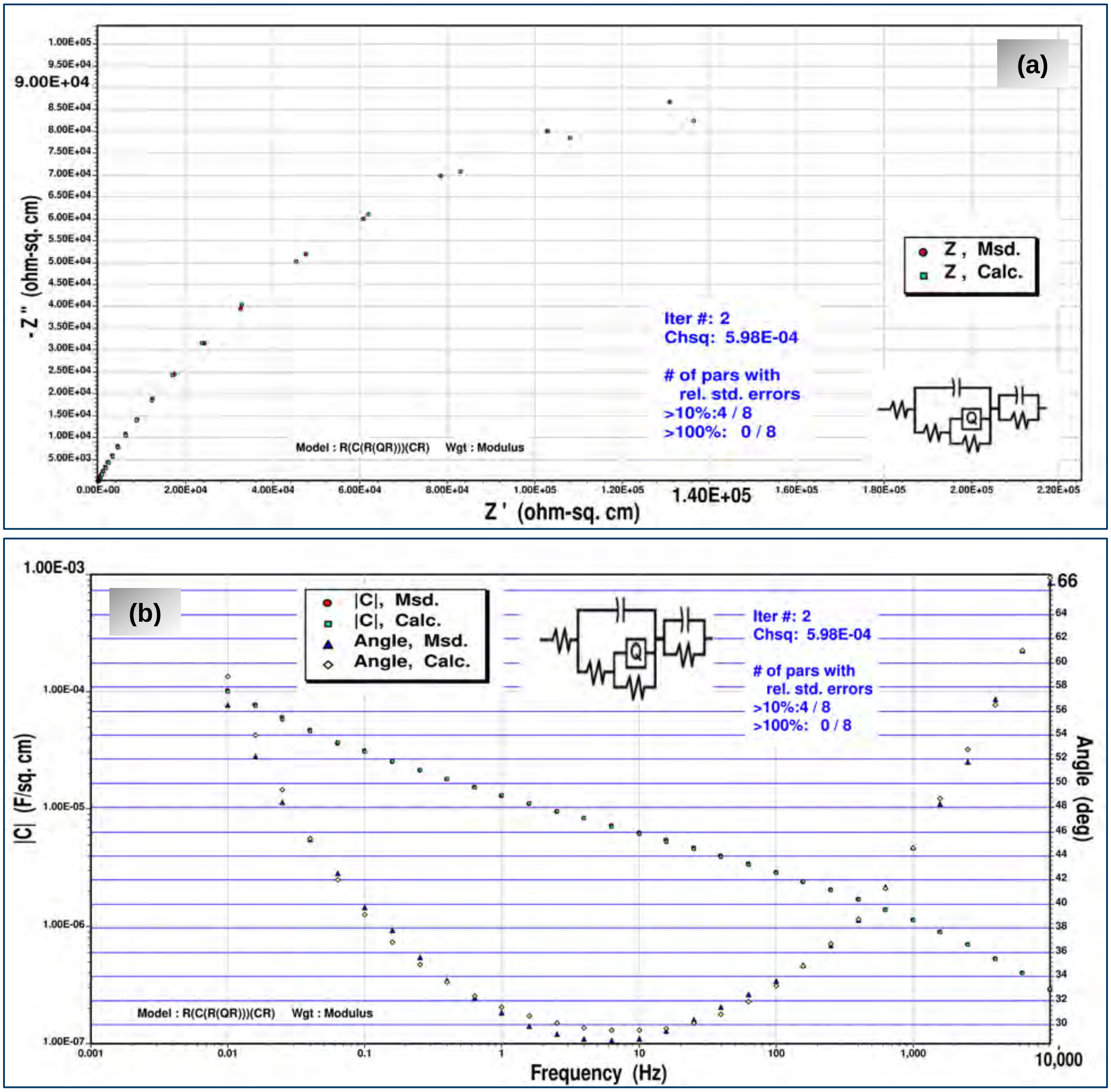


Figure A 32 EIS modeled plots for Cu-DG-A-30D, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

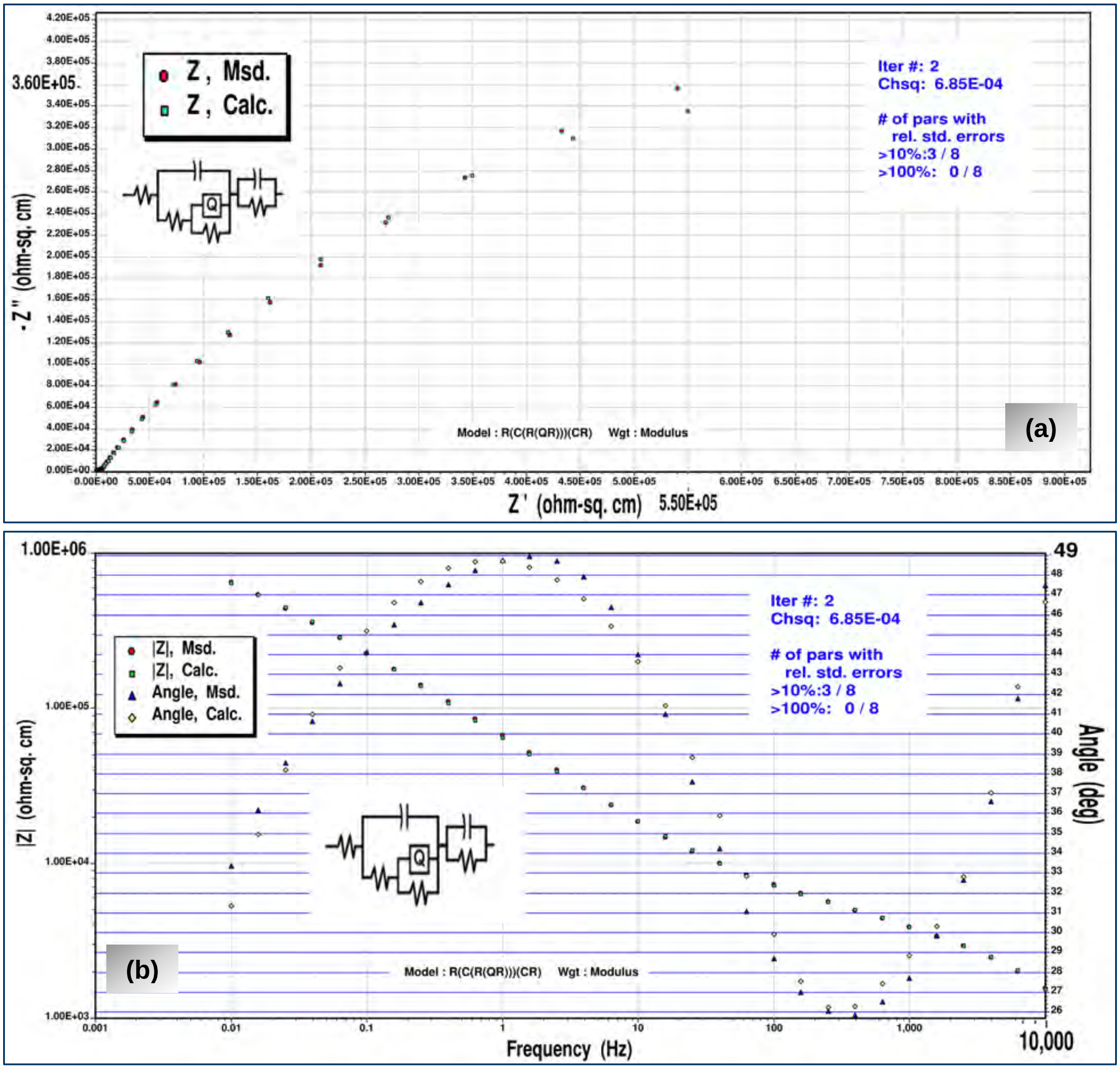


Figure A 33 EIS modeled plots for Cu-DO-B-1 day, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

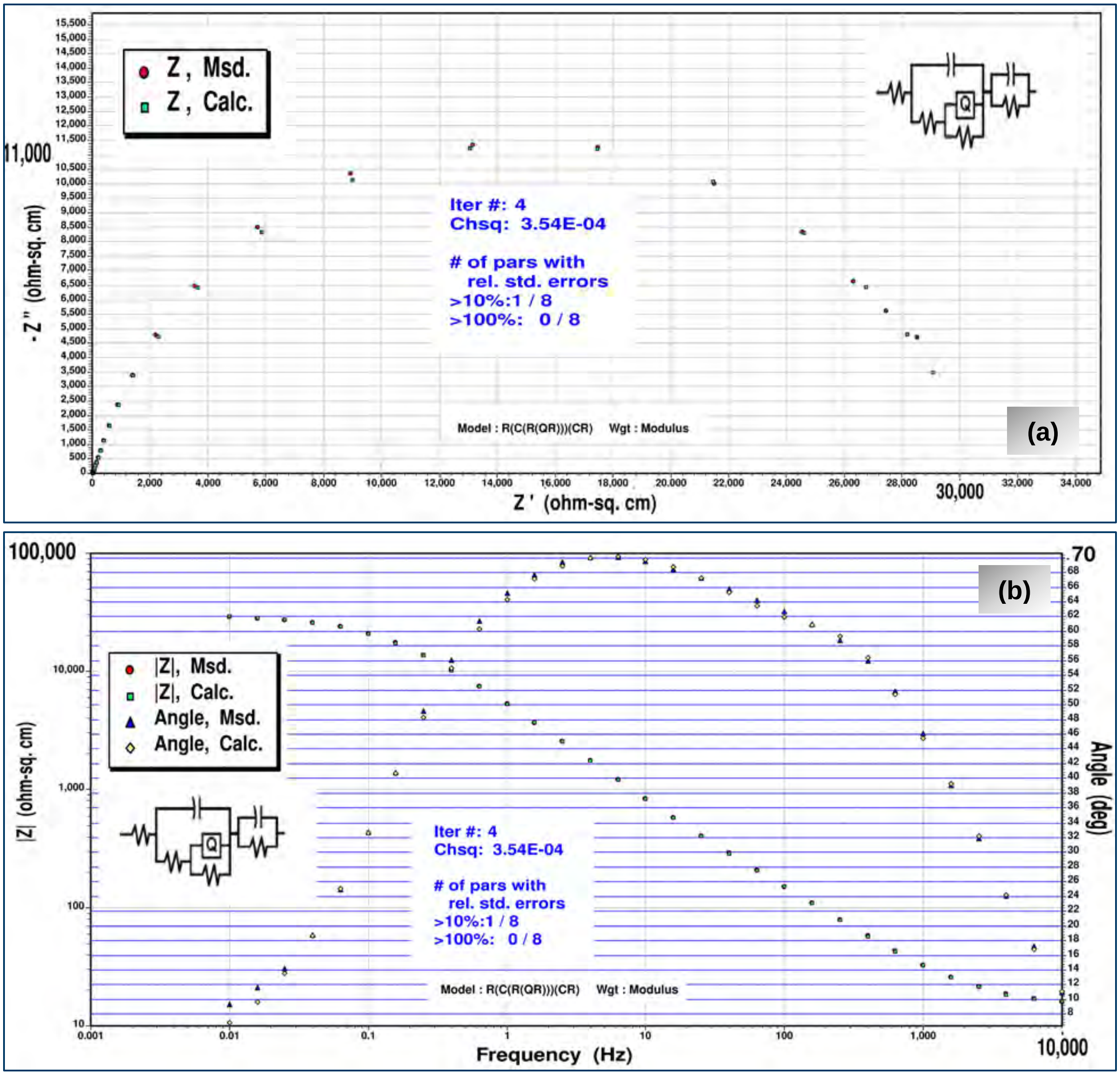
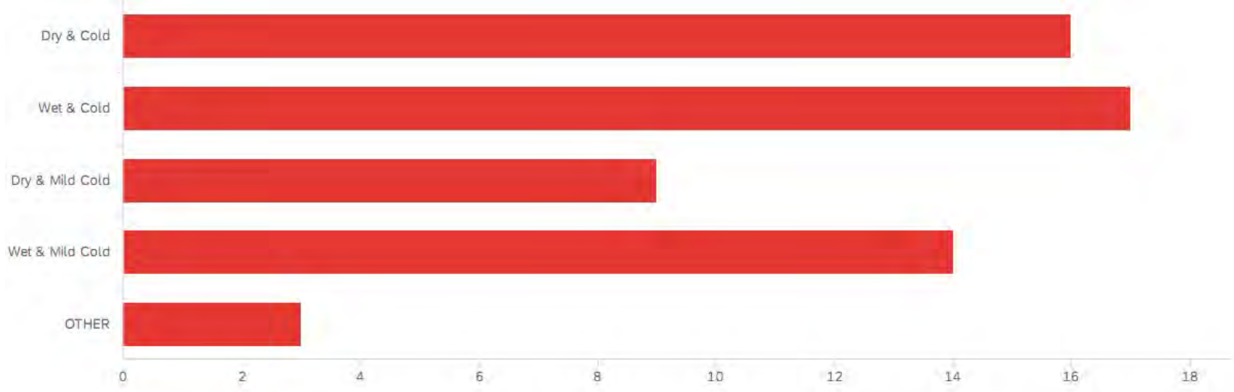


Figure A 34 EIS modeled plots for Cu-DO-B-30 D, in $MgCl_2$-Beet blend (a) Nyquist plot (b) Bode plots

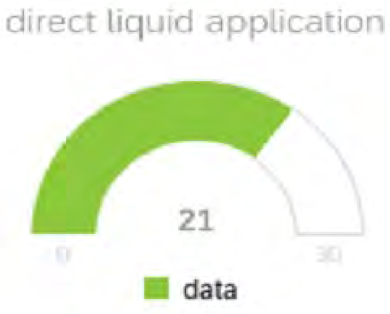
Appendix B: Survey and Market Analysis - Results

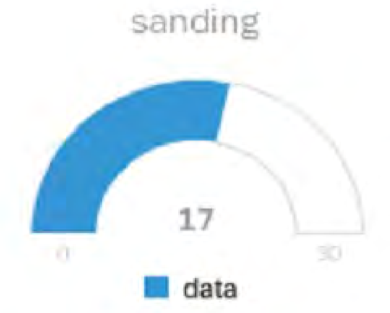
Survey Analysis (Task 1) – Results

Climatic conditions

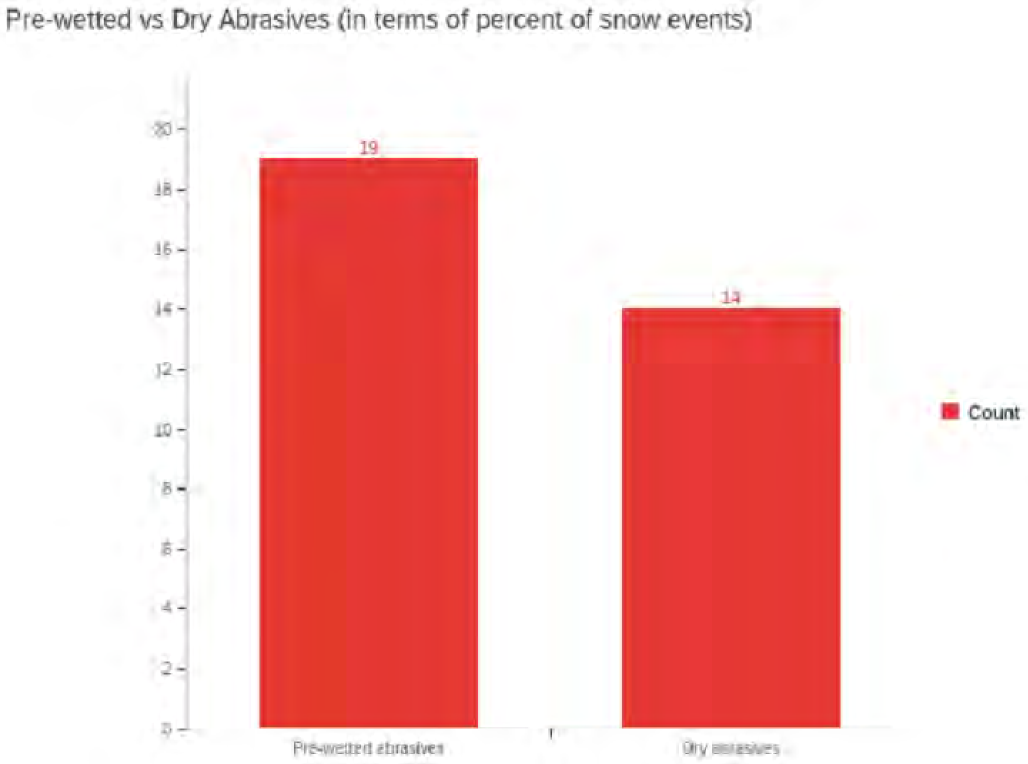


Q.1 Use of various snow control methods in terms of percent of snow events



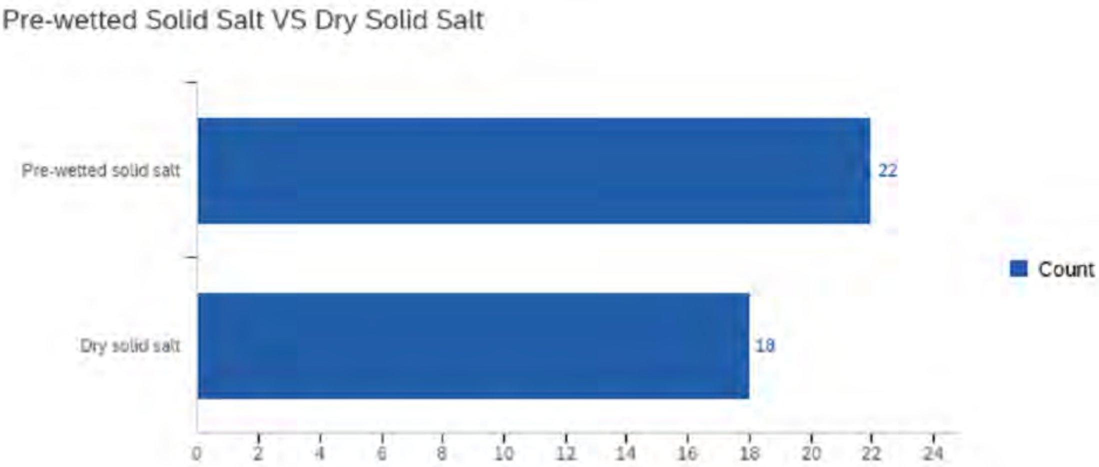


Q.2 Use of pre-wetted abrasives vs. dry abrasives, in terms of percent of snow events.



