

# Learning from Historic Lining Field Trials at Shasta Dam and Collbran Project

Science and Technology Program Research and Development Office Final Report No. ST-2023-22024-02 Technical Memorandum No. 8540-2023-43



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prepared by

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Cover Photo – Reclamation inspector collects EIS data from inside of Collbran Salt Creek Siphon.

## **Peer Review**

**Bureau of Reclamation Technical Service Center Materials and Corrosion Laboratory Group** 

#### Final Report ST-2023-22024-02

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## Learning from Historic Lining Field Trials at Shasta Dam and Collbran Project

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## **Acronyms and Abbreviations**

AC	alternative current
avg.	average
Collbran	Collbran Project Salt Creek Siphon linings field test
cm <sup>2</sup>	centimeters squared
C <sub>coat</sub>	coating capacitance
C <sub>dl</sub>	double layer capacitance
CPE	constant phase element
CPE <sub>coat</sub>	coating capacitance
CTE	coal tar enamel
CV	coefficient of variation
deg	degrees
DFT	dry film thickness
ECM	equivalent circuit model
EIS	electrochemical impedance spectroscopy
F	Farads
ID	identifier
in	inches
OCP	open circuit potential
mV	millivolts
Hertz	Hz
Reclamation	Bureau of Reclamation
RC	resistor-capacitor
R <sub>ct</sub>	charge transfer resistance
R <sub>pore</sub>	pore resistance
R <sub>sol</sub>	solution resistance
Shasta	Shasta Dam Unit 5 Penstock linings field test
UT	ultrasonic thickness
wt.	weight

## Symbols

°F	degrees Fahrenheit
%	percent
θ	phase angle
$ \mathbf{Z} $	impedance magnitude

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## **Executive Summary**

To evaluate the performance of various lining systems for corrosion protection, Reclamation scientists conducted field trials at the Shasta Dam Unit 5 Penstock (Shasta) in 1949 and at the Collbran Project Salt Creek Siphon (Collbran) in 1959 on 26 and 42 coatings systems, respectively. The current research is the first investigation since 1972 where lining performance at Shasta and Collbran are evaluated together in a single report. The updated data collected on the various test lining materials contributes to understanding why some products provide long-term corrosion protection, with a goal to identify and develop new products with similar characteristics. Furthermore, this work helps researchers and inspectors understand how to effectively evaluate aging coating systems.

Researchers analyzed data collected during a 2019 inspection which included photographs and field electrochemical impedance spectroscopy (EIS) data from six of the original 26 tested lining systems at Shasta. Inspection photographs showed good visual coating appearance in the cleaned areas used for field EIS testing; however, EIS results showed low to moderate corrosion protection. All surviving Shasta test linings showed some level of capacitive behavior; a material with excellent barrier properties will not allow current to flow through, and instead will store electrical charge, acting as a capacitor does in an electrical circuit. Although beyond the scope of this project, application of an equivalent circuit model (ECM) to the Shasta EIS data could help determine the origin of this capacitive response.

Collbran data collection centered around a 2022 inspection where traditional visual inspection and field EIS testing data were analyzed in this study. Visual inspections found 13 of 28 lining systems exhibited no or minimal damage after 63 years. EIS data was combined with visual inspection results to correlate dielectric characteristics with the visual appearance by applying an ECM to each type of lining system. The results showed increased capacitor values for the poorest performing systems. ECM outcomes suggest the double layer capacitance results may provide a strong differentiating factor for evaluating coating performance.

The findings from Shasta and Collbran together demonstrate certain formulations containing coal-tar, red lead or vinyl can achieve corrosion protection for over 50 years of service. Additionally, Collbran showed lining systems comprised of chlorinated rubber or epoxy mastic can also provide many decades of defense for immersed steel structures. Before pursuing coating development efforts based on these results, the authors must first determine the material properties and other physical characteristics of the successful linings, then correlate ECM results to the attributes which contributed to longevity.

The preliminary field EIS results presented here show that ECM methods can aid analysis of field EIS data, but correct interpretation and validation is essential to reaching meaningful and actionable conclusions. Expanding ECM proficiencies within Reclamation requires collaboration with experts within the EIS field. Employing modern analytical techniques will be key to verifying ECM results.

Future work includes:

- Validating ECM for two-cell field EIS data.
- Determining material (electrical, thermal, mechanical) properties and physical characteristics (adhesive performance, rust creep resistance, etc.) of successful coatings materials.
- Correlating ECM results with
  - o material properties, physical characteristics, and
  - corrosion rates and degradation mechanisms.
- Validating ECM results experimentally with quantitative techniques such as elemental mapping of long-term coating test panels.
- Identifying acceptance thresholds for field EIS data and/or ECM results.

## 1. Introduction

Corrosion protection is fundamental to achieve infrastructure sustainability and optimize service lifetimes of essential structures. Bureau of Reclamation (Reclamation) applies and maintains exterior coatings and interior linings to key metallic components as the first line of defense against corrosion. To evaluate the performance of various lining systems for corrosion protection, Reclamation scientists conducted field trials at the Shasta Dam Unit 5 Penstock (Shasta) in 1949 and at the Collbran Project Salt Creek Siphon (Collbran) in 1959. Some of the trial materials remain commercially available today. The test linings were evaluated periodically through the 1960's. The Collbran test linings were last inspected in 1969. The results of that inspection were reported alongside the results of the 15<sup>th</sup> year inspection of the Shasta test linings [1]. The current research is the first investigation since 1972 where Shasta and Collbran are evaluated together in a single report.

Understanding how linings provide long-term corrosion protection is vital for infrastructure sustainability. This research project provides an update on the condition of the test linings for the Shasta and Collbran sites at their respective 70- and 63-year anniversaries. Updates to this early Reclamation research provides unique insight into the performance of aging lining systems. Reclamation strives for a 50-year service life for coatings and linings; learning which coatings in these historical field tests are still providing corrosion protection at 60-plus years will assist researchers in determining the coatings properties that can achieve the desired lifetime. The data collected on the various test lining materials from this research project contributes to understanding why some products provide long-term corrosion protection. This aids the process of identifying and developing new products with similar characteristics. Furthermore, this work helps researchers and inspectors understand how to effectively evaluate aging coating systems.

## 2. Evaluation Methods

Reclamation inspectors used multiple methods to evaluate the test linings at Shasta and Collbran to generate the data presented in this report. Brief descriptions of each method follow. <u>Section</u> <u>3.2 Shasta Experimental Details</u> and <u>Section 4.2 Collbran Experimental Details</u> provide further details on the inspections conducted at each site.

## 2.1 Visual Inspection

For the historically reported data from Shasta and Collbran, visual inspection was the sole method for evaluating the field performance of a lining system. In those field site evaluations, the linings were evaluated on a scale of Class A-D, from fully performing to failure to perform, respectively. The descriptions of each rating class are provided in Table 1 [2, 3]. Visual

inspection for field performance typically includes qualitative assessments, such as estimates of the area affected by blistering, cracking, or flaking.

Table 1.—Citteria of visual inspection for historic evaluations shasta and combran test innings							
Class A	Fully performing the intended function and showing no deterioration or defects						
Class B	Essentially performing the intended function, but showing slight deterioration and/or minor defects						
Class C	Function of the material seriously impaired, numerous and/or serious defects evident						
Class D	Failure to perform the intended function and having serious defects						

Table 1.—Criteria of visual inspection for historic evaluations Shasta and Collbran test linings

### 2.2 Dry Film Thickness Measurement

Dry film thickness (DFT) measurements are a common method of evaluating the quality of protective coatings in the field. This method relies on magnetic and electromagnetic induction to measure the distance between the surface of a coating and the underlying substrate. In the current work, Reclamation inspectors took DFT measurements and compared them with the originally reported DFT data to assist in identifying the test lining sections. Researchers also used DFT data to assess thinning and erosion effects.

### 2.3 Ultrasonic Thickness Measurement

Ultrasonic thickness (UT) measurement can be used to determine the metal thickness at discrete locations. These measurements are most repeatable where the substrate is exposed, and the surface is free of corrosion pitting. The UT measurement applies ultrasonic waves to a surface; the waves travel through the material thickness to the far wall and reflect back. The time it takes for the waves to return to the sensor indicates the thickness of the metal.

### 2.4 Electrochemical Impedance Spectroscopy Measurement

Electrochemical impedance spectroscopy (EIS) is a well-established technique for performing advanced analysis of the performance of protective coatings on steel substrates. The development and progression of this technique for laboratory evaluations is the subject of training, books, and reviews [4, 5, 6, 7]. The application of EIS for field evaluations of protective coatings was underway in the 1990's and was recently formalized through ASTM D8370, "Standard Test Method for Field Measurement of Electrochemical Impedance on Coatings and Linings" [8].

EIS applies alternating current (AC) across a range of frequencies to evaluate the electrical properties of a material. A material with excellent barrier properties will not allow current to flow through, and instead will store electrical charge, acting as a capacitor does in an electrical circuit. A material with poor barrier properties allows ionic charge transfer, acting like a resistor in an electrical circuit. Resistive behavior in a coating system reflects lower corrosion protection.

Coatings scientists commonly evaluate EIS results at the lowest test frequencies, i.e., less than 1 Hertz (Hz), which evaluates the resistances and capacitances of the complete coating-electrolyte-substrate system [7].

To evaluate the quality of a protective coating, EIS test results can be evaluated with Nyquist and Bode plots. Nyquist plots show the real impedance, i.e., traditional resistive behavior, on the x-axis and imaginary impedance on the y-axis. Bode plots show the applied current frequency on the x-axis, with impedance magnitude (|Z|) and phase angle ( $\theta$ ) displayed on the y-axis, either using stacked plots or left and right y-axes. The |Z| is derived from the real and imaginary impedance components of a material and reflects the magnitude of the resistance to electrical flow in the system, which is related to the probability of corrosion. Higher |Z| values indicate slower ionic flow and charge transfer reactions, i.e., greater corrosion protection. A coating provides good protection when |Z| at low frequencies are in the magnitude of 10<sup>8</sup> ohm-centimeter squared (cm<sup>2</sup>) or greater and poor protection when 10<sup>6</sup> ohm-cm<sup>2</sup> or lower [9]. As coating degradation progresses, ionic pathways increase, which allows a coating to absorb a greater volume fraction of water, and |Z| values decrease. With increasing water uptake, |Z| values will approach 10<sup>4</sup> ohms-cm<sup>2</sup>, the impedance level observed for bare steel [5].

The  $\theta$  value reflects the capacitive/resistive behavior of a material. An ideal coating performs as a pure capacitor and stores charge rather than passing current. Most coatings do not behave as ideal coatings, even when new, and instead exhibit  $\theta$  values near -90 degrees (deg) at low frequencies. As a coating ages and resistive materials or elements begin to dominate the EIS spectra, the  $\theta$  values move to -45 deg or higher, with a purely resistive element exhibiting  $\theta$  values at or near 0 deg.

Capacitive behavior in materials reflects the complex nature and exemplifies the value of the AC technique. Thorough investigation of capacitive behavior requires an equivalent circuit model (ECM) to extract the values of the physical elements used in the model. The physical elements used for protective coatings are typically a single resistor-capacitor (RC) pair for a non-degraded coating and two RC pairs for a degraded coating [5]. The first RC pair represents the coating resistance and capacitance, and the second models emerging dielectric properties, typically the development of an oxide layer, electrochemical reactions at the interface, etc. [5]. RC pairs are also referred to by the number of time constants in the EIS spectra. Good knowledge of the material being investigated and other experimental techniques may be required to confirm the physical materials or processes responsible for the additional time constants [10]. ECM requires use of both the Nyquist and Bode plots to evaluate for the number and type of circuit elements needed and the goodness of a resulting fit.

For the inspections outlined in this report, EIS measurements followed procedures outlined in a previous Reclamation report on EIS evaluation practices and once published, ASTM D8370 [11].

## 3. Shasta Dam – Unit 5 Penstock

### 3.1 Background

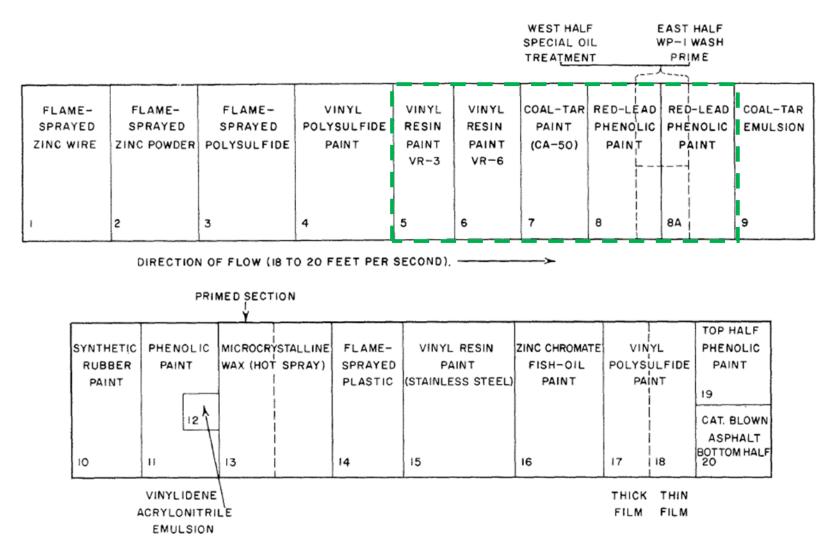
Reclamation's Shasta Dam was completed in 1945 and is located on the Sacramento River, approximately nine miles northwest of Redding, California. The dam and its attached powerplant are part of the Central Valley Project, which provides water for irrigation, municipal and industrial facilities, and environmental needs. Shasta Powerplant has five hydroelectric power generators, and power generated here is managed and sold by the Western Area Power Administration.

The Shasta field trial is in a 160-foot-long section of the Shasta Dam Unit 5 Penstock. The initial trial included 26 lining systems comprised of 20 different coatings materials applied in 1949. The weather was warm and clear with the metal temperature ranging from 80 degrees Fahrenheit (°F) to 110 °F. Following thorough sandblasting to remove all rust and mill scale, the linings were applied to the dry surface according to the manufacturer's instruction—either by the respective manufacturer's representative or with their witness. The remainder of the Unit 5 Penstock was lined with coal tar enamel (CTE). The exterior of the pipe was coated with red lead primer and a phenolic aluminum topcoat.

The test section is around the midpoint of the penstock and has a slope of about 16 deg. Typical conditions for the penstock include continuous water immersion, little to no sediment erosion, and minor abrasion from occasional pieces of foreign material.

Figure 1 gives a schematic of the 160-foot-long test section layout [2]. Table 2 gives the lining system identifier (ID) for the linings systems receiving Class A or Class B ratings at the 8<sup>th</sup> year inspection alongside the 15<sup>th</sup> year ratings, and pertinent application notes [12]. Eight years after the installation, Reclamation coatings inspectors assigned a Class A rating to eight of the lining systems comprised of four materials: phenolic, two red-lead phenolics, and vinyl VR-3 [2]. Section 8-2, an augmented red led phenolic with an oil-based primer was the only one of those superior eight lining systems to be demoted to a Class B rating at 15 years [1, 12].

In 2009, contractors applied 100 percent (%) solids epoxy repairs to the original CTE lining. The work led to the discovery that some of the test linings from the 1949 study were still in place. The surviving areas of test linings are outlined within the original layout with a green dashed box in Figure 1 and noted with an asterisk in Table 2 [13].



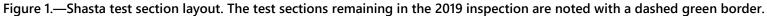


Table 2.—Shasta lining systems that received a Class A or Class B rating after eight years in service listed alongside their rating after 15 years in service and the number (no.) of coats applied and the average (avg.) total DFT reported at the time of installation

Test Section ID	Lining System Description	No. of Coats⁵	Avg. DFT (mils)	8 <sup>th</sup> Year Rating	15 <sup>th</sup> Year Rating
3	Flame-sprayed polysulfide	1 <sup>¶</sup>	31	В	В
4	Vinyl polysulfide	4	7.0	В	В
5*	VR-3 vinyl	4	4.2	Α	А
6*	VR-6 vinyl	6	10	В	В
7*°	Coal tar CA-50 or CTE	5	20	В	В
8-1*	Red lead phenolic direct to metal	4	5.0	Α	А
8-2+	Red lead phenolic with oil primer	5	5.0	Α	В
8-3	Red lead phenolic with wash primer	5	5.0	А	В
8A-1*	Red lead phenolic <sup>*</sup> direct to metal	5	8.0	Α	А
8A-2*	Red lead phenolic <sup>*</sup> with oil primer	6	8.0	Α	А
8A-3	Red lead phenolic <sup>*</sup> with wash primer	6	8.0	А	А
10	Synthetic rubber	4	9.0	В	С
11	Phenolic	4	5.0	В	С
17	Vinyl polysulfide	10	18	В	С
19	Phenolic	4	4.0	Α	А
20	Catalytically blown asphalt	1 <sup>1</sup>	90	В	В

\* Sections included in the 70-year inspection.

° The original CA-50 lining may have been repaired with CTE.

+ The field identification of 8-2 and 8-2A was not verified; only one of these linings was evaluated in 2019 [13].

<sup>‡</sup> Red lead phenolic augmented with zinc chromate and zinc oxide pigments.

§ Includes primer and finish coats.

¶ Multiple passes were applied to build a single coat.

### **3.2 Shasta Experimental Details**

In October 2019, Reclamation coatings inspectors performed a visual inspection and collected EIS measurements from the remaining areas of the 70-year-old test lining systems and the 10-year-old 100 % solids epoxy lining in the Shasta Unit 5 Penstock, shown in Figure 2. The complete results of that inspection are summarized separately [14]. While the 2019 inspection was not conducted as part of the Historic Linings project, which did not commence until 2021, the data collected in 2019 has been incorporated into the findings presented here and in a previous interim report [13]. Figure 2a identifies the surviving areas of the original test sections which are considered in this report. Figure 2b shows EIS cells placed along the left and right sides of the invert along the original test section length.