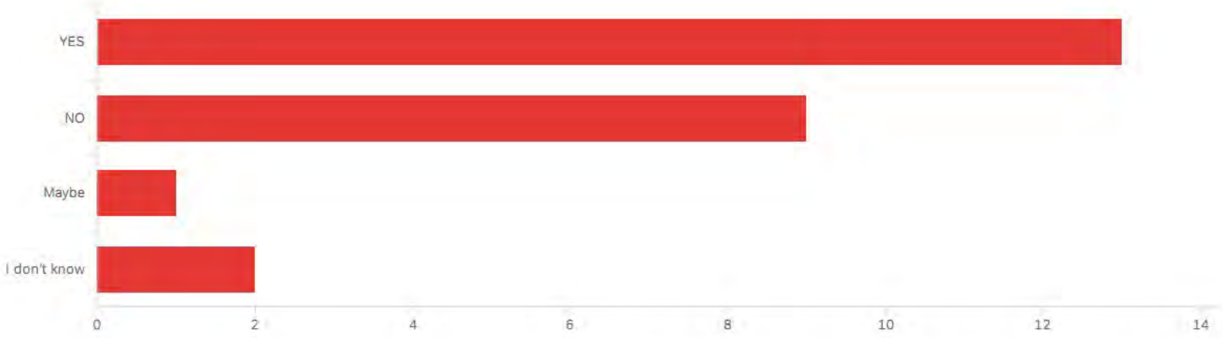
Pre-wetted vs Dry Abrasives (in terms of percent of snow events)

Q.3 Use of pre-wetted solid salt vs. dry salt, in terms of percent of snow events.



Pre-wetted Solid Salt VS Dry Solid Salt

Q.4 Use of corrosion inhibitors and ABPs in deicers and pre-wetting products.



Q. 4(b) Types and brand names of corrosion inhibitors or ABPs

Corrosion inhibitors & ABPs	**Brand Names (if any)**	**State DOT / Agency**
mag chloride	*Not provided*	Idaho Transportation Department
Mag Chloride	*Not provided*	Kansas DOT
Beet juice sugar	*Not provided*	Ohio DOT
Cat 1, QPL	Compass	Wyoming DOT
Mag Chloride	IMPAP	*Not provided*
Sodium nitrite	Salt away	*Not provided*
AMP	Envirotech	*Not provided*
beet juice	Beet heet	Indiana DOT
highly refined carbohydrate concentrate	Beet Heet	Ohio DOT
Beet Juice	*Not provided*	Kansas DOT
Cat 4B & 4C	Slice/Kicker	Wyoming DOT
Sulfamic Acid	Neutro-Wash	*Not provided*
Beet 55	Smith Fertilizer & Grain	*Not provided*
beet juice	Geomelt	Indiana DOT
Apex	Apex C	Ohio DOT
Ice B Gone Magic	SEACO	*Not provided*
proprietary	Headwaters "Hot"	Indiana DOT
Boost	Calcium Chloride with Boost	Ohio DOT

Corrosion inhibitors & ABPs	Brand Names (if any)	State DOT / Agency
Calcium Chloride	Calcium Chloride with Boost	Ohio DOT
unknown	Don’t know	Idaho Transportation Dept.
Beet Heat	K-tech	*Not provided*
proprietary	Magic Minus Zero	Indiana DOT
Beet Juice	GeoMelt	Ohio DOT
Not provided	magic minuszero	New Hampshire DOT
Not provided	BOOST	Michigan DOT
Not provided	Delce Master PLus	Michigan DOT

Q.5 Types of metals/alloys commonly used within the agency in DOT equipment and SMVs.

Q.5 Other

a. AR 400/500 (abrasion resistant steels)

b. Brass and AR steel

Q.6 Risk of deicer corrosion to various types of equipment or vehicles your agency owns.

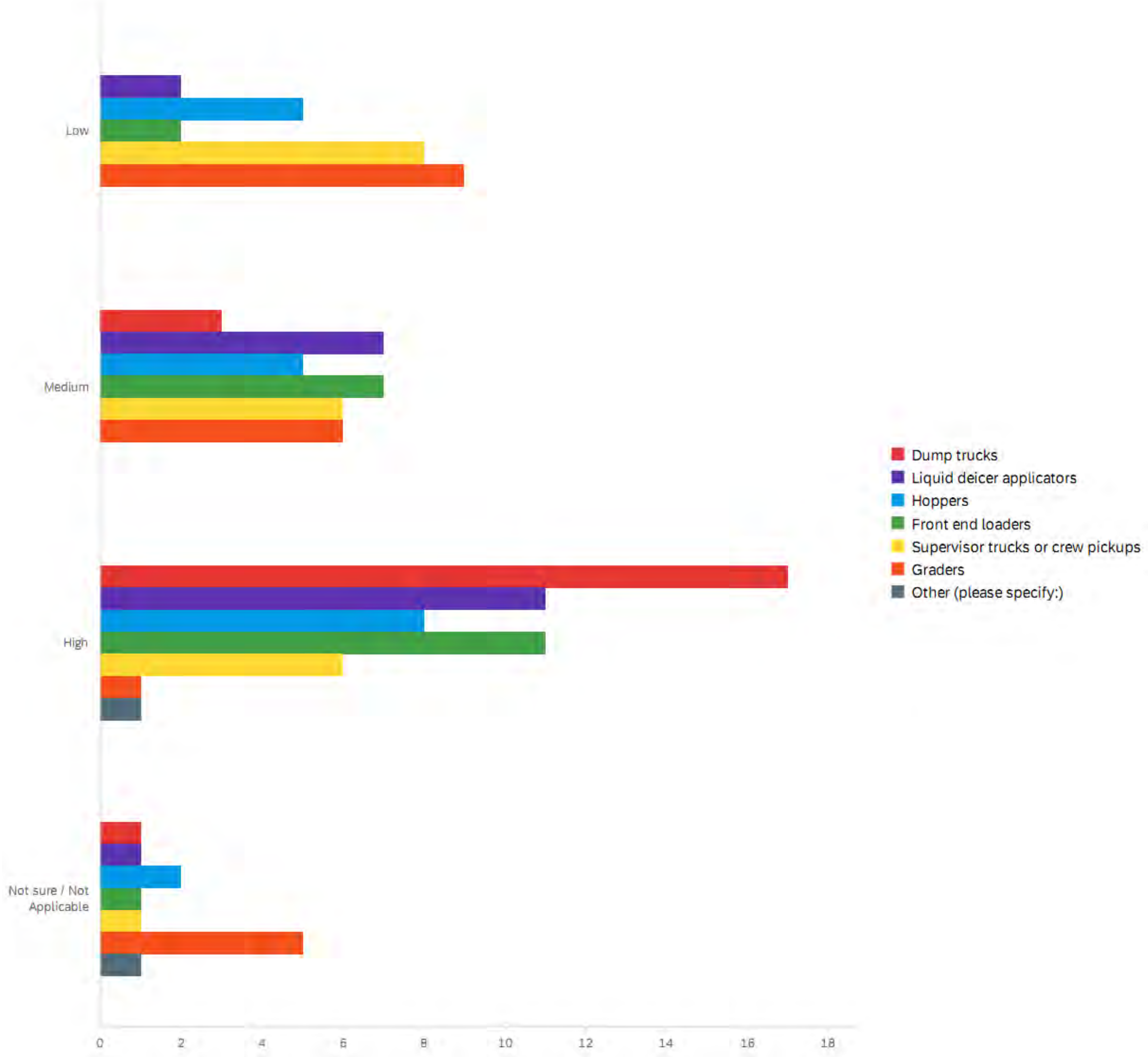


Table B 1 Rank given to DOT equipment in terms of their corrosion susceptibility (1 is the most susceptible)

Rank (In terms of corrosion susceptibility)	DOT equipment
1	Dump trucks
2	Liquid deicer applicators
	Front end loaders
3	Hoppers
4	Supervisor trucks or crew pickups
5	Graders

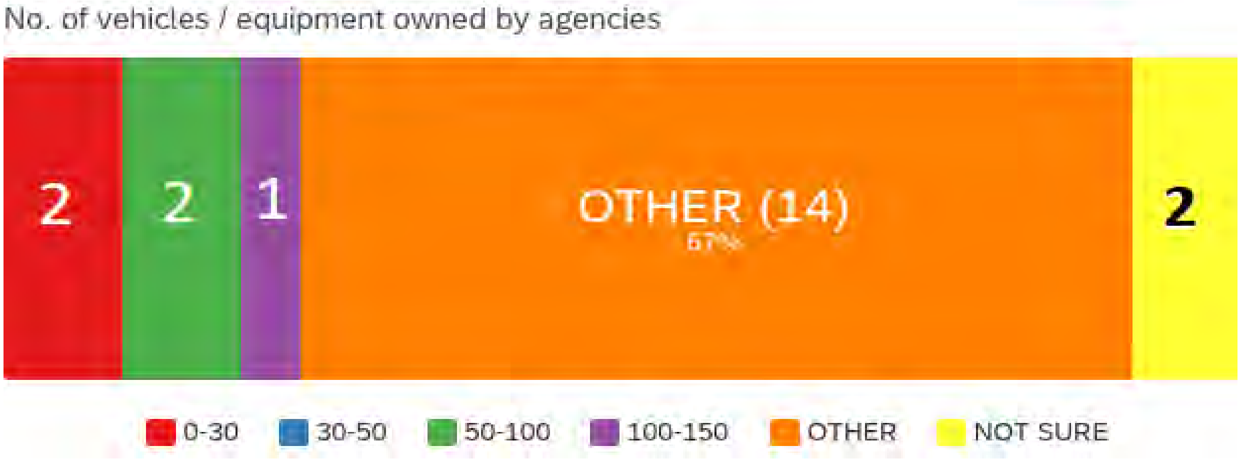
Q.7 Risk of deicer corrosion to various types of components of agency SMVs.

Based on the responses, Table B 2 was developed.

Table B 2 Components of DOT equipment and vehicles, ranked from most to least susceptible to corrosion attack

Rank **(**In terms of corrosion susceptibility**)**	**Components used in DOT Equipment**
1	Brackets & supports
2	Frames
3	Fittings
4	Brake drums & discs
5	Electrical components (e.g., wiring, connectors, terminals)
6	Brake air cans
7	Metallic housings
8	Radiators / Exhaust systems/Mufflers
9	Wheels
10	Engine & drive train components

Q.8 Approximate number of agencies owned SMVs.



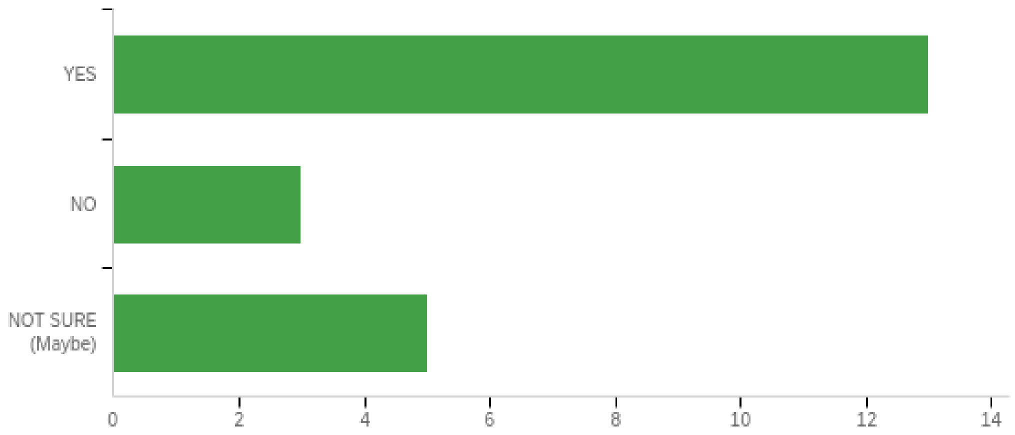
Q.8 Other (Table B3)

Table B 3 Approximate number of SMVs owned by agencies / DOTs

No. of approx. vehicles (category, OTHER)	Agency / DOT
300	Vermont Agency of Transportation
800	Kansas DOT
5,000	Ohio DOT
358	Wyoming DOT
800 plus	New Hampshire DOT
probably close to 1,000	Massachusetts DOT
600	*Not provided*
900	*Not provided*
2500	*Not provided*
350	Delaware DOT
around 400 pieces of equipment	*Not provided*
1100 plow trucks	Indiana DOT
350	Michigan DOT
Over 4,000	Ohio DOT

Q. 10 Have you redesigned the components to reduce corrosion?

Redesigned Components to Reduce Corrosion



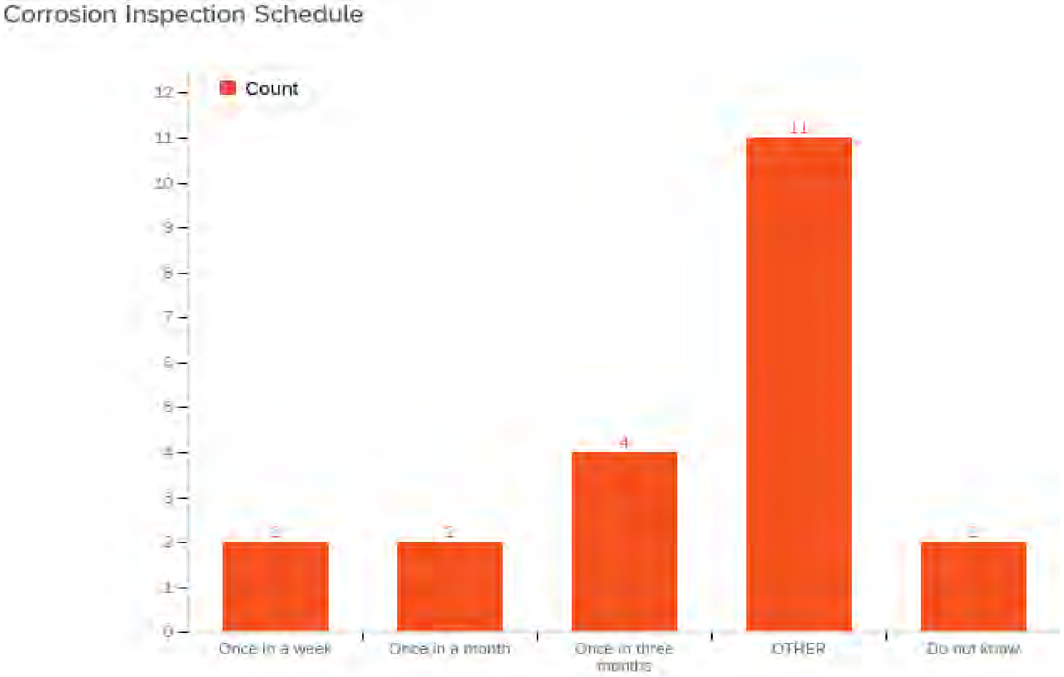
Q. 10(b) Further details on Q. 10 are added to Table B 4

Table B 4 Further details on redesigning components

Please provide information on the components and how they changed.	Which particular aspect of designing can your agency invest in?	Approximately how much your agency can invest/annum on improving the design?	State DOT/ Agency
Utilize **stainless steel** wherever possible	purely from a specifications stand point to potential bidders who would build our equipment	zero	East End Crossing Partners
our state has spec'd for **stainless steel** boxes and slide-in V-box Sanders	unknown	unknown	Unknown
stainless steel oil pans and hydraulic lines	don`t know	don`t know	Idaho Transportation Department
Majority of carbon steel components have been changes to nonferrous material	We can design our own equipment as we see fit and the material we use.	We write into the specifications for our builds. We do not have a dollar amount.	Ohio DOT
We have moved to **stainless steel** beds and hoppers from carbon steel construction.	Require heavier rust proofing on our engine oil pans.	Unknown.	Ohio DOT
Beds and other components changed to **stainless steel**, aluminum and plastic	The electrical system and the frame components	Not sure	Indiana DOT
Specd. out **stainless steel** fittings, boxes	None	None	*Not provided*
Replace transmission line with **stainless steel** fitting and Hydraulic hose., Replace power steering lines with stainless lines, replaced and spec changes to all engines in dump trucks to stainless oil pans	Spec out Stainless wherever financially feasible	Don't have a number	Vermont Agency of Transportation

Please provide information on the components and how they changed.	**Which particular aspect of designing can your agency invest in?**	**Approximately how much your agency can invest/annum on improving the design?**	**State DOT/ Agency**
Connectors change yearly. Changed from steel beds to **stainless steel**	vehicle components to reduce replacement due to corrosion	+$50k	Wyoming DOT
Body corners are now Stainless Steel, spreaders are **Stainless Steel**	Physical design	$0.00	New Hampshire DOT
Carbon steel truck beds to **stainless**	*Not provided*	*Not provided*	West Virginia Department of highways Equipment Division
Spreader bodies and dump boxes are now **stainless steel**.	More slide in spreaders with auger feed systems.	*Not provided*	Idaho Transportation Department
converted dump body to **stainless**	*Not provided*	*Not provided*	Kansas DOT

Q. 11 Corrosion inspection cycles for agencies' equipment and SMVs

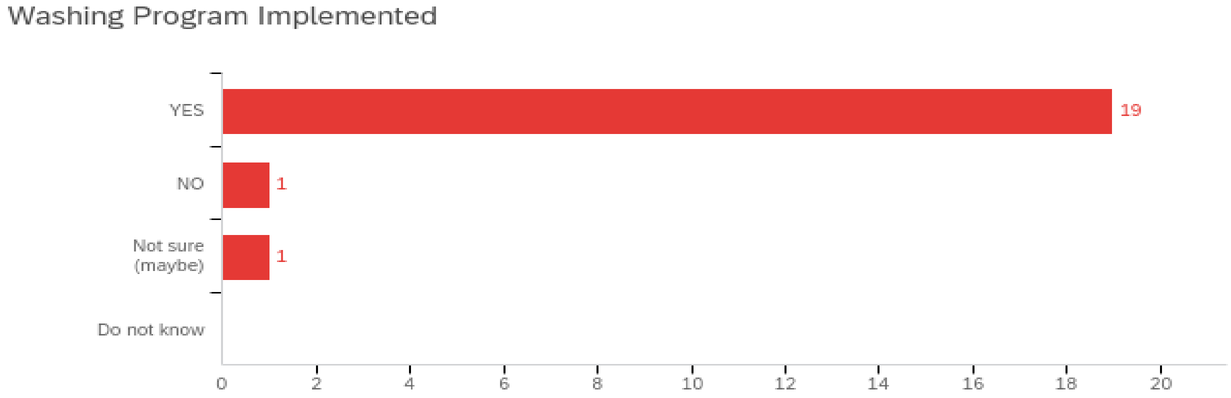


Q. 11 Other (Table B 5)

Table B 5 Details on corrosion inspection cycles (Q. 11 OTHER)