Figure 2.—The test linings section of the Shasta Unit 5 Penstock looking (a) downstream with labels of the surviving test lining section IDs, and (b) upstream with EIS test cells in place.

For EIS evaluation of the test linings, the inspectors cleaned and dried the test areas before affixing four 5-cm diameter test cells with silicone adhesive to provide an evaluation area of approximately 20 cm<sup>2</sup> for each lining system. Once the adhesive had dried, inspectors filled each cell with a water and sodium chloride solution and let the evaluation areas hydrate overnight. The prepared EIS test areas are shown in Figure 2b. Two of four cells were evaluated for each measurement. Inspectors measured the open circuit potential (OCP) prior to recording data and

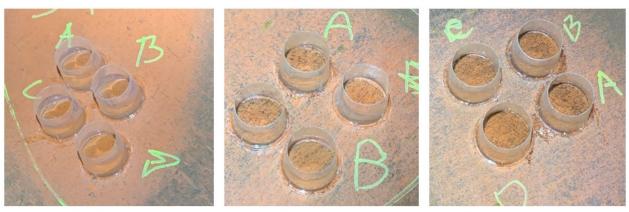
took three random measurements while alternating the test cells to provide an average impedance value of the evaluation area for each test section. The EIS frequency range was set to 10<sup>5</sup> through 10<sup>-1</sup> Hz, data collection was set to five points per decade, and the amplitude of the applied voltage was set to 50 millivolts (mV). Photos of EIS test cells show close spacing of the cells, as seen in Figure 3. The nearest distance between test cells is approximately 1 cm and may have been less in some cases. Industry consensus has since set test cell spacing at a minimum of 5 cm per ASTM D8370, which was published in 2021 [8]. Without standard practices yet established, Reclamation's field EIS methods and practices were still in development in 2019. Additionally, many of the EIS data files from Shasta were corrupted and required alternate processes to extract the data used for the Historic Linings project.



Section 5

Section 6

Section 7



Section 8-1

Section 8A-1

Section 8-2 or 8A-2

Figure 3.—Field EIS test cells of the surviving test lining sections at Shasta. Each cup is roughly 5 cm in diameter.

### 3.3 Results and Discussion

#### 3.3.1 Visual Inspection

The 2019 inspection report noted that all linings in the test section were in poor condition with several spots of spalled or missing coating and substrate corrosion present [14]. A recent assessment of the 2019 inspection report, conducted as part of the Historic Linings research, closely examined the images of each EIS test cell location, and concluded the appearance of many of the surviving coatings could be consistent with a Class B rating and did not warrant immediate maintenance [13]. The report also noted that it was not clear whether the lining in Section 7 was the original CA-50 test lining system, or whether it had been repaired with CTE. Additionally, the process of correlating inspection images with the original lining system descriptions could not differentiate the identity of the 8-2 and 8A-2 test locations.

#### 3.3.2 Electrochemical Impedance Spectroscopy

See Appendix A – Shasta EIS Bode Plots for the full set of Bode plots produced from the EIS data collected during the 2019 inspection. The current section presents summary analyses of the data in box-and-whisker plots. Figure 4 provides box and whisker plots of |Z| and  $\theta$  at 0.1 Hz for each test lining. Each box and whisker plot shows the median as a horizontal line through the box. The lower and upper edge of the box indicate the first and third quartiles of the dataset, respectively, while the lower and upper whisker are the respective minimum and maximum data points. The plot indicates the range of resulting data and relative spread of the data to each other. Highly repeatable data provides a compressed box-and-whisker compared to variable data, which imparts various extended shapes to the box-and-whisker. For example, System 7 has a wide box because the data are split equally near the maximum and minimum—two are near  $8 \cdot 10^6$  ohms, two are near  $2 \cdot 10^6$  ohms, and one is near  $5 \cdot 10^6$  ohms. By contrast, results with longer whiskers indicate that the data is more evenly spread across the range or that less clustering occurred across the replicate set.

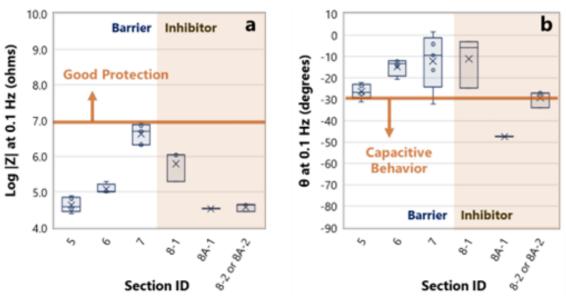


Figure 4.—Shasta test linings data for a) Log |Z| and b)  $\theta$ ; data is shown in an inclusive median box and whisker plot of all replicates at 0.1 Hz; inner data points and outliers are designated with open circles.

Figure 4 also incorporates the system type as background shading in the plot. No shading indicates the lining is a barrier coating; these systems have a more straightforward interpretation of the EIS results, in which higher |Z| values generally indicate higher corrosion protection. The systems shaded and labeled as "Inhibitor" contain an inhibitive protection mechanism through a red lead primer. Inhibitor coatings interrupt active corrosion cells by forming protective oxides [9]. This protective mechanism requires more sophisticated understanding of the EIS spectra, moving beyond a simplified "higher |Z| provides greater corrosion protection" interpretation. ECM can be used to interpret EIS spectra more completely for systems containing inhibitors. This method is used in the analysis of Collbran data in 4.3.4.2 Equivalent Circuit Modeling. Because multiple data files were corrupt and are not readable by the modeling software, a thorough analysis of the impedance characteristics of the Shasta linings is not possible.

Figure 4a includes a line at  $10^7$  ohms. Above this value, lining systems provide good barrier protection against corrosion. Based on this analysis alone, the highest performing linings are Systems 7 and 8-1. Most systems show a narrow data range, especially those with |Z| near  $10^4$  to  $10^5$  ohms. This low |Z| is typical of bare steel, oxides, or other unintended current pathways. Further, the steel surface temperature, if warmer than typical laboratory temperatures, will show a decreased |Z| because |Z| has an inverse relationship with temperature [15].

Figure 4b includes a line at -30 deg to aid in interpretation of this plot. Near 0 deg the resistive elements dominate EIS data at 0.1 Hz. As  $\theta$  values become more negative, capacitive behavior has an increasing influence. At -45 deg, diffusion-limited corrosion typically occurs, known as Warburg diffusion, and as  $\theta$  approaches -90 deg, capacitive behavior dominates the data. For EIS results showing a single RC pair for the coating, the capacitive behavior is an indication of good barrier protection. However, this analysis becomes more complex when a second RC pair is needed to model the interface because oxides and/or corrosion reactions are significant contributors.

All systems show some capacitive behavior, with  $\theta$  values of Systems 5 and 8-2 or 8A-2 near -30 deg. System 8A-1 has a narrow  $\theta$  range near -45 deg. The nature of the capacitive behavior for each system would require ECM to characterize, which would help to determine if the behavior is a contribution of the coating, the interface, or some combination thereof.

## **3.4 Summary of Findings**

All Shasta test linings evaluated showed low barrier protection, indicated by low |Z| values. System 7 exhibited the highest values near 10<sup>7</sup> ohms followed by System 8-1 near 10<sup>6</sup> ohms (where 10<sup>7</sup> ohms or greater indicates good corrosion protection). All systems showed some capacitive behavior that would require ECM to further characterize. Due to close test cell placement, Shasta field EIS data may not accurately represent the actual impedance of the test lining sections.

The 2019 Shasta inspection was conducted before the Historic Linings projected commenced, with the Unit 5 Penstock interior making up a small part of the overall objective of the work conducted as part of a ropes-access site visit. The limited photographic documentation of the

Shasta test section was not sufficient for a thorough visual assessment, limiting the visual assessment to the images of each EIS test cell location. Photos show the EIS evaluation areas are in good condition, prompting a general recommendation that maintenance be deferred [13]. However, the overall poor visual appearance of the entire test lining areas reported in 2019 and low |Z| values measured with field EIS, suggest the linings are degraded and may no longer be providing ample corrosion protection to the underlying steel. This highlights some key takeaways.

- The 2019 inspection of the Shasta Unit 5 Penstock provided important lessons in Reclamation's development of field EIS practices. The issues encountered during this developmental phase supported and informed the development of ASTM D8370. In particular, the standard prescribes a minimum 5-cm spacing between EIS test cells to ensure no current passes across the coating surface (i.e., a short circuit between the cells) via adsorbed surface moisture. If a short circuit occurs across the surface, the EIS tests measure the surface solution resistance in addition to or instead of the materials and reactions beneath the cells.
- EIS data analysis must consider both |Z| and  $\theta$  values together. The |Z| measurements can be as low as 10<sup>4</sup> ohms—effectively bare steel—with no visible corrosion showing through the lining. When such low values are accompanied by  $\theta$  values consistent with capacitive properties, as seen in EIS data for Sections 7 and 8-1, it suggests the impedance measurements includes the contribution of an oxide layer formed beneath the coating. In these cases, ECM is an especially important step to understanding the impedance behavior (and level of corrosion protection being provided) by the coating.

## 4. Collbran Project – Salt Creek Siphon

## 4.1 Background

Reclamation's Collbran Project includes multiple dams, powerplants, canals, pipelines, and penstocks. As part of the project, Vega Dam was completed in 1960. This dam provides irrigation water to the 29.1-mile Southside Canal, an unlined canal with 13 siphons and other various hydraulic structures. The Salt Creek Siphon is a 6-foot diameter underground segment of this canal.

In 1959, a steel pipe fabricator manufactured seven 40-foot-long pipe sections of six-foot diameter pipe, labelled with the letters A-G. The interior and exterior surfaces of each pipe section was abrasive blast cleaned to a white metal finish using a mix of 16/30 steel grit and garnet sand. The fabricator applied 36 interior lining systems comprised of 42 coatings materials at the fabrication site according to manufacturer's instructions. One of the test lining systems, VR-3 vinyl with an inhibitive primer, was applied to two non-adjacent test sections, making a total of 37 separate test sections. Each of the 36 lining systems was assigned a number and the lining test section was assigned an alpha-numeric identifier corresponding to the lining number and pipe section letter. At the time of application, the test systems were categorized into five

general types of coating-materials: resins, synthetic rubber and asphaltic coatings, metallic coatings, and surface conditioners [16]. The current work generalizes each test lining according to the mechanism by which it protects against corrosion with the following three categories: metallized, inhibitive, and barrier. The pipe exteriors were coated with CTE with an asbestos outer wrap and Kraft paper. The seven pipe sections that make up the linings field test are installed on the 11.4 % grade section of the Salt Creek Siphon near the outlet end. Table 3 lists the 29 surviving lining sections evaluated in this report, the lining system ID, and description with application data provided by the original records [16].

System ID	Description	Coats	Avg. DFT (mils)
1A	Chlorinated rubber	4	4.5
3A	Coal tar epoxy	3	21.0
4B	Inhibitive resin primer and coal tar polyurethane	5	16.5
5B	Coal tar polyurethane	3	17.0
6B	Coal tar polyepoxy	2	21.0
7B	Coal tar epoxy	3	22.0
8B	Phenolic with mica primer and phenolic	3	25.0ª
9C	Aluminum metal; vinyl butyral primer; vinyl alkyl aluminum	3	9.5
10C	Zinc metal; vinyl butyral primer; vinyl alkyl aluminum	3	8.5
11C	Zinc metal; thinned VR-3 vinyl; VR-3 vinyl seal	4	12.5
12C	Zinc metal and phenolic seal	3	16.0
16D	Organic zinc and phenolic red lead	4	12.0
21D	VR-3 vinyl	4	8.5
22E	Chlorinated rubber primer and liquid neoprene	7	25.0 <sup>b</sup>
23E	Chlorinated rubber primer with inhibitive pigment; liquid neoprene	7	25.0 <sup>b</sup>
24E	Chlorinated rubber primer with inhibitive pigment; liquid neoprene; chlorosulfonated polyethylene (aluminum)	6	15.5
25Ea	Inhibitive primer and VR-3 vinyl	5	5.5
25Eb	Inhibitive primer and VR-3 vinyl	5	5.5
26E	Vinyl wash primer and neoprene	6	10.0
27E	Neoprene	5	10.0
28F	Epoxy mastic	2	13.5
29F	Vinyl mastic	3	8.5
30F	Vinyl wash primer and vinyl red lead	7	6.5

Table 3.—Test lining sections at Collbran Salt Creek Siphon with ID, number of coats, and avg. total lining thickness, rounded to the nearest 0.5 mil

<sup>a</sup> Reported as 25+

<sup>b</sup> Coating not cured sufficiently for accurate measurement.

System ID	Description	Coats	Avg. DFT (mils)
31F	Vinyl wash primer and phenolic red lead	4	7.0
32F	Phenolic red lead	3	6.0
33G	Metal conditioner and phenolic red lead	4	6.0
34G	Metal conditioner and VR-3 vinyl	5	7.0
35G	Epoxy phenolic	2	12.5
36G	Asphalt primer and asphalt enamel	2	94.0

Table 3.—Test linings at Collbran Salt Creek Siphon with ID, number of coats, and avg. total lining thickness, rounded to the nearest 0.5 mil

The siphon experiences minimal temperature fluctuation because it is buried. The linings were immersed year-round for the past 63 years, unless intentionally dewatered for maintenance. During irrigation season, May to October, the water flows through the siphon at up to 8.8 feet per second [16]. These conditions reduce hygrothermal stress caused by cycles of changing temperature and moisture. Hygrothermal stress causes increased internal stress and lining degradation. Thus, the test linings condition after 63 years at Collbran may be reflective of less severe service conditions than what is typical at other Reclamation sites.

### 4.2 Collbran Experimental Details

In October 2022, Reclamation coatings inspectors performed a visual inspection and collected DFT, UT, and EIS measurements from the surviving areas of the 63-year-old test linings. Prior to the inspectors' arrival, Reclamation field personnel pressure washed as much of the siphon's test section as possible to facilitate inspection. Upon entering the siphon, the group of inspectors used notes from historic records to identify each test section and labeled each with the ID and lining name with chalk to aid inspection. All 37 original test segments were identified inside the siphon. Eight of the 37 sections—2A, 13C, 14C, 15C, 17D, 18D, 19D, and 20D— and much of the invert length appeared to have been relined with CTE sometime between 1960 to 2022. Except for some invert areas, the remaining 29 sections appeared to have the originally applied test linings intact. For each of those surviving test linings, inspectors thoroughly cleaned one or more areas of nearly 10 sq ft to allow for visual assessment and EIS testing. Note that 25Ea and 25Eb are the same material in separate sections, so <u>Section 4.3 Results and Discussion</u> considers 28 unique lining systems.

Inspectors evaluated each of the remaining lining sections on overall visual condition. They also estimated the area percentage affected by blistering and the area percentage with other forms of coating damage such as rust nodules, spalling, erosion, etc. Additionally, inspectors estimated the average blister diameter for affected areas. After the inspection was concluded, visual inspection data was reviewed with photos and the team determined the final visual rating results using the historic rating system described in Table 1.

For EIS evaluation of the test linings, inspectors used ASTM D8370 [8]. Inspectors affixed a minimum of six approximately 5-cm diameter saturated felt pads to the lining surface with foil tape to hydrate the test area overnight. The felt pads were saturating in dilute Harrison's solution

(0.35 weight (wt.)% ammonium sulfate and 0.05 wt.% sodium chloride). The tape was removed from one set of hydrated areas per measurement and magnetic test cells affixed to electrically connect the test area to the laptop-controlled potentiostat. Inspectors used to rag to carefully wipe the surface around each magnet dry. Each magnetic test cell contains a coupled electrode for current and potential measurement by the instrument. Figure 5 shows a prepared EIS test area. Inspectors measured the OCP prior to recording data. The amplitude of the applied voltage was set to 700 mV root mean squared. The EIS frequency range was 10<sup>5</sup> to 10<sup>-1</sup> Hz, and data collection was set to five points per decade. Inspectors recorded surface temperature, air temperature, and relative humidity (RH) for each test area. Inspectors analyzed each pair of specimens for the resulting spectra features and calculated mean and standard deviation for the low frequency data for each test section.

Inspectors collected three DFT measurements from each lining. Inspectors collected two to five ultrasonic thickness measurements of the pipe wall in areas where bare metal was found exposed, mostly along the invert.

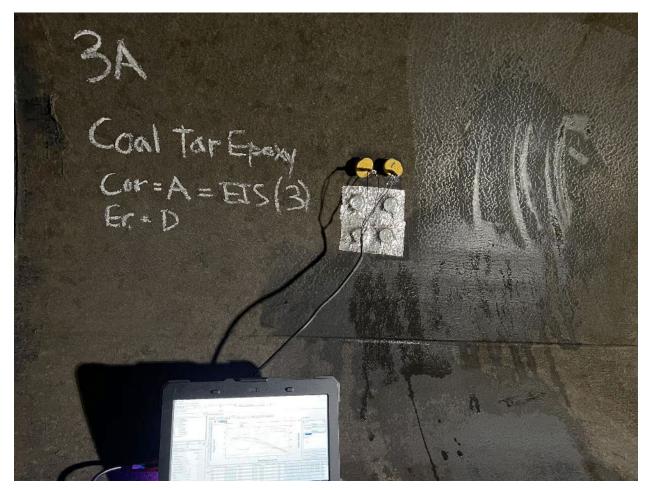


Figure 5.—EIS data collection from Collbran test linings System 3A. Two magnetic cells connect the uppermost test area to the potentiostat during the test. Pads saturated with electrolyte remain taped to the surface below to keep the lower test areas hydrated.