Corrosion Inspection Cycles (OTHER)	State DOT / Agency
Annually and during other inspections. We do a G inspection every year to check for corrosion.	Ohio DOT
Once a year	New Hampshire DOT
should be done more frequently; we purchase salt neutralizer	*Not provided*
Bi-Annually	*Not provided*
semi annually	Delaware DOT
Semi annually	Idaho Transportation Department
Each piece of equipment gets washed after each winter storm. During the summer equipment gets washed after every minor and major periodic maintenance.	*Not provided*
After every storm, trucks are inspected and cleaned.	Indiana DOT
The drivers check for corrosion issues as they wash their vehicles.	Ohio DOT
At scheduled services.	Bozeman MT
Twice a year but we have rigorous cleaning processes to follow after each winter event.	East End Crossing Partners

Q. 12 Washing program implemented by the agencies



Q. 12(a) Frequency of washing

Categories	No. of responses
Once a week	5
Twice a week	None
Once a month	None
OTHER	14

Q. 12(a) OTHER (Table B 6)

Table B 6 Details on frequency of washing (Q. 12b, OTHER)

Frequency of washing (OTHER)	State DOT /Agency
varies from shed to shed	Idaho Transportation Department
After every storm event	Vermont Agency of Transportation
After Snow event	Kansas DOT
After each storm.	Ohio DOT
after a storm event	Wyoming DOT
When weather and operations allows	New Hampshire DOT
After every storm	*Not provided*
After every major event or 1x/wk.	*Not provided*
after events	Delaware DOT
After each storm event.	Idaho Transportation Department
After storms	Idaho Transportation Department
after every storm	Indiana DOT
After each storm.	Ohio DOT
After every storm	East End Crossing Partners

Q. 12(b) Policy related to washing program (Table B 7)

Table B 7 Policy related to washing of vehicles

Details of washing policy (if any)	State DOT /Agency
states that equipment should be kept reasonable clean	Idaho Transportation Department
Operators are trained to wash and maintain equipment to a standard policy	Vermont Agency of Transportation
We stress high volume/low pressure. Especially around electronic and wiring.	Ohio DOT
no formal policy, just best practice. Supervisor inspection to insure max effort is taken.	Wyoming DOT
Must be conducted in an approved washing area, not all locations are approved designated washing areas. (water shed concerns)	New Hampshire DOT
Salt Release Wash Agent	West Virginia Department of highways Equipment Division
water only where rinsate drains to storm drain	*Not provided*
Wash before coming into shop for repairs.	Idaho Transportation Department

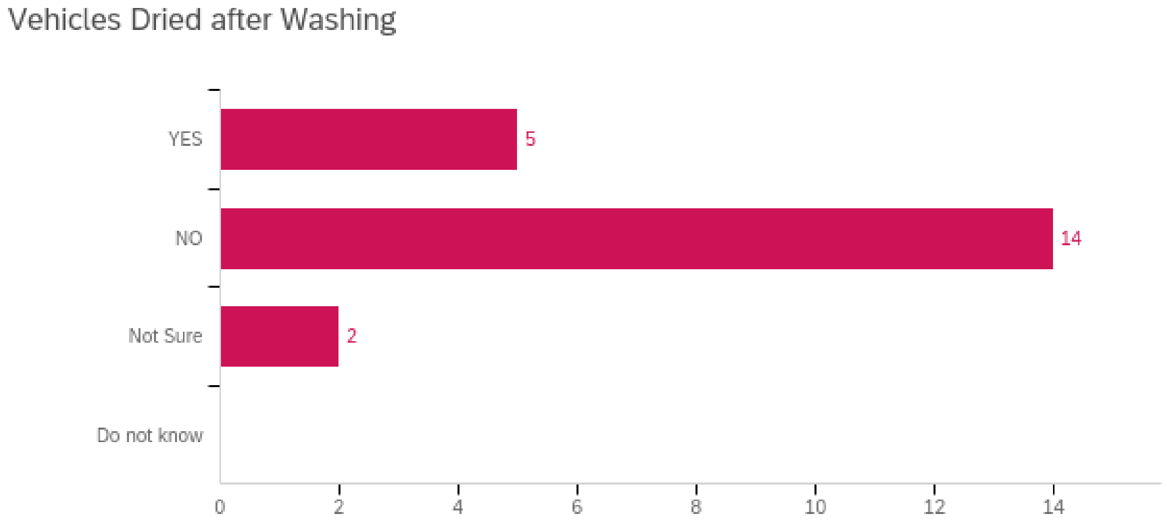
Details of washing policy (if any)	State DOT /Agency
Equipment is to be washed as soon as possible after storms subside.	Idaho Transportation Department
Vehicles are washed after every winter storm.	*Not provided*
Trucks are cleaned thoroughly after every storm	Indiana DOT
N/A	Ohio DOT
Use salt neutralizer products, use car wash soap, use high pressure hot water and scrub the equipment with a car was brush and soapy water.	East End Crossing Partners

Q. 12(e) Types of water used in the washing of vehicles (Table B 8)

Table B 8 Types of water used in the washing (Q. 12e)

Types of Water Used for Washing	State DOT / Agency
tap water	Idaho Transportation Department
Tap Water	Vermont Agency of Transportation
Tap water	Kansas DOT
Treated water from a city facility.	Ohio DOT
Most are well water. Those on City Water it is tap.	Wyoming DOT
well water, some wells are contaminated with salt.	New Hampshire DOT
tap water	Massachusetts DOT
tap water	*Not provided*
N/A	West Virginia Department of highways Equipment Division
Tap	*Not provided*
tap water.	Idaho Transportation Department
City water and well water depending on availability.	Idaho Transportation Department
tap water	Unknown (Blank)
tap water	Indiana DOT
Depending on the facility, we use public treated water and well water.	Ohio DOT
Tap water	East End Crossing Partners

Q. 13 Drying of vehicles after washed or exposed to snow/rain



Q. 13c Why drying is not done? (Table B 9)

Table B 9 Reasons of not drying the vehicles after washed or exposed to the snow/rain (Q 13c)

Main reason for not drying agency vehicles after exposure to wet conditions?	State DOT / Agency
No method for drying available. Stored inside heated garage they may have time to dry between storm event and may not	Vermont Agency of Transportation
Time and facility	Kansas DOT
We cannot dry the areas that are most affected by corrosion. We do store most of our trucks in heated buildings that helps to dry them.	Ohio DOT
personnel time, space constraints, lack of materials	New Hampshire DOT
parked outside in below freezing conditions	Massachusetts DOT
we air dry them; we lack the facilities and time, frankly	*Not provided*
Cost	Unknown (Blank)
lack of equipment/facilities	Delaware DOT
Time	Idaho Transportation Dept.
No facility to dry, vehicles are parked inside and drip dry when possible.	Idaho Transportation Department
Too many vehicles.	Indiana DOT
We park the vehicles in a heated building where they can defrost and then dry off after use. We do not have the man power to hand dry each snow plow after use.	Ohio DOT
No time or heated vehicle storage.	Bozeman MT

Q. 15e Maintenance procedures for vehicles that do not receive any additional coatings (other than the OEM's applied paints)

Responses gathered in Table B 10

Table B 10 Details on maintenance procedures for agencies vehicles with no additional protective coatings other than the OEM paints

Maintenance practices employed to control corrosion	State DOT / Agency
fluid film at time of maintenance.	New Hampshire DOT
Regular wash and dry	*Not provided*
Inspection, washing.	*Not provided*
regular washing and drying and daily inspections	*Not provided*
regular washing	Indiana DOT
Occasional washing outside when it is above freezing.	Bozeman MT

Q. 17 Best anti-corrosion coatings that are used by your agency

Responses gathered in Table B 12, and B13

Table B 11 Coatings used that were best in terms of corrosion protection

Best in terms of corrosion protection	State DOT / Agency
Fluid Film	Vermont Agency of Transportation
Fluid film	Ohio DOT
proper sand, primer, paint	New Hampshire DOT
Fluid Film	West Virginia Department of highways Equipment Division

Table B 12 Coatings used that were best in terms of overall life of the coating

Best in terms of overall life of the coating	State DOT / Agency
Metalizing	Ohio DOT
proper sand, primer, paint	New Hampshire DOT

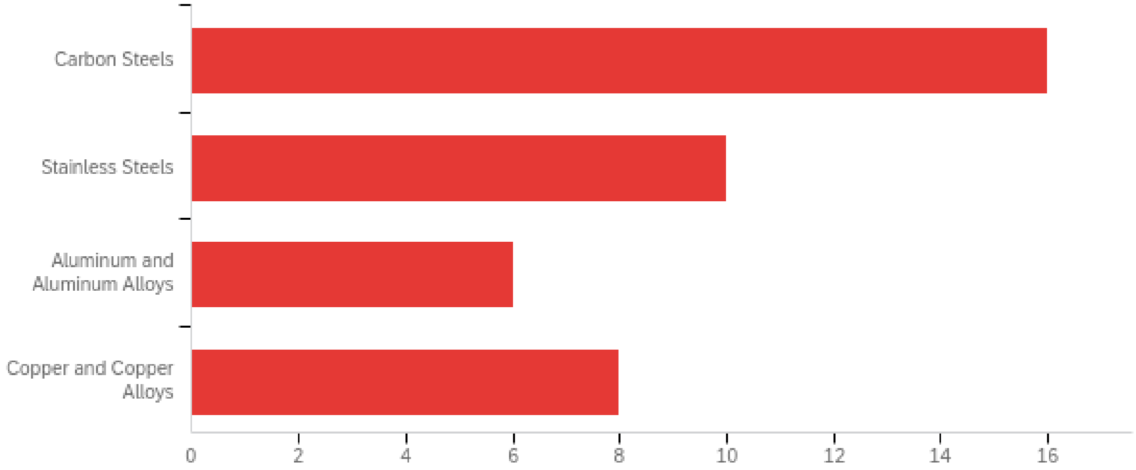
Q. 20 Lessons learned by your agency in recent years in following categories (Table B 13)

Table B 13 Lesson learned by agencies (only appropriate responses are shown)

products that worked or failed to work	modes of coating application	appropriate preparation of the metal surface	appropriate coating thickness	level of training required	cost of products	typical service life of coatings	relevant knowledge gaps
Used Linseed oil for a few years, Not effective enough for cost, Used a number of coatings over the years, Found Fluid film cost effective for the amount of protection	Air spraying and Paint gun	Wash with NUTRILIZER Air dry a few days then Spray Fluid film	*Not provided*	Min	$100-$500 PRE UNIT. $40 A GALLON PLUS Neutralizer	6 months	none
Fluid film and Crown work well for the dollars spent.	Rock gun	Clean dry surface	Varies on products.	Low	Cost of products are relatively low compared to savings in corrosion damage	Fluid film and Crown are seasonal applications.	None.
good paint is key	spray	sanding is key	think primer	professional painter	quality paint manufacturer	5 years	*Not provided*
Rhomar Neutra wash and shimmer and shine soap		sand and prime		Ongoing	*Not provided*	2 to three year for under coatings, Fluid Film is applied at least twice a year.	New employees

Market Analysis (Task 2) – Results

Q. 1 For which materials your agency uses the protective coatings?



Q. 1 Protective coatings used for various materials/alloys

Responses are given in Tables B14, B15, B16, and B17

Table B 14 Coatings used by agencies/DOTs for carbon steels

Coatings for Carbon Steels	State Agency / DOT
PetroWrap Anti Corrosion Tape	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance
over rust, oil based paint	MnDOT
Fluid Film	IDOT
Powder coating or epoxy based paints	Illinois DOT
Paint	Ohio DOT
Fluid Film	WV Department of Transportation
KENZO CERAMIC COATING FROM IGL COATINGS	Town of Lexington Public Works
We use Rhomar Armour Seal and Lubra seal	Iowa DOT
Fluid Film	MADOT
Zinc Etching Primer Plus Paint	PE Statewide
We use a product called Lubraseal from Rhomar	Utah Department of Transportation
Fluid Film	Vermont Agency of Transportation
Varies depending on the environment	AMPP
Krown Products KL73	City of West Des Moines

Coatings for Carbon Steels	State Agency / DOT
Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - Atco None of these I would recommend	WYDOT

Table B 15 Coatings used by agencies/DOTs for stainless steels

Coatings for Stainless Steels	State Agency / DOT
PetroWrap Anti Corrosion Tape	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance
KENZO CERAMIC COATING FROM IGL COATINGS	Town of Lexington Public Works
Rhomar Lubra Seal	Iowa DOT
Parker fittings stainless steel	MADOT
Krown Products KL73	City of West Des Moines
Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - Atco None of these I would recommend	WYDOT

Table B 16 Coatings used by agencies/DOTs for aluminum / aluminum alloys

Coatings for Aluminum and Aluminum Alloys	State Agency / DOT
Thermal spray Zn-Al alloys	AMPP
Oxide Coating	PE Statewide
KENZO CERAMIC COATING FROM IGL COATINGS ANF FLUID FILM	Town of Lexington Public Works
Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - Atco None of these I would recommend	WYDOT

Table B 17 Coatings used by agencies/DOTs for copper / copper alloys

Coatings for Copper and Copper Alloys	State Agency / DOT
Caig DeoxIT	IDOT
Dielectric grease on exposed terminals	PE Statewide
Dielectric silicone-based grease on battery lugs	Vermont Agency of Transportation
Wiring connectors - dielectric grease Keeps the air and salt out	WYDOT

Q. 1a Approximate costs for the protective coatings used for various materials/alloys

Responses are given in Tables B18, B19, B20, and B21

Table B 18 Approximate costs associated with the protective coatings used for various carbon steels

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for carbon steels
$3.17	5 minutes labor to install over new fittings and connectors	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance	PetroWrap Anti Corrosion Tape
I do not know.	Normal washing is all that is required in both cases	Illinois DOT	Powder coating or epoxy based paints
7.65	0	Town of Lexington Public Works	KENZO CERAMIC COATING FROM IGL COATINGS
approximately $40 per sq ft	0	Iowa DOT	We use Rhomar Armour Seal and Lubra seal
its about $100.00 per sq ft	*Not provided*	MADOT	Fluid Film
$5.00	$2.50	Karl Raschkes, PE Statewide Equipment Engineer	Zinc Etching Primer Plus Paint
$3.20	$3.20	Vermont Agency of Transportation	Fluid Film
varies	varies	AMPP	Varies depending on the environment
55gallon is $3320	*Not provided*	City of West Des Moines	Krown Products KL73

Table B 19 Approximate costs associated with the protective coatings used for stainless steels

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for stainless steels
$3.17	5 minutes labor to install over new fittings and connectors	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance	PetroWrap Anti Corrosion Tape

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for stainless steels
7.65	0	Town of Lexington Public Works	KENZO CERAMIC COATING FROM IGL COATINGS
$40 approximately per sq ft	0	Iowa DOT	Rhomar Lubra Seal
55gallon is $3320	*Not provided*	City of West Des Moines	Krown Products KL73
$3.17	5 minutes labor to install over new fittings and connectors	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance	PetroWrap Anti Corrosion Tape
7.65	0	Town of Lexington Public Works	KENZO CERAMIC COATING FROM IGL COATINGS
$40 approximately per sq ft	0	Iowa DOT	Rhomar Lubra Seal

Table B 20 Approximate costs associated with the protective coatings used for aluminum / aluminum alloys

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for aluminum / aluminum alloys
Factory applied.	*Not provided*	PE Statewide	Oxide Coating
varies	Varies	AMPP	Thermal spray Zn-Al alloys
Factory applied.	*Not provided*	PE Statewide	Oxide Coating

Table B 21 Approximate costs associated with the protective coatings used for copper / copper alloys

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for copper / copper alloys
0.25cents	0.10cents	Vermont Agency of Transportation	Dielectric silicone-based grease on battery lugs
$2.00	$2.00	PE Statewide	Dielectric grease on exposed terminals

Provide the approximate initial application cost per square foot?	Maintenance cost per square foot?	Agency / DOT	Coatings for copper / copper alloys
Very Little	Very Little	WYDOT	Wiring connectors - dielectric grease Keeps the air and salt out

Q. 2 Four coatings that worked best against brine-laden environments. Indicate experience with other exposure conditions that are cold vs hot, dry vs wet, abrasion by sand, and likewise. Responses are given in Table B 22

Table B 22 Name of 4 coatings that worked best against brine-laden environments and in other conditions

1	2	3	4	Agency / DOT
over rust - works well to extend life of material. holds up fine in all conditions except abrasive areas. works best when painted over	oil based paint - works well to extend life of material. holds up fine in all conditions except abrasive areas.	*Not provided*	*Not provided*	MnDOT
Fluid film protects against steel oxidation.works well in cold and hot environments	Works well against brines	works well against salt solutions	*Not provided*	MADOT
We paint our truck frames and cabs with a primer and automotive paint. I believe they apply an additional coating of another material on the frames, but I am not sure what it is.	*Not provided*	*Not provided*	*Not provided*	Ohio DOT
Cold climate	Hot climate	Salt exposure	Wet climate	Illinois DOT

1	2	3	4	Agency / DOT
STILL EVALUATING IT AS IT'S ONLY BEEN ON THE EQUIPMENT FOR ONE YEAR	FLUID FILM WORKS WELL ON HYDRAULIC FITTINGS, BUT REQUIRES WEEKLY APPLICATIONS	*Not provided*	*Not provided*	Town of Lexington Public Works
Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	WYDOT
Fluid Film requires consistent reapplication and will wash off withing 6 months during winter months. We have gone to an application of twice a year reapplication. Fluid film does stay applied throughout the summer months.	Dialectic grease is applied labially to battery terminals to prevent corrosion. During battery change out or signs of corrosion. Reapplication is minimal if applied correctly the first time. We also applied Dielectric to all electrical connections including starter terminals when placing units into service and yearly PMs.	*Not provided*	*Not provided*	Vermont Agency of Transportation
Primer/Paint	Cold vs Hot: High heat can cause the dielectric to run.	Dry versus wet: Wet is more of an issue.	Abrasion: Removes the protective coatings	PE Statewide