### 4.3 Results and Discussion

Coatings and linings are heterogeneous materials with different resins, curing agents, pigments, and additives. Variability across a coating system is inevitable. Surface preparation, coating application, or environmental conditions during application can significantly affect the corrosion protection of the coating/lining materials. Additionally, there is variability in the exposure conditions; coatings in some locations may be subject to damage from sediment erosion or floating debris, hygrothermal stress, etc., while other settings are less severe. Any coating/lining system that provides long-term corrosion protection (i.e., more than 50 years) is the result of choosing a suitable material for the given service environment and correctly applying it to a dry, contamination-free surface during appropriate environmental conditions.

Wetting and drying cycles and temperature fluctuation create hygrothermal stresses which typically reduce the service life of any given lining. The Collbran Salt Creek Siphon service environment was constant water immersion since it was not dewatered during the non-irrigation season, except for occasional maintenance. The pipe was buried, resulting in a narrow range of temperature fluctuation, i.e., minimal thermal expansion contraction of the linings. During the irrigation season, the water velocities were up to 8.8 feet per second with sediment flowing along the invert.

Due to the variability in materials, application, and exposure conditions, linings degrade at different rates in any given square foot of surface area. The weakest point or high stress point will most likely be the first point of failure. Example degradation modes include physical damage (impact), molecular events (chain scission/hydrolysis), or physical changes resulting from the molecular events such as stress cracking or inelastic strain. For this reason, quantitative measurements must have multiple replicates.

#### 4.3.1 Visual Inspection

The results of the visual inspection are shown in Table 4 alongside the ratings reported from the 10-year inspection. Three of the 29 lining sections received an overall Class A rating for visual assessment per classification definitions in Table 1—Systems 1A, 3A, and 34G. As may be expected, this is a significant decrease from the 1969 inspection ratings, where all except three systems rated Class A. The progression of degradation varied, with some 1969 Class A rating of Class A or B. The 2022 inspection resulted in ten Class B, six Class C, and nine Class D ratings. The Class B ratings included Systems 5B, 7B, 8B, 9C, 11C, 21D, 24E, 25Ea/25Eb, 28F, and 30F. Of the Class C and D ratings, those with over 50 % blistering or other damage included Systems 4B, 12C, 16D, 27E, and 32F.

ID	Lining System Description	1969 Rating	2022 Rating	Other Coating Damage (% Area) <sup>a</sup>	Blister (% Area)ª	Avg. Blister Diameter (in) <sup>b</sup>	Remarks
1A	chlorinated rubber	А	А	0	0	N/A	No damage evident; invert appears to have been repaired
3A	coal tar epoxy	А	А	0	0	N/A	No damage evident
4B	inhibitive resin primer and coal tar polyurethane	В	D	50	20	> 1	Blisters combine to form large spalls and delaminations; bare steel and rust nodules evident; not able to clean without causing damage
5B	coal tar polyurethane	А	В	1	1	1⁄2	No damage reported
6B	coal tar polyepoxy	А	С	10	5	≥ 2	Overall good coating appearance with only a few larger blisters which are spalled with large rust nodules beneath
7B	coal tar epoxy	А	В	0	1	1	Overall good coating appearance
8B	phenolic with mica primer and phenolic	В	В	1	0	N/A	Several small rust spalls or blisters observed near crown; rust-through microcracks evident within cleaned area at invert
9C	aluminum metal; vinyl butyral primer; vinyl alkyl aluminum	A	В	1	0	N/A	Good coating appearance at 3 o'clock position; several rust spots at invert; invert has been repaired with black coating

Table 4.—Visual inspection results for 2022 including estimated damage and blister areas, remarks, and 1969 Class ratings for comparison

ID	Lining System Description	1969 Rating	2022 Rating	Other Coating Damage (% Area) <sup>a</sup>	Blister (% Area)ª	Avg. Blister Diameter (in) <sup>b</sup>	Remarks
10C	zinc metal; vinyl butyral primer; vinyl alkyl aluminum	А	С	10	5	1⁄4	Significant blistering, decreasing in size with increasing distance from invert; one small corrosion spot near second EIS measurement; invert damage appears to be galvanically coupled
11C	zinc metal; thinned VR-3 vinyl; VR-3 vinyl seal	А	В	1	0	N/A	Minor damage near the invert; good coating appearance in cleaned area
12C	zinc metal and phenolic seal	В	D	80	80	1/4	Blisters and rust nodules evident; blisters are uniform and appear aligned with application brush strokes; several large, square sections do not have damage whereas blisters are uniform in all other areas
16D	organic zinc and phenolic red lead	A	D	95	90	1⁄4	Topcoat is blistering and delaminating at the primer layer; blistering and damage is relatively consistent across coating; blister size varies; 10-20 % of the blisters are through the primer and corrosion is evident; several large rust nodules appear near crown
21D	VR-3 vinyl	А	В	0	1	< <sup>1</sup> / <sub>8</sub>	Several tiny blisters evident
22E	chlorinated rubber primer and liquid neoprene	A	D	35	35	1/4 - 1/2	Coating is smooth; microcracking is evident with highest density near welds and toward the pipe invert where rust-through is evident; damage is similar in appearance to what is typical for CTE; heavy, uniform blistering covers coating between 4 o'clock and 8 o'clock

Table 4.—Visual inspection results for 2022 including estimated damage and blister areas, remarks, and 1969 Class ratings for comparison

ID	Lining System Description	1969 Rating	2022 Rating	Other Coating Damage (% Area) <sup>a</sup>	Blister (% Area)ª	Avg. Blister Diameter (in) <sup>b</sup>	Remarks
23E	chlorinated rubber primer with inhibitive pigment; liquid neoprene	A	D	35	35	1/4 - 1/2	Uniform blistering is concentrated between 4 o'clock and 8 o'clock position; very fine microcracking with small amount of rust-through is evident and more pronounced with decreasing distance from the invert
24E	chlorinated rubber primer with inhibitive pigment; liquid neoprene; chlorosulfonated polyethylene (aluminum)	A	В	0	1	1⁄4	Several unperforated blisters are spread throughout cleaned area; blistering appears to be more frequent near the invert
25E a	inhibitive primer and VR-3 vinyl	А	В	1	1	N/A	Overall good coating appearance; a few blisters evident near the invert; invert has been repaired
25E b	inhibitive primer and VR-3 vinyl	А	В	1	1	1/2	Overall good coating appearance; blisters are larger near the crown
26E	vinyl wash primer and neoprene	А	С	10	10	1/16	Overall good coating appearance; damage is relatively uniform with tiny blisters across much of the surface, possibly more near the invert
27E	neoprene	A	С	80	80	<sup>1</sup> / <sub>16</sub>	Many small blisters, even and concentrated, running together; more damage near the interface with adjacent section; rust nodules from blistering appear around 2 o'clock and 10 o'clock positions; staining and rust nodules evident across the crown
28F	epoxy mastic	А	В	1	0	N/A	Invert appears to have been repaired with a black coating; a single large, roughly 1-in rust nodule appears just above the invert repair

Table 4.—Visual inspection results for 2022 including estimated damage and blister areas, remarks, and 1969 Class ratings for comparison

ID	Lining System Description	1969 Rating	2022 Rating	Other Coating Damage (% Area) <sup>a</sup>	Blister (% Area)ª	Avg. Blister Diameter (in) <sup>b</sup>	Remarks
29F	vinyl mastic	A	С	3	3	> 1	Overall good coating appearance: some surface roughness is evident, which appears consistent with dry spray; randomly dispersed blisters across the pipe section; localized blistering clusters at the interfaces with adjacent coating sections
30F	vinyl wash primer and vinyl red lead	А	В	1	1	1	Overall good coating appearance; a few isolated blisters below the 4 o'clock position
31F	vinyl wash primer and phenolic red lead	A	С	2	2	<sup>1</sup> ⁄4 ≤ 1	Blisters and rust nodules evident; increased blistering between the crown and spring line; cracking is evident in the cleaned section near the invert; damage appears consistent with alkyd oxidation; EIS test cells include cracking
32F	phenolic red lead	A	D	70	70	1⁄4 - 1+	Rust nodules and many blisters; blistering is more significant below the spring line and densest around the interface with the adjacent pipe section
33G	metal conditioner and phenolic red lead	A	D	20	20	½ - 1	Blisters and rust nodules evident; topcoat is delaminating and flaking; damage appears consistent with alkyd oxidation; damage goes up to the 1 o'clock and 11 o'clock positions, and appears most significant near the invert and spring line
34G	metal conditioner and VR-3 vinyl	A	A	0	0	N/A	Gray topcoat is worn through near the invert, where the white mid-coat is revealed from roughly the 5 o'clock to 7 o'clock positions

Table 4.—Visual inspection results for 2022 including estimated damage and blister areas, remarks, and 1969 Class ratings for comparison

ID	Lining System Description	1969 Rating	2022 Rating	Other Coating Damage (% Area) <sup>a</sup>	Blister (% Area)ª	Avg. Blister Diameter (in) <sup>b</sup>	Remarks
35G	epoxy phenolic	A	D	10	10	<sup>1</sup> / <sub>8</sub> - 1	Blisters and rust nodules evident around the spring line, most severe around the 3 o'clock position; cleaned area (gray in color) appears relatively damage-free, and may be a repaired area
36G	asphalt primer and asphalt enamel	A	D	35	35	<sup>1</sup> / <sub>8</sub> - 1	Wide, shallow blisters evident in cleaned area; cracks appear over blistering, which appears more abundant below the spring line and near the invert; coating appears thick with many drips and sags; possible rust nodules near the crown

Table 4.—Visual inspection results for 2022 includi	and a string state of all string and a string of the light string string of	
120104 - V(s)(2) (0.000)	a actimated damade and nilitter areas	c remarks and typy thas ratings for comparison

#### 4.3.2 Dry Film Thickness

DFT results are reported as averages in Figure 6, shown with the originally reported average coating thicknesses. Average DFT values of Collbran linings measured in 2022 are plotted with the original average and maximum DFT values recorded in 1959. Error bars represent the standard deviation of the data. System 36G was beyond the limit of the DFT gauge, over 100 mils.