1	2	3	4	Agency / DOT
fluid film works good for chassis components and attachments. Can be washed off and reapplied.	Caig DeoxIT is good for electrical connections, reduces oxidation and leaves light protective coating.	*Not provided*	*Not provided*	IDOT
KL works fine for both mild steel and stainless. Applied in 50-60degree temps, stays on 4-6 months as long as you dont use soaps with detergents in them	*Not provided*	*Not provided*	*Not provided*	City of West Des Moines
Spray on works in all climate.	*Not provided*	*Not provided*	*Not provided*	WV Department of Transportation
We apply these products in a couple locations, it works well against salt solutions	*Not provided*	*Not provided*	*Not provided*	Utah Department Of Transportation
The armour seal works well if the surface is prepped correctly. the coating will need to be re applied annually	*Not provided*	*Not provided*	*Not provided*	Iowa DOT
I am really too far removed from project estimating. I haven't done that for several decades.	*Not provided*	*Not provided*	*Not provided*	AMPP
PetroWrap Anti Corrosion Tape works well when installed correctly. Once installed over hydraulic	*Not provided*	*Not provided*	*Not provided*	Rhode Island Department of Transportation Division of Highway &

1	**2**	**3**	**4**	**Agency / DOT**
fitting ends and hose ends it seems to protect from corrosion indefinitely or until removed.				Bridge Maintenance
over rust - works well to extend life of material. holds up fine in all conditions except abrasive areas. works best when painted over	oil based paint - works well to extend life of material. holds up fine in all conditions except abrasive areas.	*Not provided*	*Not provided*	MnDOT
Fluid film protects against steel oxidation.works well in cold and hot environments	Works well against brines	works well against salt solutions	*Not provided*	MADOT
We paint our truck frames and cabs with a primer and automotive paint. I believe they apply an additional coating of another material on the frames, but I am not sure what it is.	*Not provided*	*Not provided*	*Not provided*	Ohio DOT
Cold climate	Hot climate	Salt exposure	Wet climate	Illinois DOT
STILL EVALUATING IT AS IT'S ONLY BEEN ON THE EQUIPMENT FOR ONE YEAR	FLUID FILM WORKS WELL ON HYDRAULIC FITTINGS, BUT REQUIRES WEEKLY APPLICATIONS	*Not provided*	*Not provided*	Town of Lexington Public Works
Dielectric grease works best for the	Dielectric grease works best for the	Dielectric grease works best for the	Dielectric grease works best for the	WYDOT

1	**2**	**3**	**4**	**Agency / DOT**
application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	
Fluid Film requires consistent reapplication and will wash off withing 6 months during winter months. We have gone to an application of twice a year reapplication. Fluid film does stay applied throughout the summer months.	Dialectic grease is applied labially to battery terminals to prevent corrosion. During battery change out or signs of corrosion. Reapplication is minimal if applied correctly the first time. We also applied Dielectric to all electrical connections including starter terminals when placing units into service and yearly PMs.	*Not provided*	*Not provided*	Vermont Agency of Transportation
Primer/Paint	Cold vs Hot: High heat can cause the dielectric to run.	Dry versus wet: Wet is more of an issue.	Abrasion: Removes the protective coatings	PE Statewide
fluid film works good for chassis components and attachments. Can be washed off and reapplied.	Caig DeoxIT is good for electrical connections, reduces oxidation and leaves light protective coating.	*Not provided*	*Not provided*	IDOT
KL works fine for both mild steel and stainless. Applied in 50-60degree temps,	*Not provided*	*Not provided*	*Not provided*	City of West Des Moines

1	2	3	4	Agency / DOT
stays on 4-6 months as long as you dont use soaps with detergents in them				
Spray on works in all climate.	*Not provided*	*Not provided*	*Not provided*	WV Department of Transportation
We apply these products in a couple locations, it works well against salt solutions	*Not provided*	*Not provided*	*Not provided*	Utah Department Of Transportation
The armour seal works well if the surface is prepped correctly. the coating will need to be re applied annually	*Not provided*	*Not provided*	*Not provided*	Iowa DOT
I am really too far removed from project estimating. I haven't done that for several decades.	*Not provided*	*Not provided*	*Not provided*	AMPP
PetroWrap Anti Corrosion Tape works well when installed correctly. Once installed over hydraulic fitting ends and hose ends it seems to protect from corrosion indefinitely or until removed.	*Not provided*	*Not provided*	*Not provided*	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance
over rust - works well to extend life of material. holds up fine in all conditions except	oil based paint - works well to extend life of material. holds up fine in all	*Not provided*	*Not provided*	MnDOT

1	2	3	4	Agency / DOT
abrasive areas. works best when painted over	conditions except abrasive areas.			
Fluid film protects against steel oxidation.works well in cold and hot environments	Works well against brines	works well against salt solutions	*Not provided*	MADOT
We paint our truck frames and cabs with a primer and automotive paint. I believe they apply an additional coating of another material on the frames, but I am not sure what it is.	*Not provided*	*Not provided*	*Not provided*	Ohio DOT
Cold climate	Hot climate	Salt exposure	Wet climate	Illinois DOT
STILL EVALUATING IT AS IT'S ONLY BEEN ON THE EQUIPMENT FOR ONE YEAR	FLUID FILM WORKS WELL ON HYDRAULIC FITTINGS, BUT REQUIRES WEEKLY APPLICATIONS	*Not provided*	*Not provided*	Town of Lexington Public Works
Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	Dielectric grease works best for the application stated. Amour Seal, Black Max, Lubra Seal - Rhomar Atcoat - All are easily washed foo and do not last.	WYDOT
Fluid Film requires consistent reapplication and will wash off	Dialectic grease is applied labially to battery terminals to prevent	*Not provided*	*Not provided*	Vermont Agency of Transportation

1	2	3	4	Agency / DOT
withing 6 months during winter months. We have gone to an application of twice a year reapplication. Fluid film does stay applied throughout the summer months.	corrosion. During battery change out or signs of corrosion. Reapplication is minimal if applied correctly the first time. We also applied Dielectric to all electrical connections including starter terminals when placing units into service and yearly PMs.			
Primer/Paint	Cold vs Hot: High heat can cause the dielectric to run.	Dry versus wet: Wet is more of an issue.	Abrasion: Removes the protective coatings	PE Statewide
fluid film works good for chassis components and attachments. Can be washed off and reapplied.	Caig DeoxIT is good for electrical connections, reduces oxidation and leaves light protective coating.	*Not provided*	*Not provided*	IDOT
KL works fine for both mild steel and stainless. Applied in 50-60degree temps, stays on 4-6 months as long as you dont use soaps with detergents in them	*Not provided*	*Not provided*	*Not provided*	City of West Des Moines
Spray on works in all climate.	*Not provided*	*Not provided*	*Not provided*	WV Department of Transportation

Q. 6 Coatings that are used in the past ten years by agencies / DOTs
Three choices (a, b, and c) are covered in Tables B23, B 24, and B 25

Table B 23 Coatings that are used by agencies / DOTs in the past ten years – Choice (a)

Choice (a)	Brand name	Agency / DOT
SALT NUTRALIZER (we use currently)	RHOMAR NUTROWASH	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance
over rust	Over-Rust	MnDOT
None	*Not provided*	Bozeman MT
Rhino Lining	*Not provided*	Illinois DOT
Fluid Film	*Not provided*	WV Department of Transportation
FLUID FILM	*Not provided*	Town of Lexington Public Works
Paint	Sherwin Willaims	Iowa DOT
We use Neutro-wash rhomar rust converter as well than spray fluid film	Rhomar	MADOT
Zinc Primer/Paint	*Not provided*	PE Statewide
Fluid Film	*Not provided*	Vermont Agency of Transportation
KL73 - Krown	*Not provided*	City of West Des Moines
Dilectric grease	Various manufacturers	WYDOT
Fluid Flim	*Not provided*	Unknown

Table B 24 Coatings that are used by agencies / DOTs in the past ten years – Choice (b)

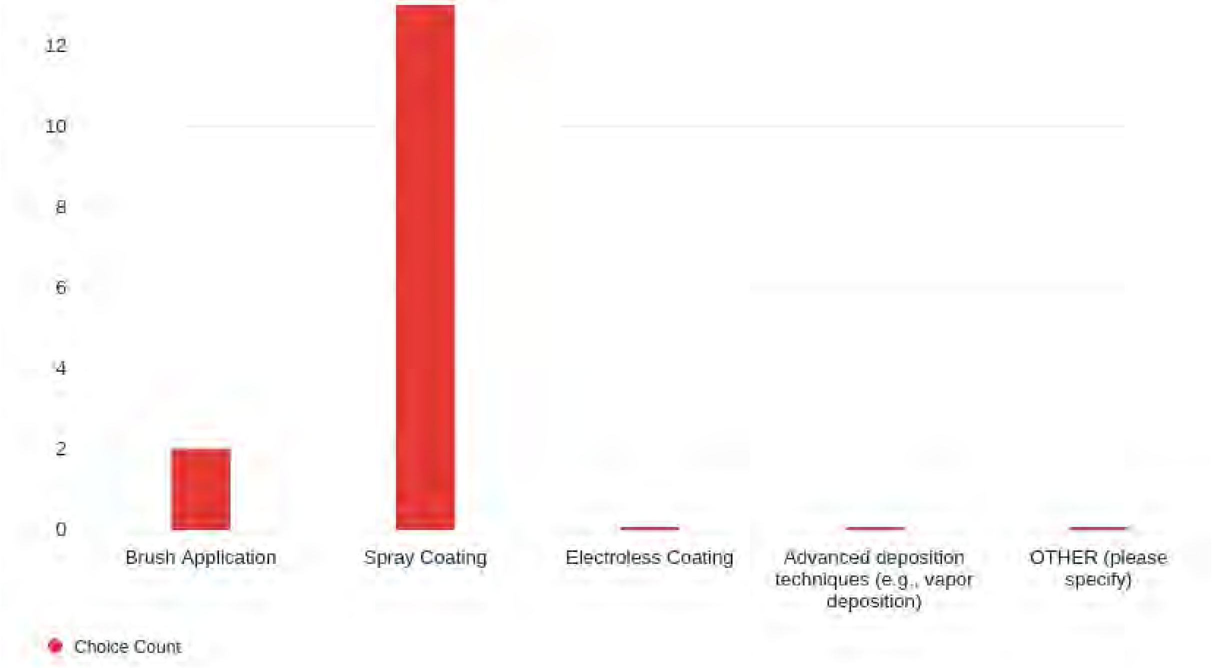
Choice (a)	Brand name	Agency / DOT
PETROWRAP (anti corrosion tape)	PETROWRAP	Rhode Island Department of Transportation
oil based paint	Sherwin Williams	MnDOT
KENZO COATING	*Not provided*	Town of Lexington Public Works
Rubberized Coating	Various brands	Iowa DOT
fluid film	fluid film	MADOT
Fluid Film	*Not provided*	PE Statewide
Nutrawash - Rhomar	*Not provided*	City of West Des Moines
Lubra Seal	Rhomar	WYDOT

Table B 25 Coatings that are used by agencies / DOTs in the past ten years – Choice (c)

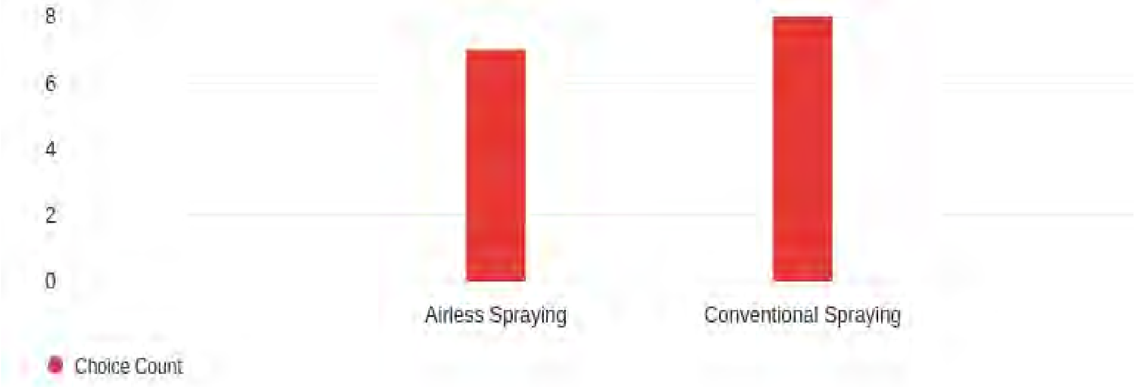
Choice (a)	Brand name	Agency / DOT
Salt Lick - Hotsy	N/A	City of West Des Moines

<u>**NOTE:** Further details on question 6 were provided in Task 2 deliverable and can be provided again electronically, on demand.</u>

Q. 9 Mode of application for the coatings in use



Q. 9(a) Specifics about spray coating



Q. 10 Optimum and widely used surface preparation method, before applying protective coatings

Q. 10 OTHER

Details are given in Table B 26

Table B 26 Details about mode of application for protective coatings used (OTHER)

OTHER (please specify)	State Agency / DOT
We install PetroWrap on all new fittings and spec our trucks to come with it preinstalled.	Rhode Island Department of Transportation
needle scaler to remove large chunks of rust	MnDOT
Steam Cleaning	WV Department of Transportation
BUFFED INTO THE PAINT	Town of Lexington Public Works
Water washing	Utah Department Of Transportation

Q. 12 Coatings recommended by agencies for specific purposes – from past ten years of experience

Details are provided in Table B 27

Table B 27 Coatings recommended for specific purposes – from past ten years of experience

Last Longer	Easy to apply	Most effective	State Agency / DOT
PetroWrap seems to last untill removed	Yes	Yes	Rhode Island Department of Transportation Division of Highway & Bridge Maintenance
fluid film, DeoxIT	fluid film, DeoxIT	fluid film, DeoxIT	IDOT
https://nhoilundercoating.com/	Fluid Film		Illinois DOT

Last Longer	**Easy to apply**	**Most effective**	**State Agency / DOT**
Automotive paint	Nu Film	Automotive paint	Ohio DOT
Fluid Film	*Not provided*	*Not provided*	WV Department of Transportation
Ceramic Coatings last 3 years	Fluid Film	Ceramic Coatinngs	Town of Lexington Public Works
Fluid Film	Fluid film	Fluid Film	Iowa DOT
fluid film and lubra seal	*Not provided*	*Not provided*	MADOT
It usually last's a season	It is sprayed on with a weed sprayer	Lubraseal	Utah Department Of Transportation
Not provided	Fluid Film	Equal	Vermont Agency of Transportation
None	Pump sprayer works well on.	None	WYDOT

Appendix C

Tables & Figures – From all Chapters

Table C 1 Ranks given to coatings based on the number of times (superscripts) they were indicated for any given material (from Q.1 of Task 2)

Ranks for coatings	**Coatings for Materials**			
	Stainless Steels	**Carbon Steels**	**Aluminum & Al Alloys**	**Copper & Cu Alloys**
1	Lubra Seal^{2} (by Rhomar)	Fluid Film^{4}	i. Kenzo Ceramic Coating^{1} (by IGL) ii. Fluid Film^{1} iii. Oxide Coating^{1} iv. Thermal spray Zn-Al Alloys^{1} v. Lubra Seal^{1} (by Rhomar) vi. Armour Seal^{1} vii. Black Max^{1} viii. Rhomar Atcoat^{1}	i. Dielectric Grease^{3}

Ranks for coatings	**Coatings for Materials**			
	Stainless Steels	**Carbon Steels**	**Aluminum & Al Alloys**	**Copper & Cu Alloys**
2	i. PetroWrap Anti Corrosion Tape[1] ii. Parker fittings stainless steel[1] iii. KL73[1] (by Krown) iv. Armour Seal[1] v. Black Max[1] vi. Rhomar Atcoat[1] vii. Kenzo Ceramic Coating[1] (by IGL)	Lubra Seal[3]	X	i. Caig DeoxIT[1]
3	X	PAINTS[3] i. oil-based paint, ii. epoxy-based paints, iii. paint	X	X
4	X	Armour Seal[2] (by Rhomar)	X	X

Ranks for coatings	Coatings for Materials			
	Stainless Steels	**Carbon Steels**	**Aluminum & Al Alloys**	**Copper & Cu Alloys**
5		i. Powder coating^{2} ii. Over Rust^{1} iii. Kenzo Ceramic Coating^{1} (IGL Coatings) iv. Zinc Etching Primer plus Paint^{1} v. KL73^{1} by Krown, vi. Black Max^{1} vii. Rhomar Atcoat^{1} viii. PetroWrap Anti Corrosion Tape^{1}		
6		Varies depending on the environment		

The numbers in the superscripts on a coating indicate the number of times it was mentioned for any given material.
Coating's rank is given based on the number of recurring responses, that are given as the superscripts.
Highlighted coatings were initially remained in for the selection phase.

Table C 2 Ranks given to coatings based on the number of times (superscripts) they were indicated by respondents, for their performance (from Q. 2, Task 2)

Ranks for coatings	Best coatings for various climates and against brine			
	Salty Conditions	Hot Climate	Cold Climate	Wet Climate
1	**Fluid Film^{4}	Fluid Film^{2}	i. Fluid Film^{1} (no info on wet climate) ii. [a]Primer/Paint^{1} (not suitable in wet climate) iii. [a]Oil-based paints^{1} iv. [a]Over Rust^{1}	i. Oil-based paints^{1} ii. Over Rust^{1}
2	i. [a]Primer/Paint^{2} ii. *Dielectric Grease^{2} ** (not suitable in hot climate)	i. Primer/Paint^{1} ii. Oil-based paints^{1} iii. Over Rust^{1} iv. Caig DeoxIT^{1} v. [c] KL^{1}	X	X
3	i. [a] Oil-based paints^{1} ii. *PetroWrap^{1} (Anti Corrosion Tape)** iii. *Caig DEox IT^{1} iv. [a] Over Rust^{1} v. ** [b] KL^{1} vi. *Armor Seal^{1} **	X	X	X

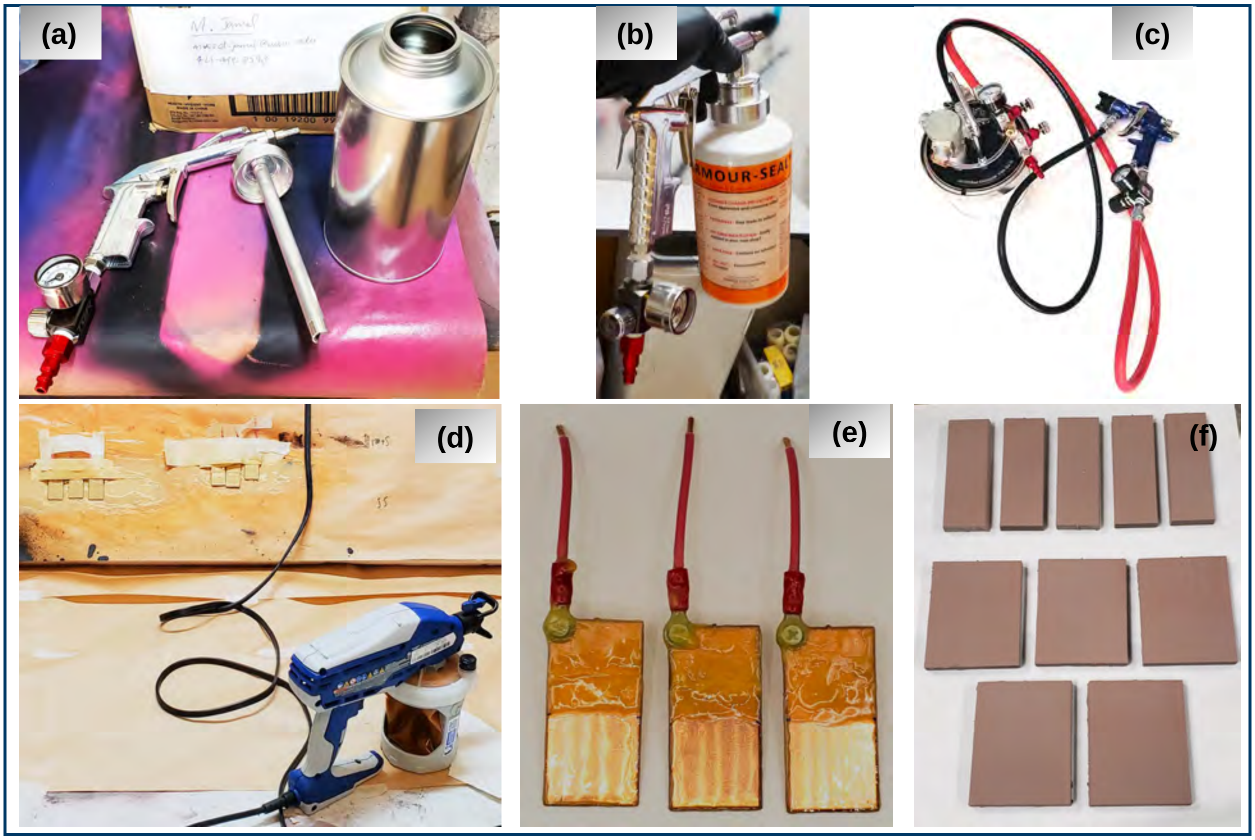


Figure C 1 Paint shop highlights: (a) pistol grip gun & a Schutz bottle for Llubra Seal (b) Armour Seal setup (c) pressure pot and conventional spray gun for Aquapon (d) airless sprayer for Fluid Film (e) Deox-IT EIS coupons (f) Aquapon – fully set

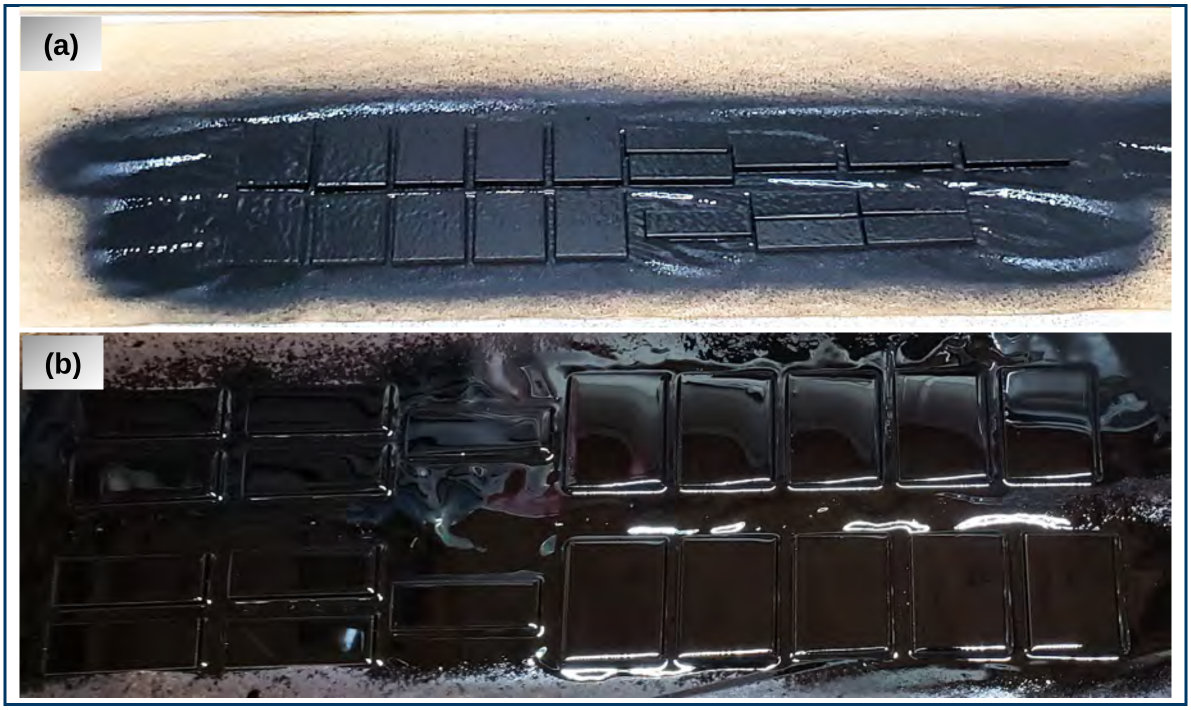


Figure C 2 Paint shop highlights: (a) Armour Seal and (b) Lubra Seal, both applied using pistol grip spray gun

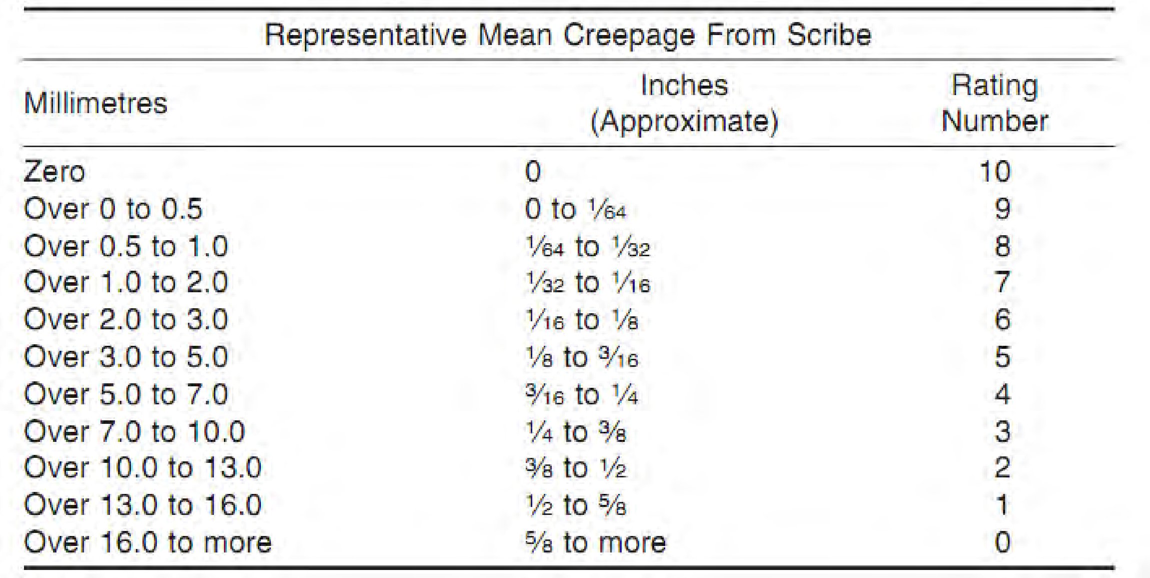
Figure C 3 Paint highlights: (a) Armour Seal dried (b) drying set up for Lubra Seal salt spray coupons

Table C 3 Chemical names and types of products selected (info is based on the SDS of the products)

No.	Product Choice	Chemical name	Type of product
1	Fluid Film	Refined Petroleum oil	Organic Lubricant
2	Armour Seal	Petroleum Hydrocarbon (Bitumen)	Organic Sealant
3	Kenzo Ceramic (base)	(i) 3-Glycidyloxypropyltrimethoxysilane (ii) Dimethyl siloxane (iii) Triethoxy-n-octylsilane	Organic Coating
4	Kenzo Ceramic (top)	(i) Polydimethylsiloxane (ii) Dipropylene glycol	Organic Coating
5	Lubra Seal	(i) Medium Aliphatic Petroleum Distillates (Stoddard Solvent) (ii) Bituminous Petroleum (Bitumen)	Organic Sealant
6	Dielectric Grease	Silicon based grease	Grease / lubricant

Figure C 4 Coatings used for all materials (a) Lubra Seal ™ and asphalt remover (b) Deox IT ® and dielectric grease (c) Armour Seal ® and Fluid Film ® (d) PPG Aquapon ®

Table C 4 Creepage rating numbers based on the mean creepage width on scribed coupons (ASTM D610-08)



Representative Mean Creepage From Scribe		
Millimetres	Inches (Approximate)	Rating Number
Zero	0	10
Over 0 to 0.5	0 to 1⁄64	9
Over 0.5 to 1.0	1⁄64 to 1⁄32	8
Over 1.0 to 2.0	1⁄32 to 1⁄16	7
Over 2.0 to 3.0	1⁄16 to ⅛	6
Over 3.0 to 5.0	⅛ to 3⁄16	5
Over 5.0 to 7.0	3⁄16 to ¼	4
Over 7.0 to 10.0	¼ to ⅜	3
Over 10.0 to 13.0	⅜ to ½	2
Over 13.0 to 16.0	½ to ⅝	1
Over 16.0 to more	⅝ to more	0

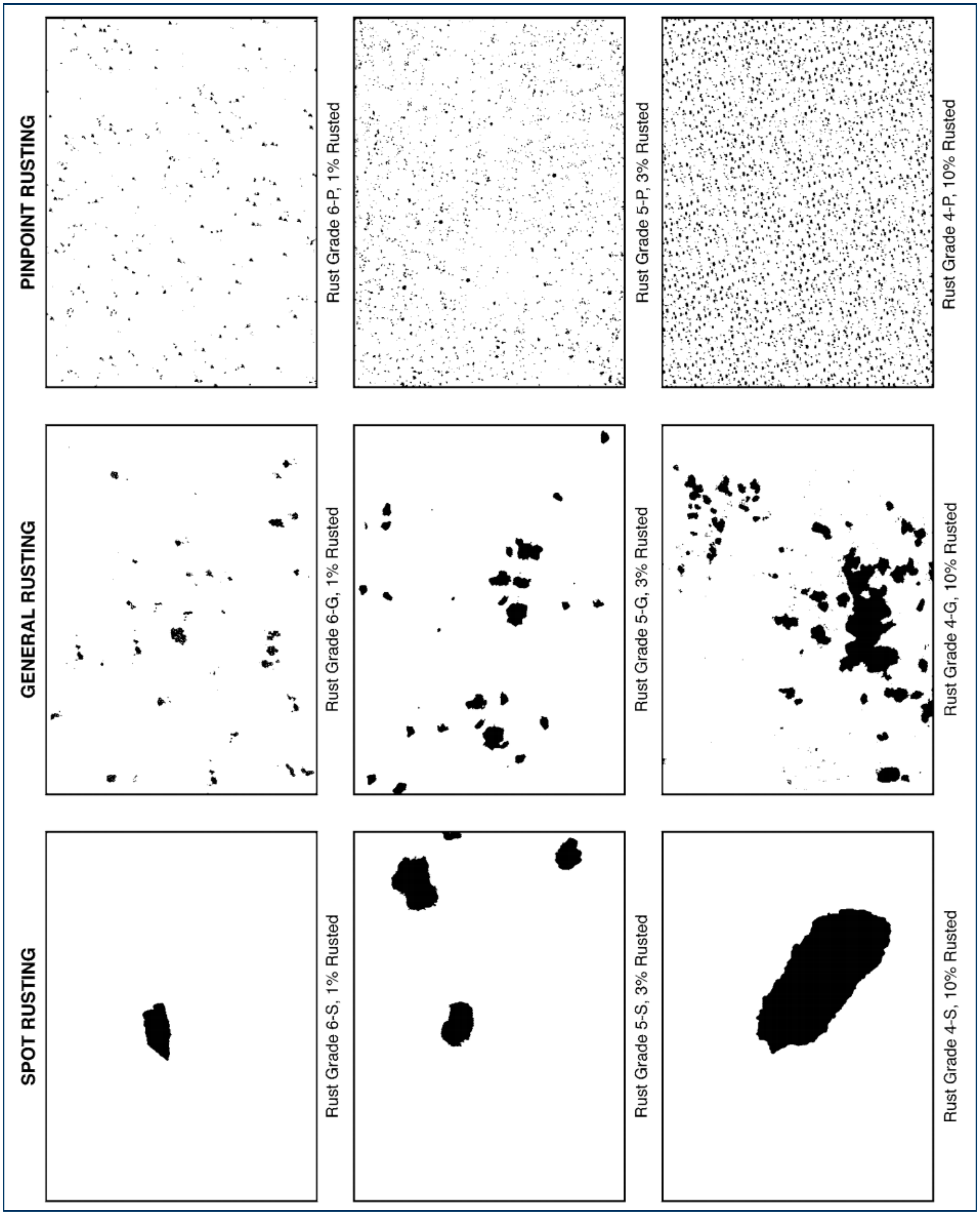
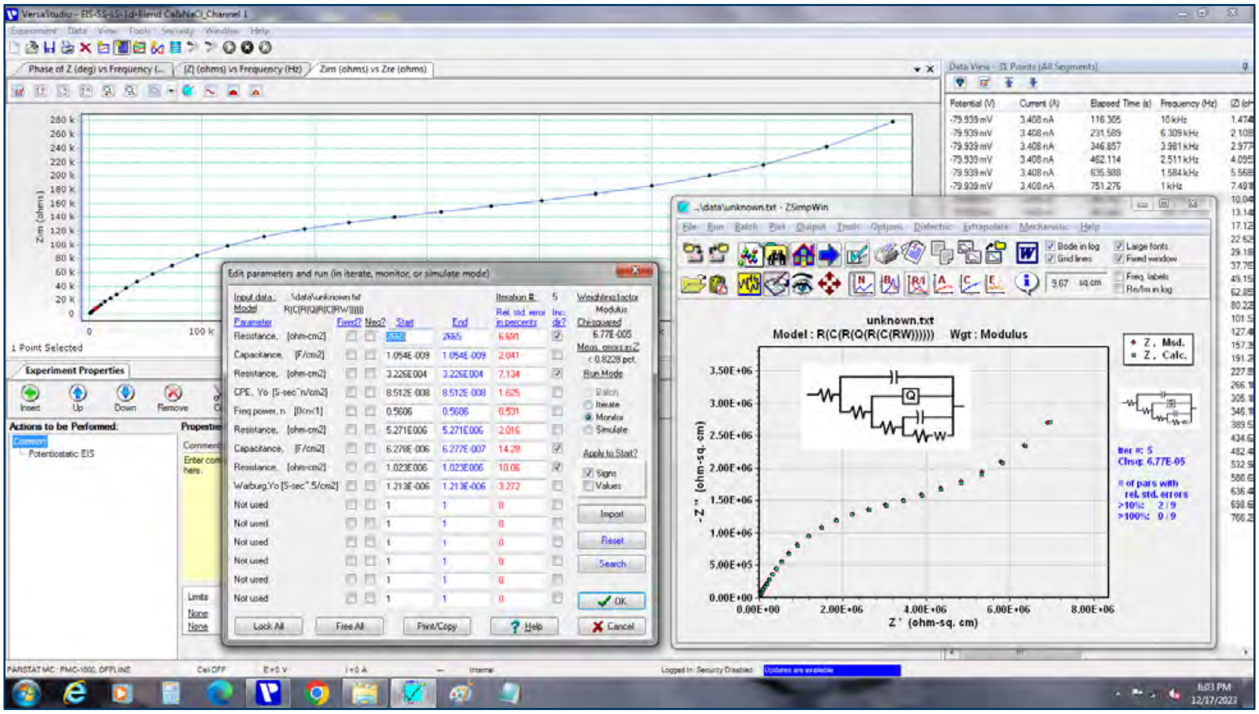


Figure C 5 A few visual examples of rust grades for Spot, General, and Pinpoint rusting (ASTM International, 2019a)



Edit parameters and run (in iterate, monitor, or simulate mode)

Input data: ..\data\unknown.txt

Model: R(C(R(Q(R(C(RW))))))

Iteration #: 5

Parameter	Fixed?	Neg?	Start	End	Rel. std. error in percents	Inc. dir?
Resistance, [ohm-cm2]	☐	☐	2665	2665	6.691	☑
Capacitance, [F/cm2]	☐	☐	1.054E-009	1.054E-009	2.041	☐
Resistance, [ohm-cm2]	☐	☐	3.226E004	3.226E004	7.134	☑
CPE, Yo [S-sec^n/cm2]	☐	☐	8.512E-008	8.512E-008	1.625	☐
Freq power, n [0<n<1]	☐	☐	0.5606	0.5606	0.531	☐
Resistance, [ohm-cm2]	☐	☐	5.271E006	5.271E006	2.016	☐
Capacitance, [F/cm2]	☐	☐	6.278E-006	6.277E-007	14.28	☑
Resistance, [ohm-cm2]	☐	☐	1.023E006	1.023E006	10.06	☑
Warburg,Yo [S-sec^.5/cm2]	☐	☐	1.213E-006	1.213E-006	3.272	☐
Not used	☐	☐	1	1	0	☐
Not used	☐	☐	1	1	0	☐
Not used	☐	☐	1	1	0	☐
Not used	☐	☐	1	1	0	☐
Not used	☐	☐	1	1	0	☐
Not used	☐	☐	1	1	0	☐

Weighting factor: Modulus

Chi-squared: 6.77E-005

Meas. errors in Z: < 0.8228 pct.