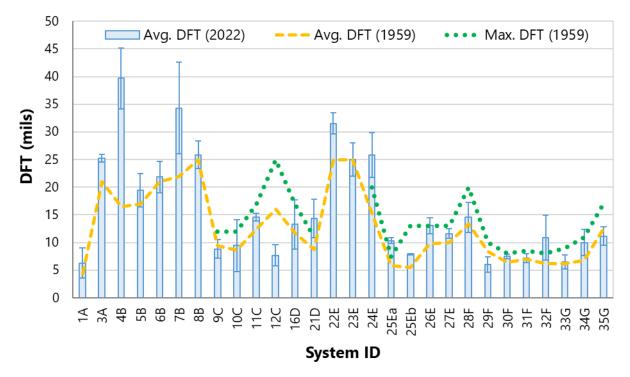


Figure 6.—Average DFT values of lining thickness measured in 2022 plotted with the original average (avg.) and maximum (max.) DFT values recorded in 1959. Error bars represent the standard deviation of the data. 36G was beyond the limit of the DFT gauge.

Reductions in DFT, as seen for Systems 9C, 12C, 16D, 29F, and 35G may be the result of erosion. Conversely, Systems 21D, 24E, 25Ea, and 32F show a marked increase in DFT when comparing the average measurement recorded in 2022 with the maximum measurement from 1959. There are multiple possible reasons for these anomalies. Since lining thickness can vary significantly from one location to another, the most obvious explanation would be that the recent DFT measurements were taken in an area with higher overall thickness than where the original measurements occurred. The effects of lining swelling from hydration levels (when DFT measurements occurred at EIS test locations), surface roughness, and equipment accuracy may also have contributed to the results.

#### 4.3.3 Metal Wall Thickness

Ultrasonic thickness measurement results are reported as averages in Figure 7. The historic reports do not make note of the original wall thickness, but drawings suggest it is 3/8 in. It is common for the manufactured pipe as received to exceed the specified value. Several results have large error bars and a mean value greater than the assumed original thickness. Others have small error bars and suggest metal loss may have occurred, such as for System 5B and 9C. The large variability in wall thickness data from each section suggests significant irregularity in the original wall thickness. Without knowing the as-manufactured dimensional tolerance of the pipe, interpretation base metal loss is not possible without further physical assessments. Thus, no conclusions are drawn from the UT data.

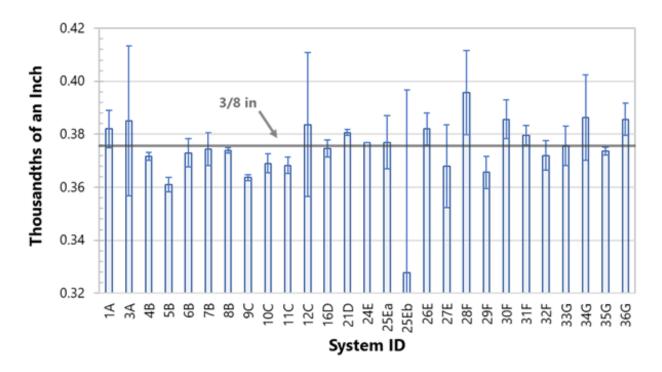


Figure 7.—Average steel thickness at Collbran test lining areas, as measured with UT, marked with the original wall thickness of 3/8 in. No data was collected for Systems 23E and 22E.

#### 4.3.4 Electrochemical Impedance Spectroscopy

The temperature ranged from 49 °F to 58 °F and is reported with the respective Bode plots. Most humidity readings were near 50 % RH with a maximum of 60 % RH. An inverse relationship exists between |Z| and surface temperature; whether RH affects the results is not yet clear [17]. No temperature corrections are used in the data analysis for |Z|.

See Appendix B – Collbran EIS Bode Plots for the full set of Bode plots produced during the inspection. The current section presents summary analyses of the data as a box-and-whisker plot and results of ECM for a subset of the higher-rated lining systems. The ECM calculates the value of each resistor and capacitor element in the model. Corrosion protection is maximized when resistance values approach infinity and capacitance values approach zero.

#### 4.3.4.1 Low Frequency Impedance Magnitude and Phase Angle

Figure 8 and Figure 9 provide box and whisker plots of |Z| and  $\theta$  at 0.1 Hz for each test lining. Each box and whisker plot shows the median as a horizontal line through the box. The lower and upper edge of the box indicate the first and third quartiles of the dataset, respectively, while the lower and upper whisker are the respective minimum and maximum data points. The plot indicates the range of resulting data and relative spread of the data to each other. Highly repeatable data provides a compressed box-and-whisker compared to variable data, which imparts various extended shapes to the box-and-whisker. For example, 11C has a wide box because the data are split equally near the maximum and minimum—three are near 10<sup>8</sup> ohms and three are near 10<sup>6</sup> ohms. By contrast, results with longer whiskers indicate that the data is more evenly spread across the range or that less clustering occurred across the replicate set.

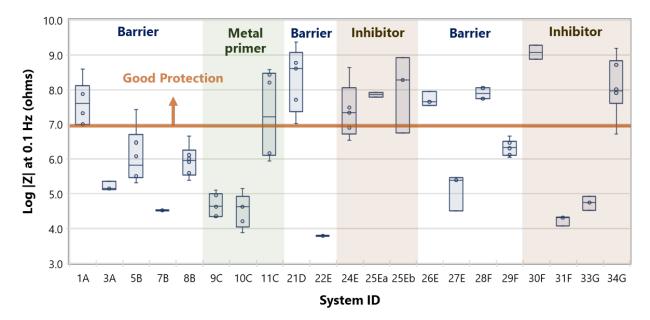


Figure 8.—Log |Z| measurements of Collbran test linings shown in an inclusive median box and whisker plot of all replicates at 0.1 Hz; inner data points and outliers are designated with open circles.

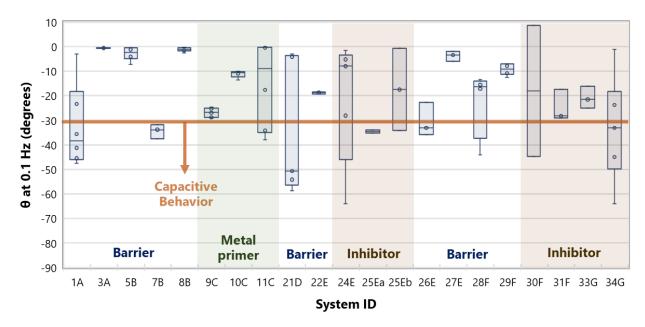


Figure 9.— $\theta$  measurements of Collbran test linings shown in an inclusive median box and whisker plot of all replicates at 0.1 Hz; inner data points and outliers are designated with open circles.

Figure 8 and Figure 9 also incorporate the system type as background shading in the plot. No shading indicates the lining is a barrier coating; these systems have a more straightforward interpretation of the EIS results, in which higher |Z| values generally indicate higher corrosion protection. The systems shaded and labeled as "Metal primer" have a metallic protective coating. The metal primer is assumed to be a conductor in the electrical circuit, meaning a low |Z| value does not necessarily indicate poor corrosion protection. The systems within the shaded "Inhibitors" area contain an inhibitive protection mechanism, such as a red lead primer. Inhibitors are thought to interrupt active corrosion cells by forming protective oxides and similar mechanisms. This protective mechanism also requires more sophisticated interpretation of the EIS spectra than relying on the general rule of, "higher |Z| provides greater corrosion protection." ECM could be used to interpret the EIS spectra fully for the systems containing metal primers and inhibitors.

Figure 8 includes a line at 10<sup>7</sup> ohms. Above this value, lining systems provide strong barrier protection against corrosion. Based on this analysis alone, the highest performing linings are Systems 1A, 11C, 21D, 24E, 25Ea, 25Eb, 26E, 28F, 30F, and 34G. Most of the results show a wide range in the data. Further, these high performers are primarily a mix of barrier and inhibitor coatings. There is one metal primer coating, System 11C, where the system's barrier topcoat is dominating the EIS data and likely protecting the underlying metal primer and steel.