Run Mode: ○ Batch ○ Iterate ● Monitor ○ Simulate

Apply to Start? ☑ Signs ☐ Values

Data View - 31 Points (All Segments)

Potential (V)	Current (A)	Elapsed Time (s)	Frequency (Hz)
-79.939 mV	3.408 nA	116.305	10 kHz
-79.939 mV	3.408 nA	231.589	6.309 kHz
-79.939 mV	3.408 nA	346.857	3.981 kHz
-79.939 mV	3.408 nA	462.114	2.511 kHz
-79.939 mV	3.408 nA	635.988	1.584 kHz
-79.939 mV	3.408 nA	751.276	1 kHz

☑ Bode in log ☑ Grid lines ☑ Large fonts ☑ Fixed window ☐ Freq. labels ☐ Re/Im in log

9.67 sq.cm

Figure C 6 Data analysis EIS: (a) raw Nyquist plot, (b) modeled plot with circuit, and (c) parameters obtained after modeling for SS-LS-A-Day 1

EIS test wet/dry cycles – Photos

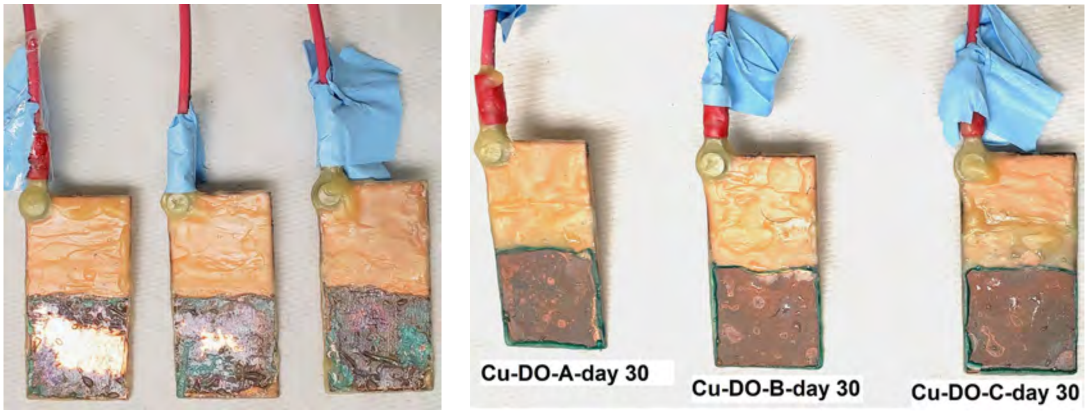


Figure C 7 EIS coupons for NaCl-CaCl$_2$ blend (a) Cu-DG after day 30 (b) Cu-DO after day 30

SAE J2334 test photos

Day 3: Photos – On the tray

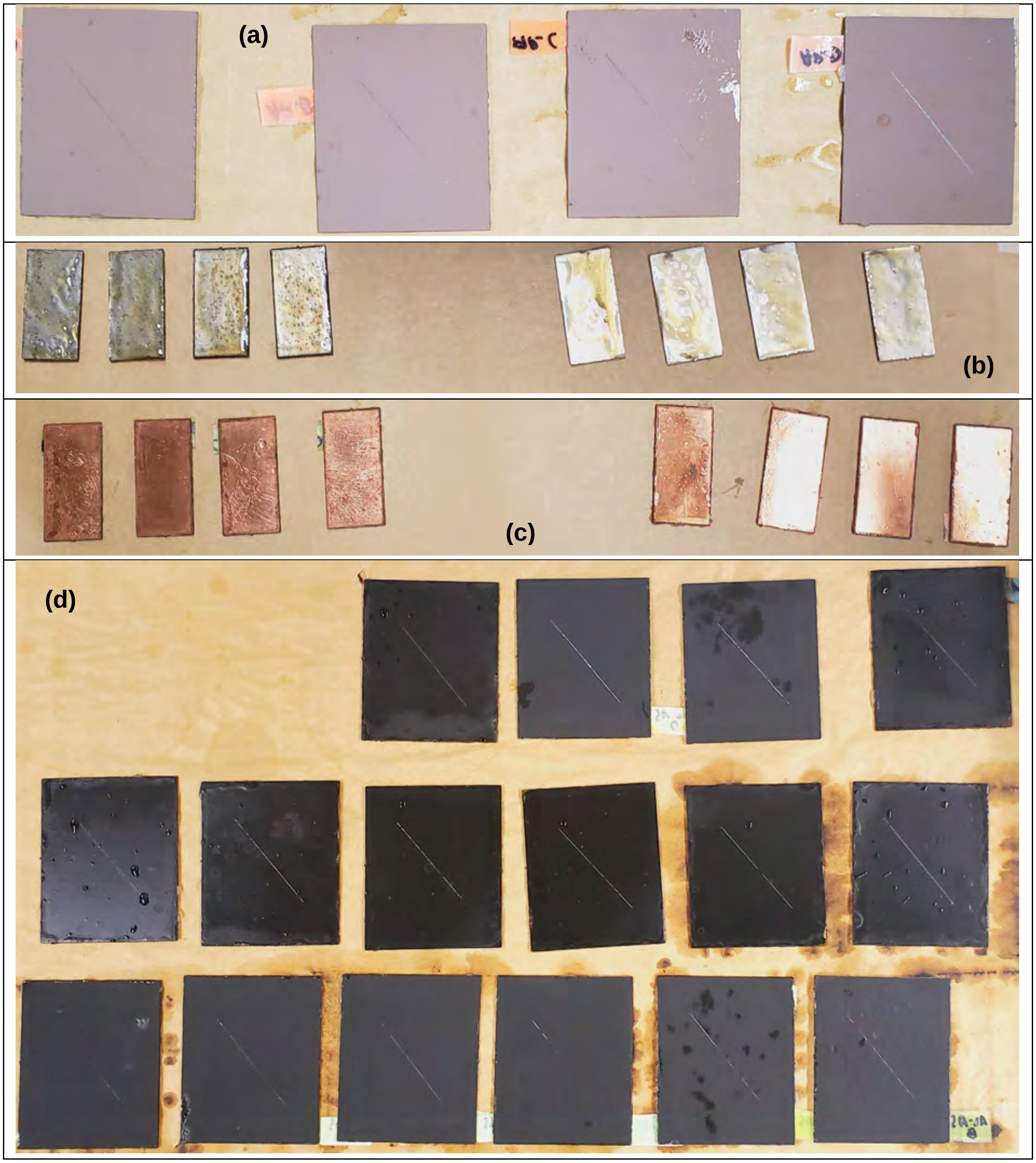


Figure C 8 Coupons for SAEJ2334 (cyclic salt spray test) – Day 1 (a) S-AP (b) S-FF (left) and Al-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, SS-AS, and Al-AS)

Day 3: Photos – On the tray

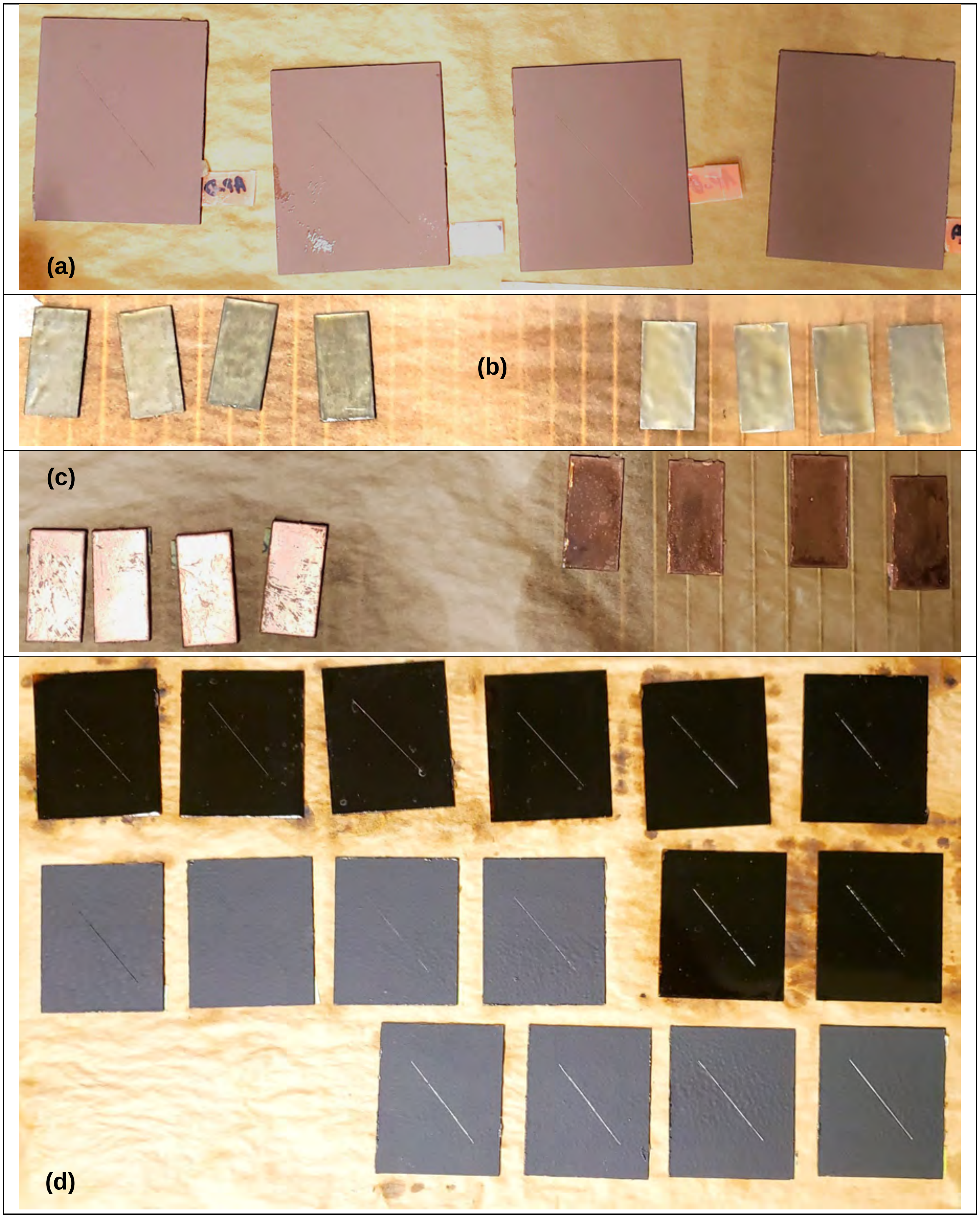


Figure C 9 Coupons for SAEJ2334 (cyclic salt spray test) – Day 3 (a) S-AP (b) S-FF (left) and Al-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, SS-AS, and Al-AS)

Day 11: Photos – On the tray

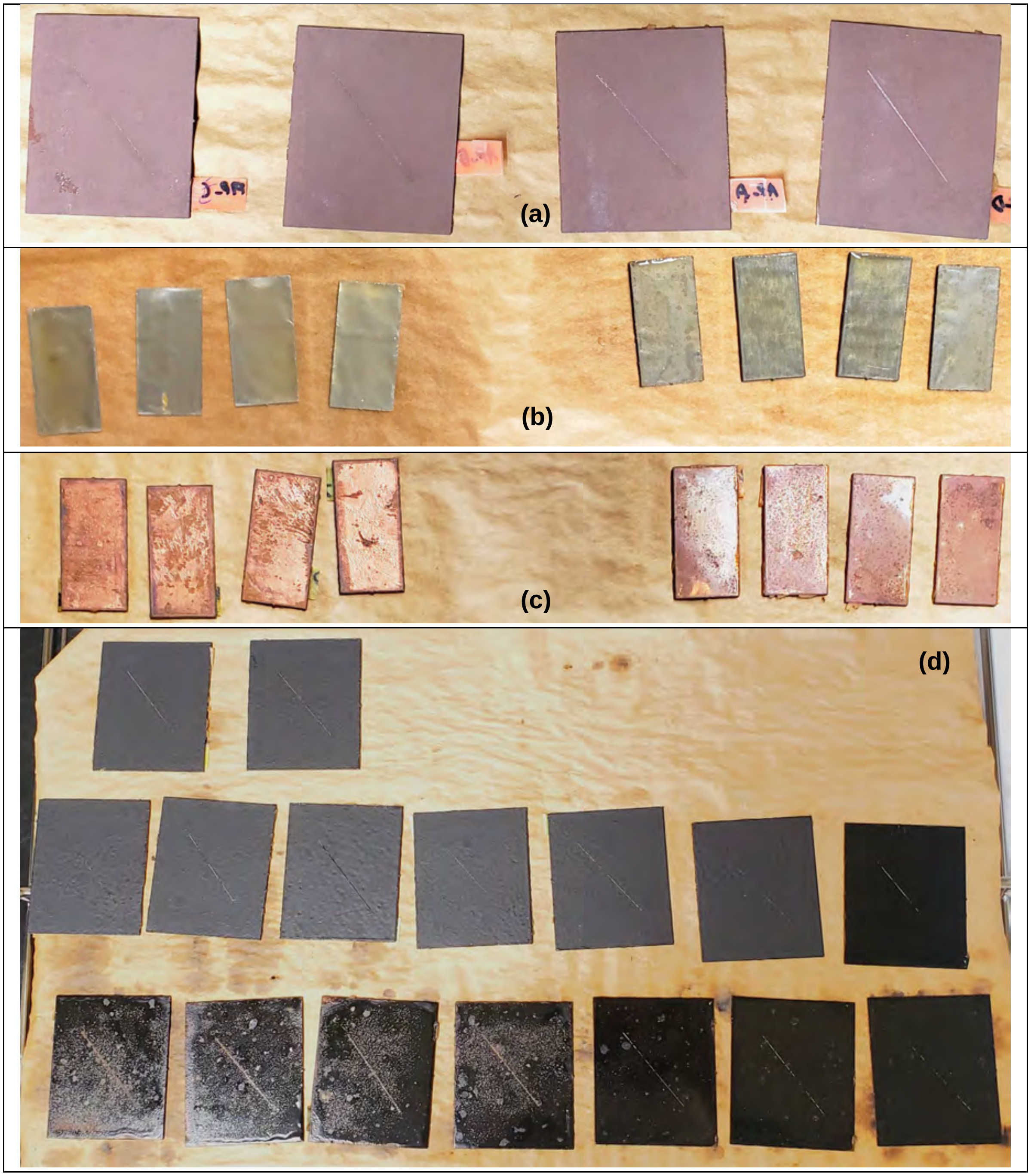


Figure C 10 Coupons for SAEJ2334 (cyclic salt spray test) – Day 11 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (SS-AS, Al-AS, S-LS, SS-LS)

Day 21: Photos – On the tray

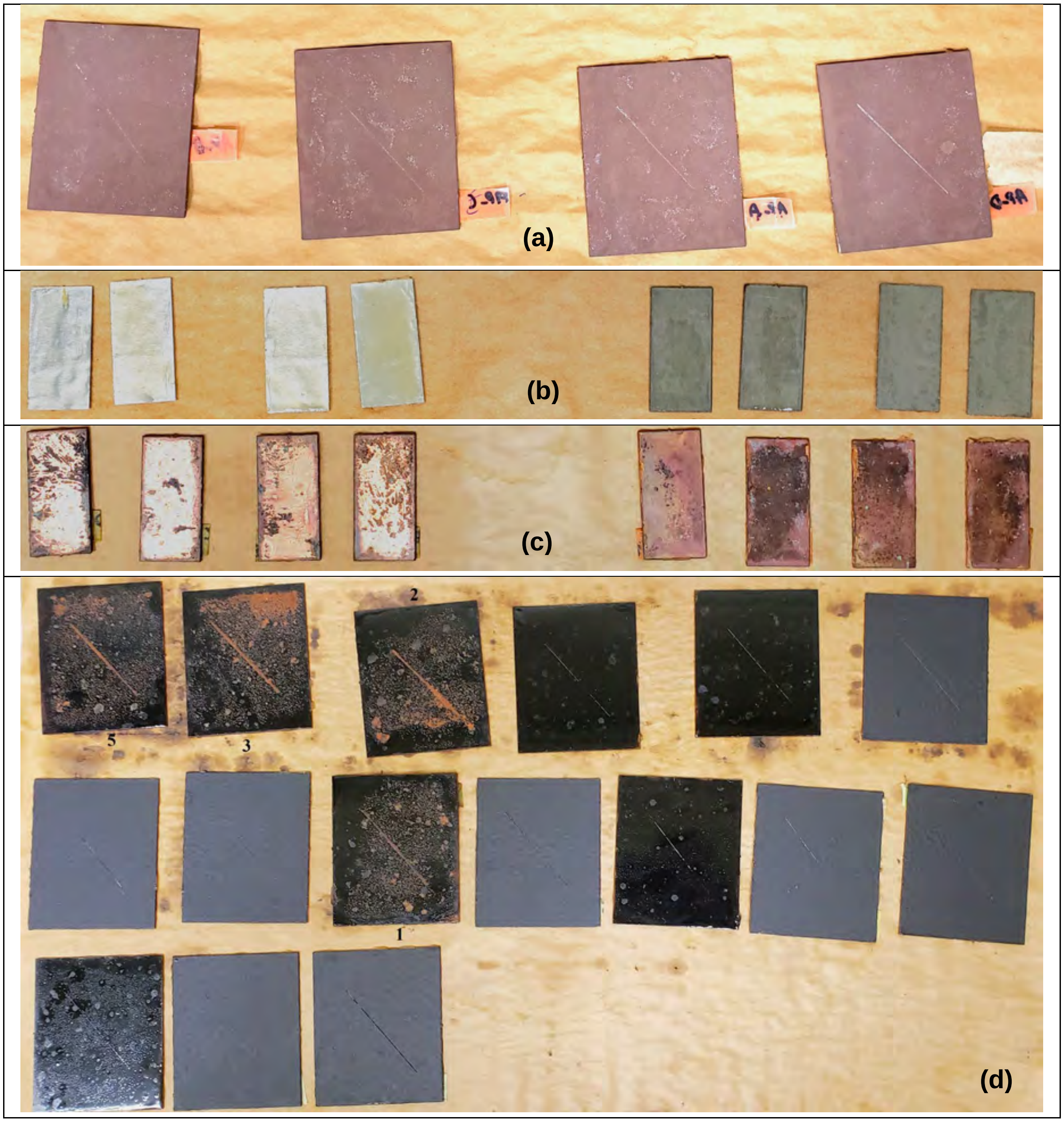


Figure C 11 Coupons for SAEJ2334 (cyclic salt spray test) – Day 21 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, Al-AS, SS-AS)

Day 31: Photos – On the tray

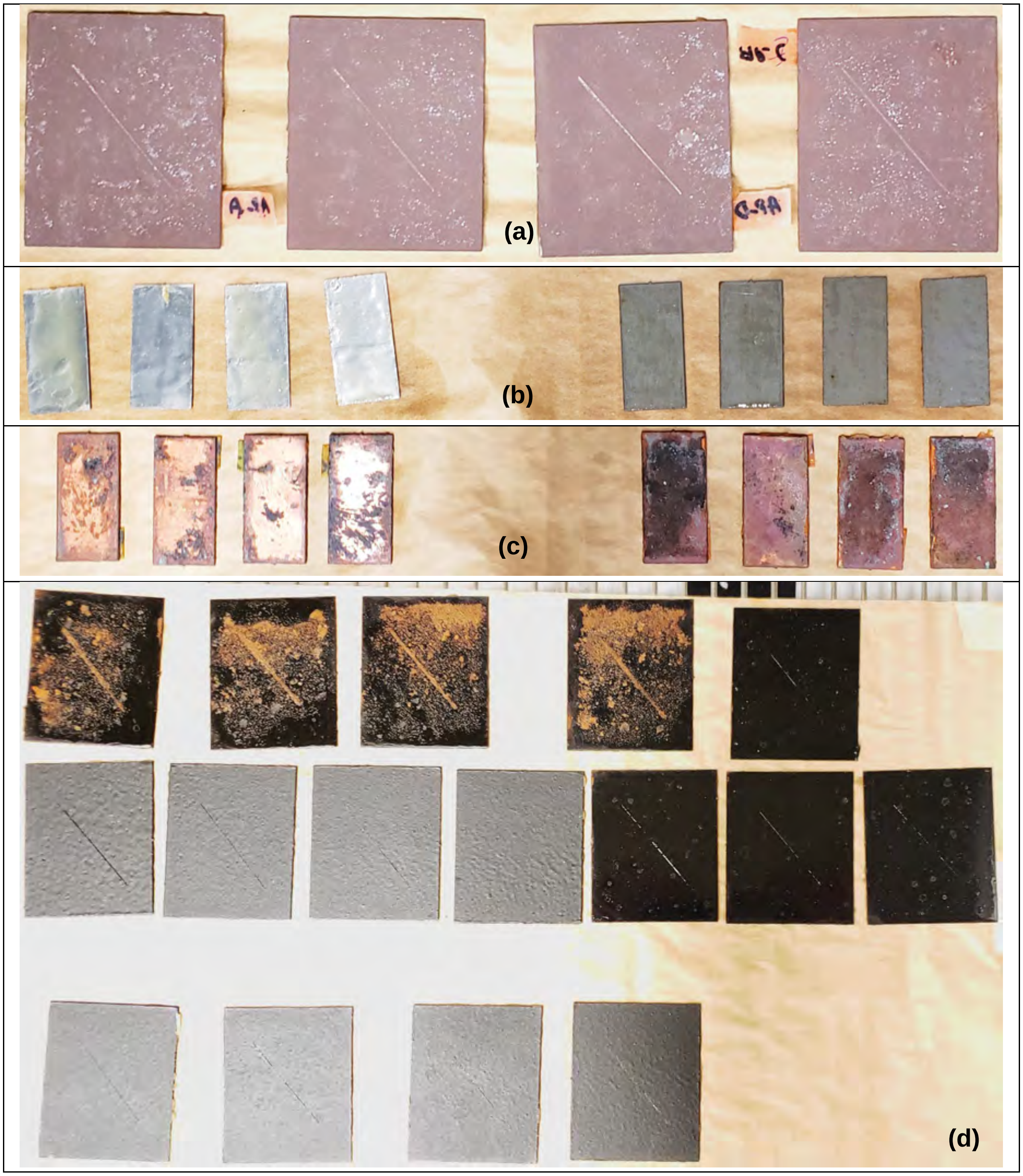


Figure C 12 Coupons for SAEJ2334 (cyclic salt spray test) – Day 31 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, Al-AS, SS-AS)

Day 41: Photos – On the tray

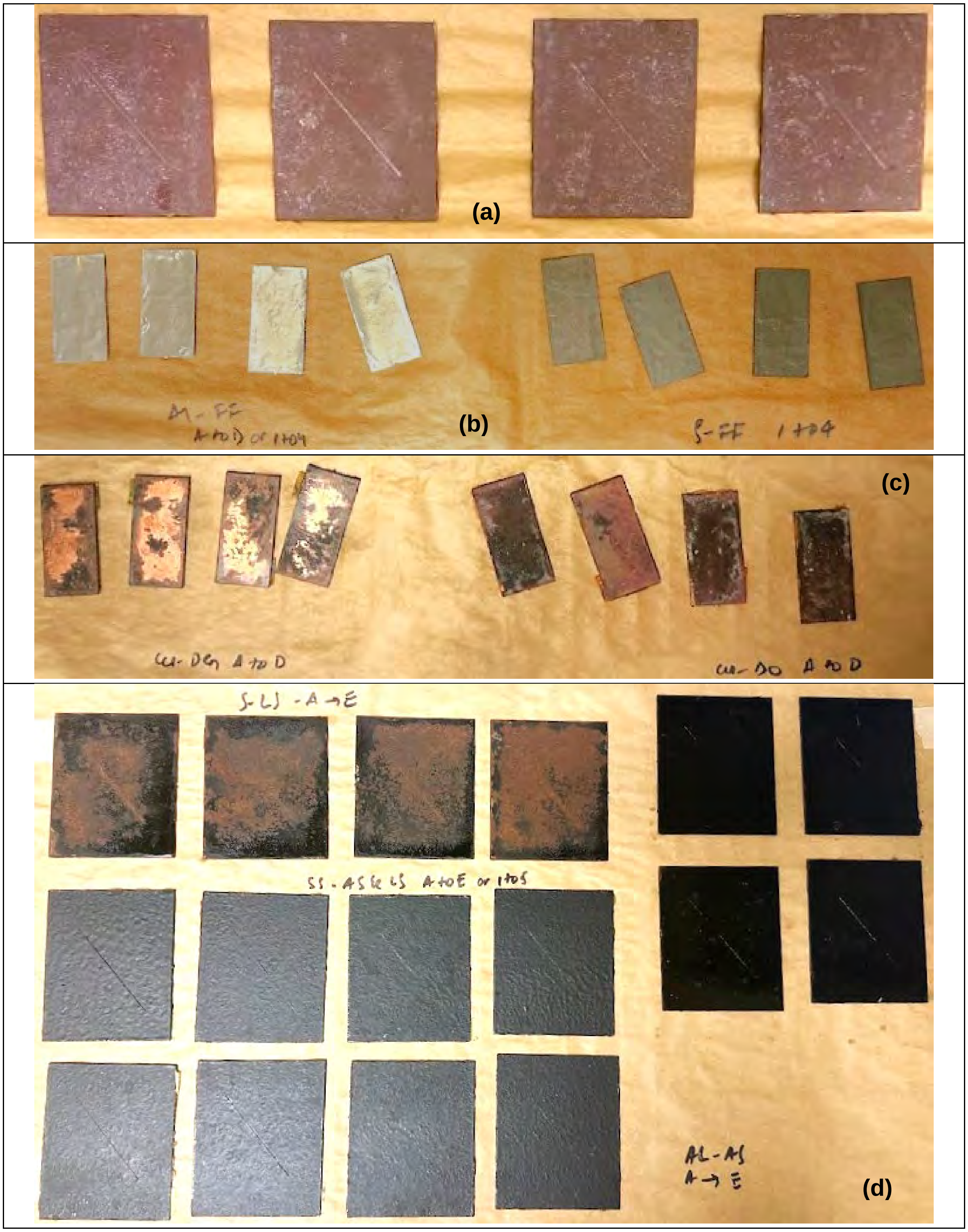


Figure C 13 Coupons for SAEJ2334 (cyclic salt spray test) – Day 41 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, Al-AS, SS-AS)

Day 50: Photos – On the tray

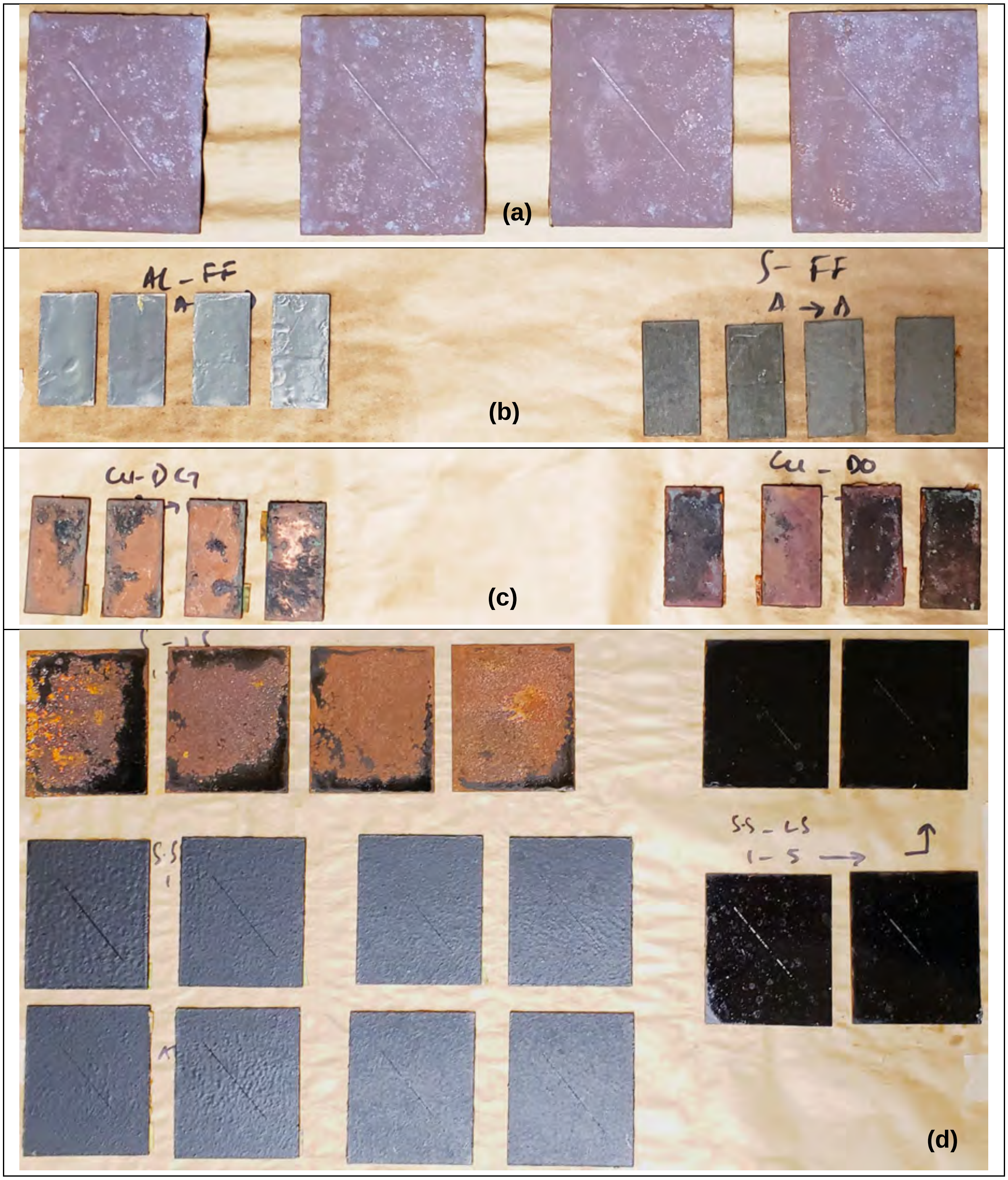


Figure C 14 Coupons for SAEJ2334 (cyclic salt spray test) – Day 51 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, SS-AS, Al-AS)

Day 60: Photos – On the tray

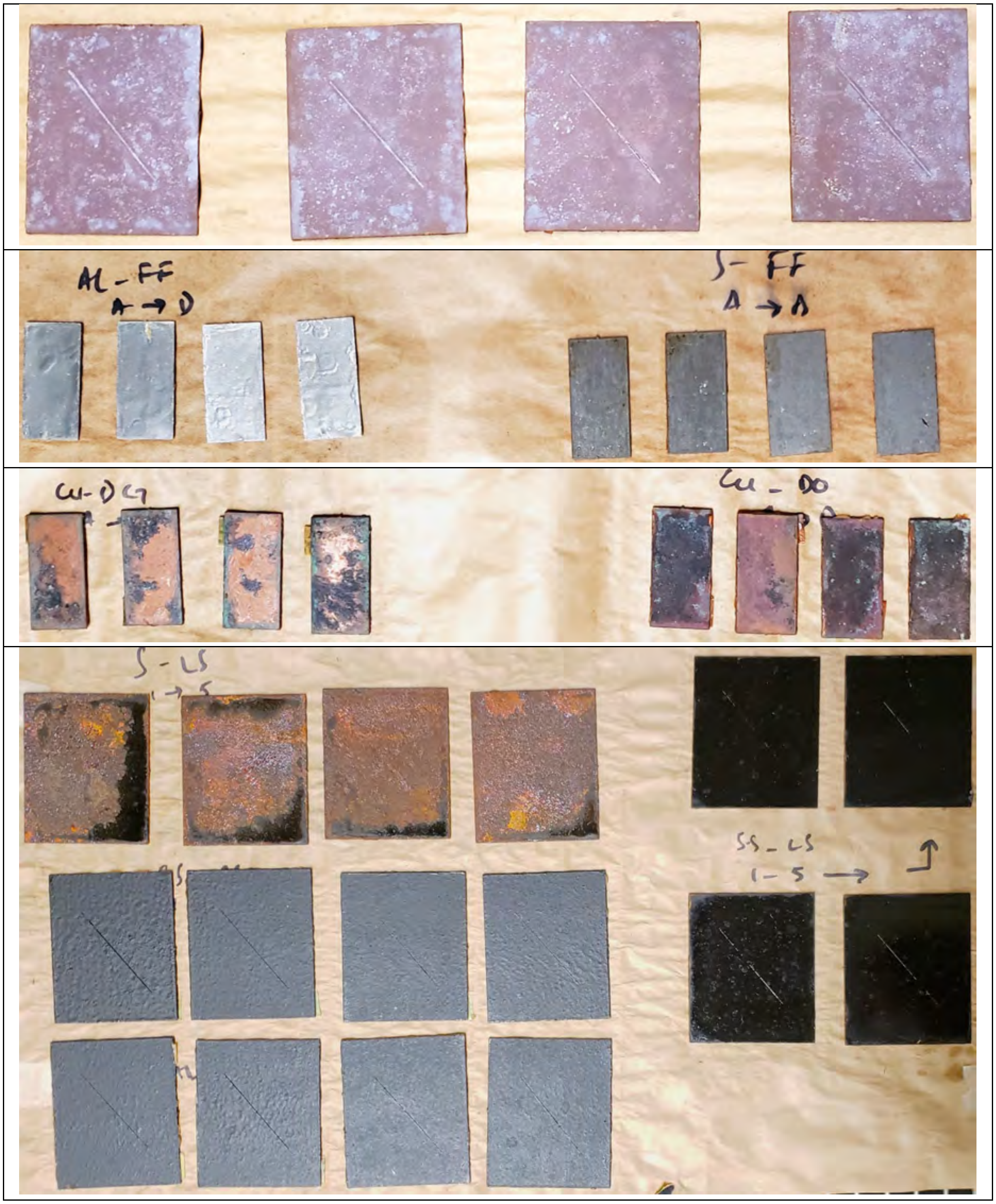


Figure C 15 Coupons for SAEJ2334 (cyclic salt spray test) – Day 60 (a) S-AP (b) Al-FF (left) and S-FF (right), (c) Cu-DG (left) and Cu-DO (right), (d) remaining scribed coupons (S-LS, SS-LS, SS-AS, Al-AS)

Day 60

SAE J2334 test photos – Inside a lightbox

The photos inside a lightbox were taken at various test intervals for more clarity and to determine the rust grades and creepage. In some cases, these images were used to observe any blistering occurring on coatings.

Day 31: Lightbox photos

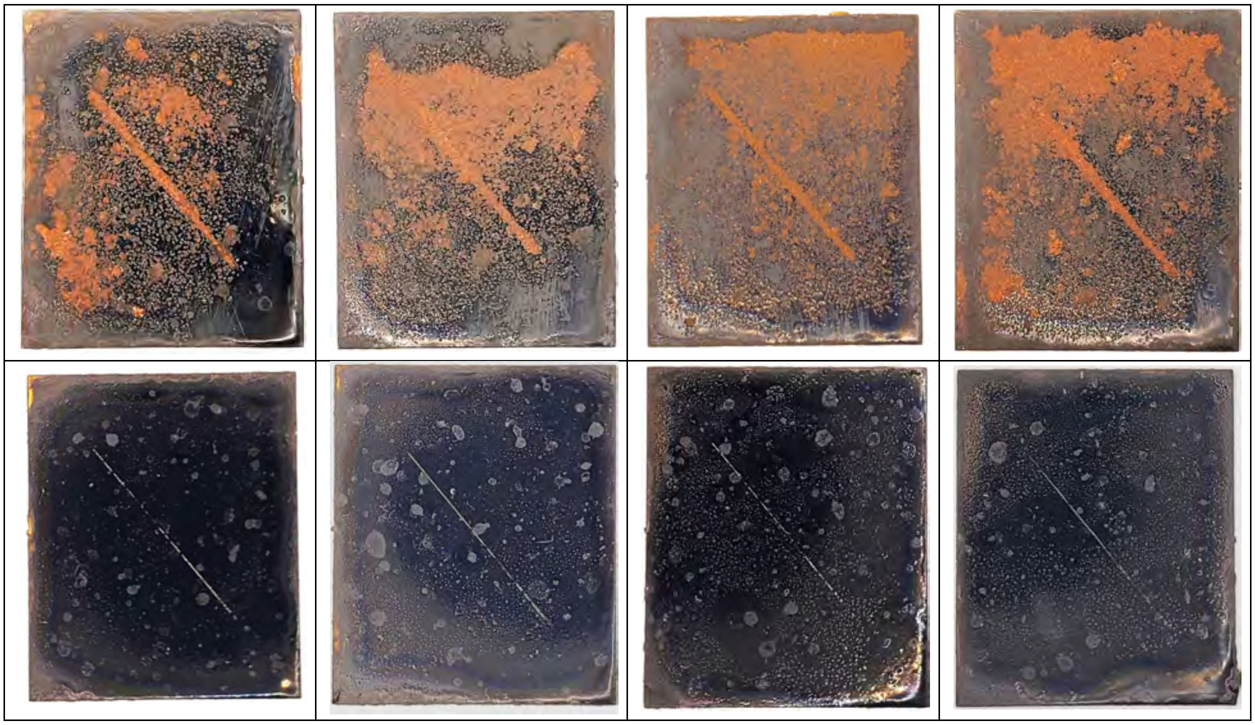


Figure C 16 Coupons for SAEJ2334 (cyclic salt spray test) – Day 31 (top) S-LS coupons after mild cleaning (bottom) SS-LS coupons