Figure 9 includes a line at -30 deg to aid in interpretation of this plot. Near 0 deg, the resistive elements dominate EIS data at 0.1 Hz. As  $\theta$  becomes more negative, capacitive behavior has an increasing influence. At -45 deg, diffusion-limited corrosion typically occurs, known as Warburg diffusion, and as  $\theta$  approaches -90 deg, capacitive behavior dominates the data. For EIS results

showing a single RC pair for the coating, the capacitive behavior is an indication of good barrier protection. However, this analysis becomes more complex when a second RC pair is needed to model the interface because oxides and/or corrosion reactions are significant contributors.

Compared to Figure 8, many of the same systems meet or exceed the line added as a threshold in Figure 9. Barrier systems which meet the |Z| threshold also meet the  $\theta$  threshold except for System 7B (Class B) which meets only the  $\theta$  threshold. One metal primer system, 11C, met both the |Z| and  $\theta$  thresholds, while System 9C (Class B) nearly met only the  $\theta$  threshold. For the inhibitor systems, Figure 9 shows capacitive behavior is present to some degree for all systems with Systems 31F (Class C) and 33G (Class D) approaching the  $\theta$  threshold. The contributions to the capacitive behavior for each system requires ECM for characterization.

#### 4.3.4.2 Equivalent Circuit Modeling

Researchers performed ECM for select lining systems, targeting those rated as Class A and barrier systems. Additional systems received cursory modeling to investigate the shifting from Class A to lower-rated systems. The primary model utilized included two pairs of RCs. The first pair represents the coating bulk layer as the coating pore resistance ( $R_{pore}$ ) and the coating capacitance ( $CPE_{coat}$ ) as a constant phase element (CPE) to account for the pseudo-capacitive nature of coating materials [5]. The second RC pair is the charge transfer resistance ( $R_{ct}$ ) and the double layer capacitance ( $C_{dl}$ ). It should be emphasized that ECM of field EIS testing is not well documented and further work is needed to confirm that  $R_{ct}$  and  $C_{dl}$  are appropriate elements given that the working electrode is not the steel substrate in a two-cell EIS configuration. Two-cell EIS, therefore, may not be measuring electrochemical reactions. The ECM used in this research also does not account for the second cell as a second model in series with that shown here; however, the results showed a good fit using traditional ECM practices.

Many of the models utilized a CPE as the  $C_{dl}$  element to obtain a better fit, i.e., CPE<sub>dl</sub>. All modeling reported below fixed the solution resistance ( $R_{sol}$ ) at 100 ohms. This element represents the electrolyte resistance for the EIS testing solution. It is presumed to be insignificant for the modeling results because it is several orders of magnitude lower than the resistances of the coating and interface elements of interest. The low value of  $R_{sol}$  creates a challenge both for the instrument to collect precise data and for the modeling itself. For this reason,  $R_{sol}$  was fixed at a ballpark value during modeling execution. Figure 10 provides an ECM example that utilizes a capacitor instead of a CPE for  $C_{dl}$ . The figure also shows the model outputs.

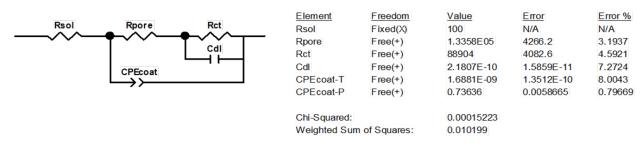


Figure 10.—Example equivalent circuit model and results using a capacitor for the double layer capacitance (CdI) and a fixed value for solution resistance (Rsol).

Each model was run several times while the goodness of fit was qualitatively interpreted on the associated Nyquist Bode plots for |Z| and  $\theta$  across all frequencies measured. For poorer fits, the model was re-run with revisions to the starting values and/or the model elements to improve the overall appearance of the fit. For the final model selected, the error output was also reviewed. Spectra that could not be resolved to an error of 50 % or less for all or most elements were not included in final evaluations. A few exceptions were made where a single model element error was 100 % to 200 %, but the overall goodness of fit for all other elements was exceptional. Data scatter (noise) caused by electromagnetic interference at the lowest measured frequencies was the primary reason for not including an ECM in final evaluations. In these cases, where possible, up to several noisy data points were excluded from the fit to produce in the results reported here.

Figure 11 provides the mean and standard of the model resistors and capacitors for Class A systems 1A, 3A, and 34G (plotted at left), Class B systems 5B, 7B, 21D, 28F, and 25Ea/25Eb combined, and Class C system 27E. All systems are barrier coatings except for 34G and 25Ea/25Eb, which are inhibitors. The reported values are not normalized for 20-cm<sup>2</sup> sample surface area.

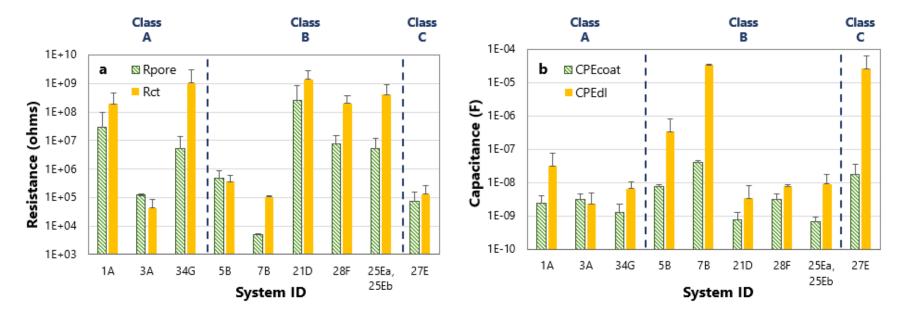


Figure 11.—Equivalent circuit modeling (ECM) results for select lining systems plotted for the mean and standard deviation of the system a) resistors and b) capacitors. The coating elements are represented with hashed bars and the interface elements as solid bars. \*ECM for System 3A utilized a capacitor element for the double layer instead of a CPE. Results are not normalized for the 20-cm<sup>2</sup> test cell area.

The Class A linings 1A and 34 G provide resistance and capacitance consistent with a coating providing good corrosion protection—relatively high resistance and low capacitance. System 1A receives more barrier protection from its  $R_{pore}$  than 34G, which receives most protection from  $R_{ct}$  and may be resulting from the system inhibitors. System 1A has a higher CPE<sub>dl</sub>, which may be caused by a higher fraction of exposed metal surface area [17].

All Class A linings have a CPE<sub>coat</sub> near 10<sup>-9</sup> Farads (F), indicating the intact coating material is a strong barrier and CPE<sub>dl</sub> suggests a low fraction of the surface area is delaminated, compared to other systems [5]. For System 3A, the resistor circuit elements indicate the lining is degraded, i.e., R<sub>pore</sub> and R<sub>ct</sub> suggest low or minimal corrosion protection. The EIS results may suggest that a lower rating would be more accurate for System 3A in evaluating the resistors alone, but the system capacitors reveal a strong barrier to corrosion protection for most of the underlying steel [5].

Of the Class B systems, EIS results suggest Systems 21D, 28F, and 25Ea/25Eb provide the highest corrosion protection and are on par with System 1A and 34G. The  $CPE_{coat}$  for 21D and 25Ea/25Eb indicate these linings materials are the highest barriers in the study. System 21D also showed the highest R<sub>pore</sub> and R<sub>ct</sub> as well as the second lowest fractional area of exposed metal (after system 3A) per  $CPE_{dl}$  values. System 21D also had the second lowest  $CPE_{coat}$  (after 25Ea/25Eb). Assuming sampling for defect-free EIS testing locations was consistently applied, the EIS modeling results suggest that unblistered and undamaged lining material of System 21D provides the highest level of corrosion protection to the underlying steel—of those modeled.

System 28F is moderately high performing, with results similar to 25Ea/25Eb for all elements except the CPE<sub>coat</sub>. The CPE<sub>coat</sub> value for 28F is most similar to that of System 3A, which demonstrated low performance with low R<sub>pore</sub> and R<sub>ct</sub> values. Systems 5B and 7B both demonstrated low performance in EIS testing. System 5B has low R<sub>pore</sub> and R<sub>ct</sub>, both in the  $10^6$  ohms range. The CPE<sub>coat</sub> for 5B suggests a lower performing barrier and at  $10^{-8}$  F, and the CPE<sub>dl</sub> is approaching  $10^{-6}$  F, suggesting moderate fractional metal exposure. System 7B is the poorest lining material evaluated by ECM with R<sub>pore</sub> less than  $10^4$  ohms and R<sub>ct</sub> at  $10^5$  ohms. The lining material itself showed a CPE<sub>coat</sub> approaching  $10^{-7}$  F, and the C<sub>dl</sub> suggests significant delamination at more than  $10^{-5}$  F.

System 27E is the only Class C lining evaluated by ECM. The results are similar to System 7B, but 27E shows greater corrosion protection with a larger  $R_{pore}$  value.