Day 43: Lightbox photos

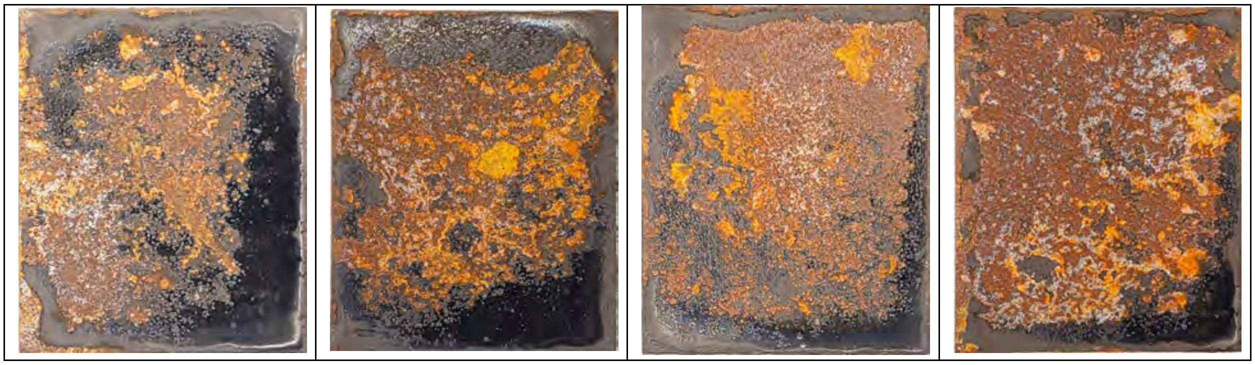


Figure C 17 Coupons for SAEJ2334 (cyclic salt spray test) – Day 43 S-LS coupons 1, 2, 3, and 5 (A, B, C, E) from left to right

Day 48: Lightbox photos

Figure C 18 Coupons for SAEJ2334 (cyclic salt spray test) – Day 48 (top) S-LS coupons (middle) Cu-DG coupons (bottom) Cu-DO coupons

Day 54: Lightbox photos

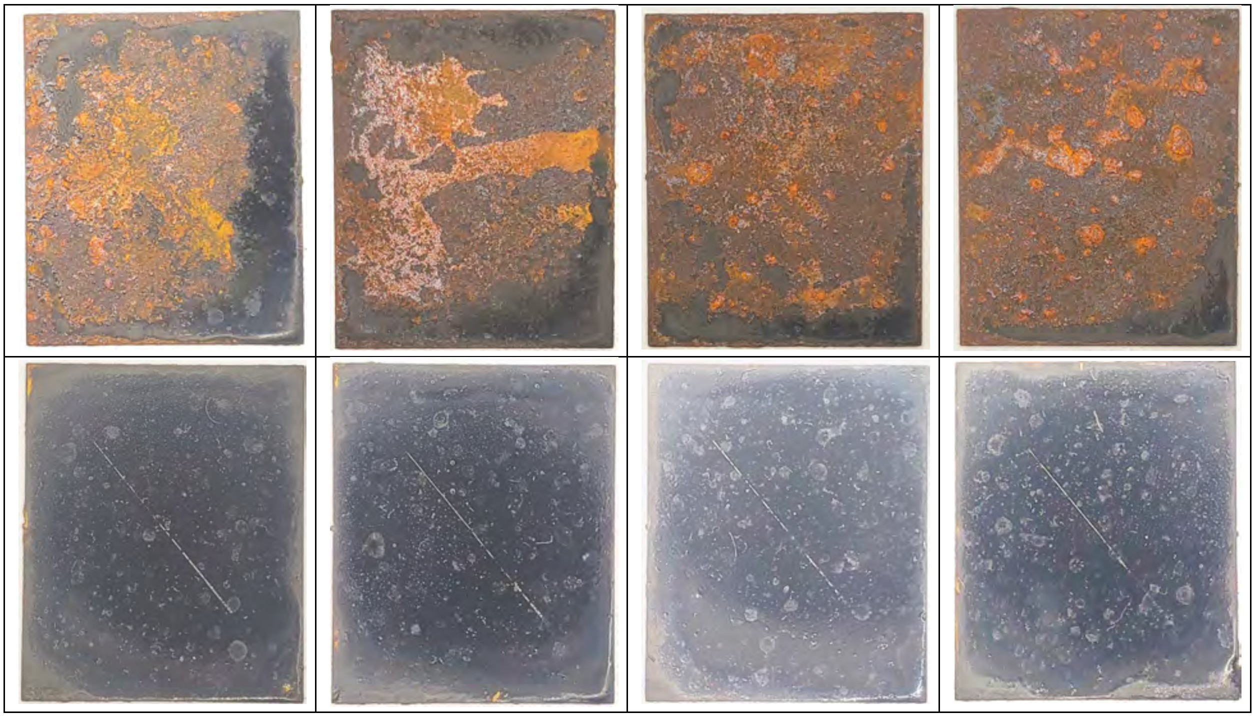


Figure C 19 Coupons for SAEJ2334 (cyclic salt spray test) – Day 54 (top) S-LS coupons (bottom) SS-LS coupons

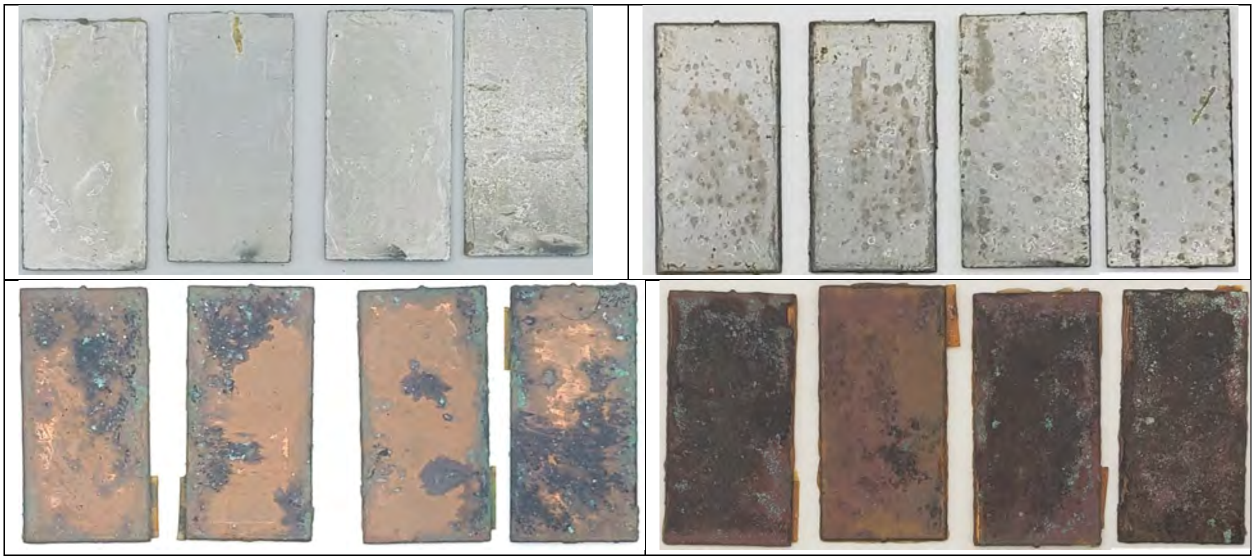


Figure C 20 Coupons for SAEJ2334 (cyclic salt spray test) – Day 54 (top) Al-FF coupons on left and S-FF coupons on right (bottom) Cu-DG coupons on left and Cu-DO coupons on the right

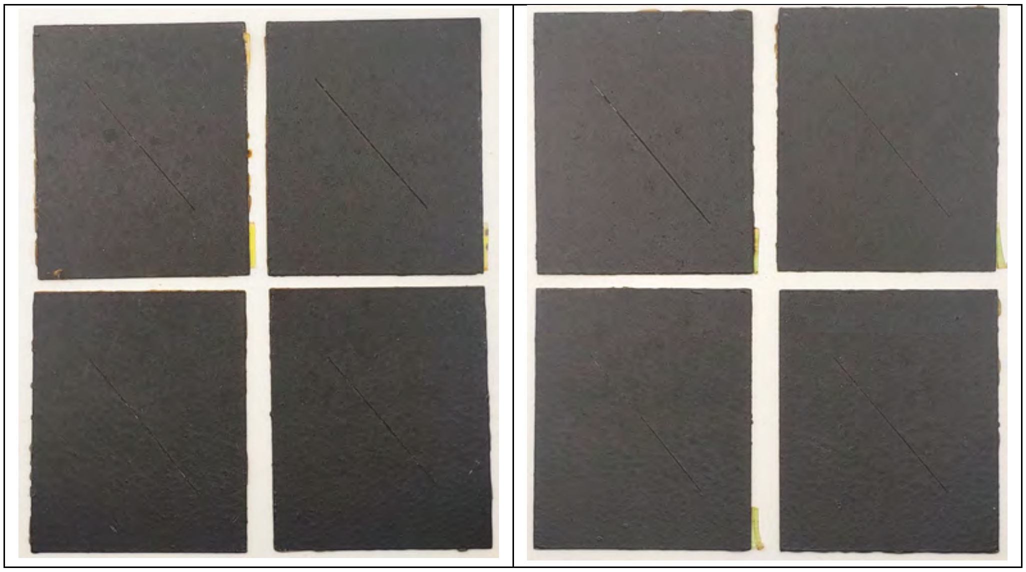


Figure C 21 Coupons for SAEJ2334 (cyclic salt spray test) – Day 54 (left) Al-AS coupons (right) SS-AS coupons

Day 60: Lightbox photos

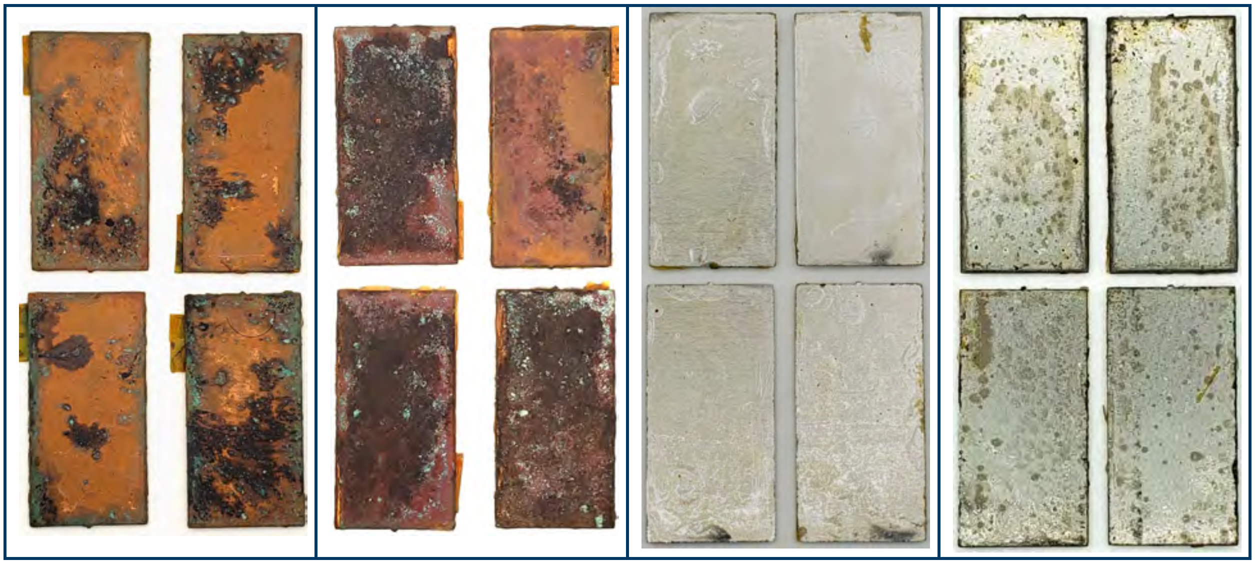


Figure C 22 Coupons for SAEJ2334 (cyclic salt spray test) – Day 60 (left to right) Cu-DG, Cu-DO, Al-FF, and S-FF coupons

Note: The flash rust on steel coupons was there before the application of Fluid Film and never increased further. The TDS of Fluid Film had no recommendations for sand blasting or surface cleaning. Even then for EIS test all steel coupons were sanded before applying Fluid Film. For the salt spray test, it was decided to apply Fluid Film without any coupon cleaning.

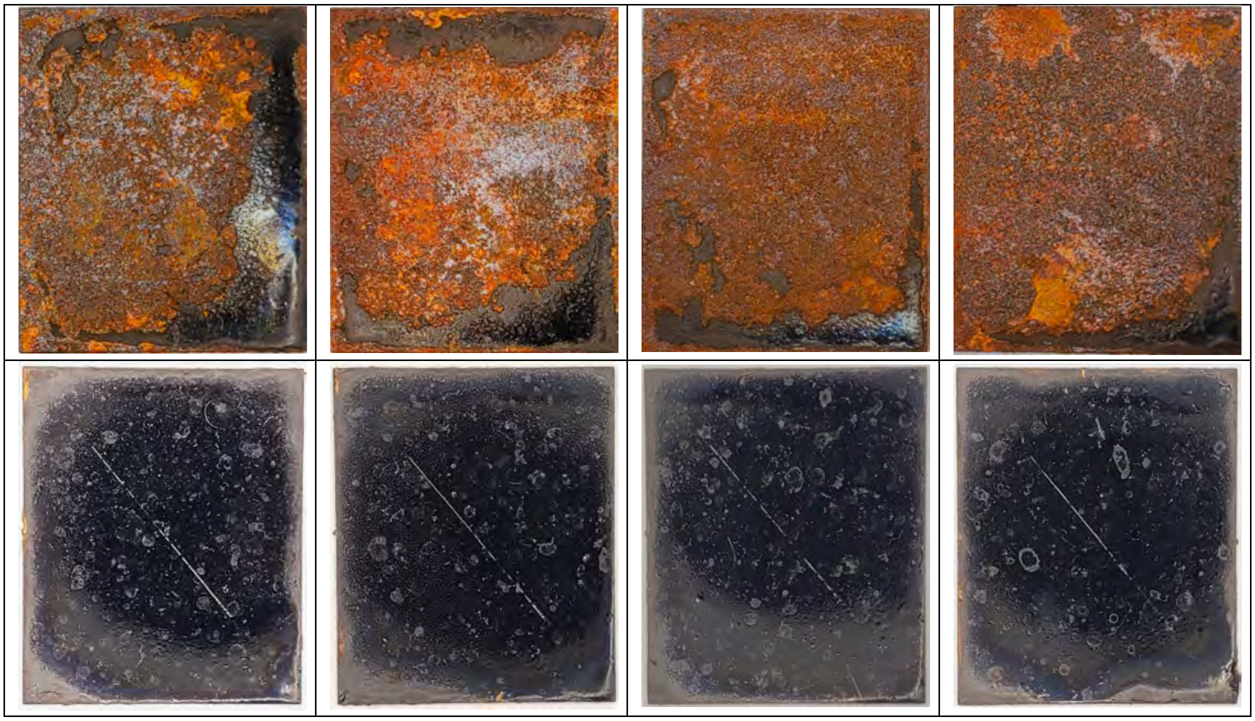


Figure C 23 Coupons for SAEJ2334 (cyclic salt spray test) – Day 60 (top) S-LS coupons (bottom) SS-LS coupons

Figure C 24 Coupons for SAEJ2334 (cyclic salt spray test) – Day 60 (left to right) Al-AS, SS-AS, and S-AP

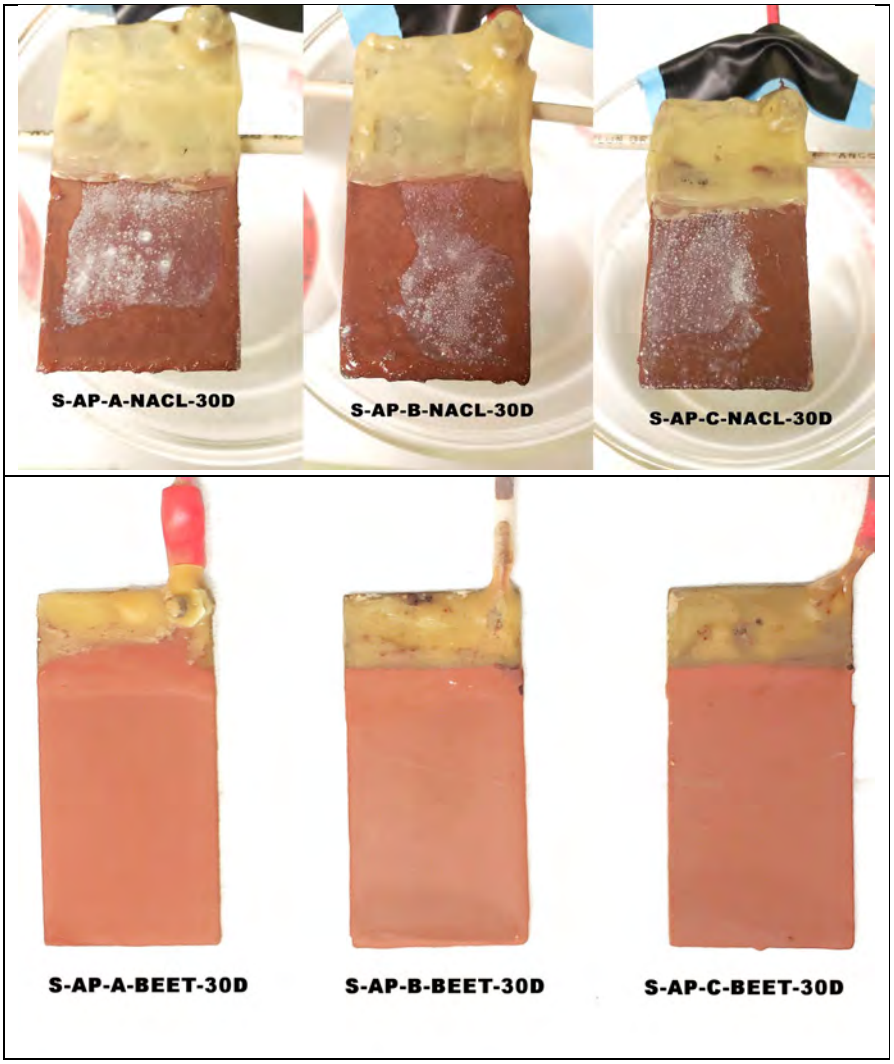


Figure C 25 EIS Coupons: (top) S-AP NaCl-$CaCl_2$ blend after 30 days (bottom) S-AP $MgCl_2$-Beet blend after 30 days

CLEAR ROADS
research for winter highway maintenance

Lead state:

Minnesota Department of Transportation
Research Services
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St. Paul, MN 55155