A subsequent analysis of the four circuit elements in the ECM calculated the coefficient of variation (CV) across all modeled systems, taking the mean and standard deviation of each System mean in Figure 11. Table 5 provides these results along with the minimum and maximum value for each circuit element. The first observation is that resistive elements produce a much greater variation than the capacitive elements. Further, the coating elements,  $R_{pore}$  and  $CPE_{coat}$ , result in a greater variation of performance compared to the interface elements,  $R_{ct}$  and  $CPE_{dl}$ . The application of these observations is unknown and could be further investigated for value to interpreting EIS results of significantly aged materials, such as for field EIS testing and laboratory EIS testing to coating failure.

Table 5 also includes the minimum and maximum calculated mean values, reporting the system for each. Recall corrosion protection is maximized when resistance values approach infinity and capacitance values approach zero. The results reiterate the previous discussion, demonstrating that System 3A and 7B the poorest resistances and System 7B had the poorest capacitances. The highest performers were System 21D and 25Ea / 25Eb, noting that System 3A also produced a low C<sub>dl</sub> result.

Table 5.—Summary statistics for the ECM calculated mean values showing coefficient of variation (CV), minimum, and maximum

Element	CV (%)	Minimum (ohms / F)*	Maximum (ohms / F)*	
R <sub>pore</sub>	217	5.0·10 <sup>3</sup> (System 7B)	2.6·10 <sup>8</sup> (System 21D)	
R <sub>ct</sub>	136	4.3·10 <sup>4</sup> (System 3A)	1.4·10 <sup>9</sup> (System 21D)	
CPE <sub>coat</sub>	62	7.0·10 <sup>-10</sup> (System 25Ea/25Eb)	4.1·10 <sup>-8</sup> (System 7B)	
CPE <sub>dl</sub>	39	3.5·10 <sup>-9</sup> (System 21D) <sup>^</sup>	3.6·10 <sup>-5</sup> (System 7B)	

\* Resistors reported in ohms and capacitors reported in Farads

^ The System 3A interface was modeled as a capacitor and had a slightly lower double layer capacitance at 2.3·10<sup>-9</sup> F.

Attempts were made to perform ECM on Systems 8B, 22E, 29F, and 30F. Each system requires a different ECM than the two RC pairs utilized in the previous examples. Therefore, results provide less direct comparison and were excluded. Systems 8B (Class B) and 22E (Class D) can be modeled using a single RC pair and appeared to be modeling the corrosion cell with no influence from a barrier lining, i.e., a achieves a good fit with only R<sub>ct</sub> and CPE<sub>dl</sub>. For System 8B, this could be a result of the microcracking reported from the visual inspection. System 29F (Class C) appeared to be a single RC pair needing a Warburg element to model diffusion-controlled reactions at the interface. This is shown in Figure 12 with Nyquist and Bode plots for System 29F using an ECM containing two RC pairs. The good overall fit except for the low frequency "tail" suggests that a single RC pair and Warburg element would provide an improved fit.

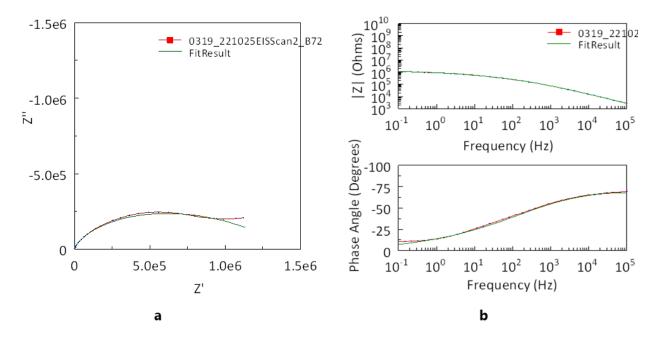


Figure 12.—Example ECM showing data and fit results for System 29F via a) Nyquist and b) Bode plots using two RC pairs. The Nyquist plot shows real impedance (Z') on the x-axis and imaginary impedance (Z'') on the y-axis. The good overall fit except for the low frequency "tail" suggests that a single RC pair and Warburg element would provide an improved fit.

System 30F (Class B) is an inhibitor system and required a single capacitor-resistor pair that appeared to be dominated by the protective coating ( $R_{pore}$  and  $CPE_{coat}$ ) as opposed to the corroding interface shown by Systems 8B and 22E. The ECM observations utilized the two measurements made at the 5:30 position. As shown in Figure 13, System 30F was unique in the EIS testing results in that it produced multiple spectra with non-typical data and noise at the lower frequencies. Specifically, the  $\theta$  values less than -90 deg cannot be modeled with physical electrical elements. No cause was identified for these spectra features. Inspectors made multiple attempts to collect data absent of these features.

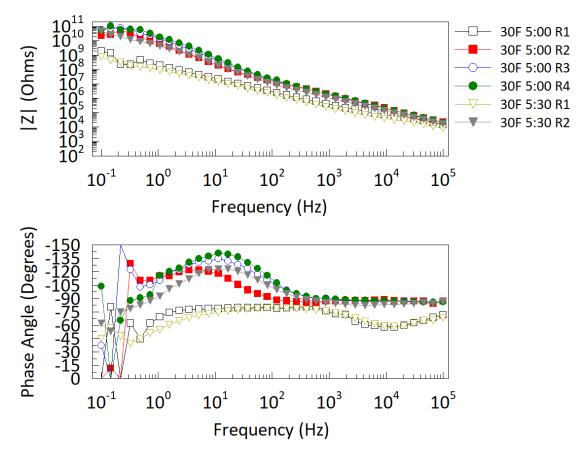


Figure 13.—EIS Bode plot results for System 30F demonstrating values less than -90 that were not modeled because an appropriate physical electrical element does not exist. The y-axes were expanded to show all data for this system.

#### 4.4 Summary of Findings

The Collbran study was fortunate to have the pipe interiors abrasive blast cleaned to white metal and the linings shop applied under controlled conditions. The controlled environment of shop applications generally produces the best outcomes for coating service life. Several linings continue to provide good corrosion protection after 63 years. This is likely a result of good surface preparation before coating application as well as low hygrothermal stress for this buried pipe in a constant immersion service environment.

Visual inspection results showed that three lining systems at Collbran remained a Class A rating after 63 years, ten linings were Class B, six linings were Class C, and nine were rated Class D. This is a significant reduction in ratings from the 10-year inspection in which 25 linings were rated Class A.

Researchers utilized field EIS testing per ASTM D8370 to evaluate the dielectric properties of defect-free samples for each lining system. The analysis included a general review of the full EIS spectra, statistics for |Z| and  $\theta$  at 0.1 Hz, and ECM analysis of select systems as two pairs of RCs.

The results suggest that System 21D and 25Ea/25Eb provide the highest overall corrosion protection, as demonstrated by good dielectric performance, i.e., high resistance and low capacitance values. Systems 1A, 34G, and 28F also showed good dielectric performance per ECM.

ECM results of EIS data indicated that Systems 3A and 7B showed the poorest resistance values and System 7B had the poorest capacitances. Despite the low resistance, System 3A showed a low  $CPE_{coat}$  and  $C_{dl}$  values, suggesting a good barrier coating and low fractional area of the coating is delaminated, suggesting the lining remains well-bonded to the substrate.

ECM of Systems 8B, 22E, 29F, and 30F required a model other than two RC pairs, being better fitted with one RC pair with or without a Warburg element. A single RC pair indicates only one material layer, either the coating or an interfacial layer such as an oxide between the substrate and lining, is contributing to corrosion protection. A Warburg element indicates diffusion-controlled reactions. These results provide less direct comparison to the linings with two RC pairs and further modeling efforts were not pursued. Future work could evaluate all EIS data in greater detail.

### 5. Lessons Learned

The 2019 inspection at Shasta did not include detailed visual inspection data for each lining and instead generalized the lining through the test section as being in poor condition, noting several spots of spalled or missing coating and substrate corrosion present [14]. Examination of inspection photos during this research showed the test linings to be in visually acceptable condition adjacent to EIS testing.

The limited photographic evidence and compromised EIS data obtained at Shasta directly informed the practices for subsequent coatings inspections. In particular, the Collbran inspection conducted in 2022 focused on proper EIS test cell set up and thorough documentation of observations for each lining system. Reclamation inspectors acquired multiple photographs showing the condition of each lining system, taken from near and far range and documenting specific flaws with size references.

A class rating for direct comparison of Shasta test linings to those at Collbran cannot be given without being able to evaluate the full test lining condition. The EIS testing of System 7 (CA-50 or CTE) provided the highest |Z| values near  $10^7$  ohms, followed by System 8-1 (phenolic red lead with zinc-based inhibitive pigments, direct to metal) near  $10^6$  ohms. These results showed capacitive behavior that would require ECM to further characterize. Nonetheless, the Shasta EIS results combined with the Collbran EIS results show particular systems containing coal-tar, red lead, vinyl, chlorinated rubber, or epoxy mastic can achieve corrosion protection for over 50 years of immersion service.

Results generally suggest that Collbran linings are in better condition than Shasta, with 13 of the remaining 28 linings (46%) receiving a visual rating of Class A or B. For 46% of the lining systems to exhibit no or minimal damage after 63 years (except for repairs made along the invert

to mitigate erosion damage) is a remarkable outcome. In addition to the ten-year age difference between Shasta and Collbran linings, researchers attribute the performance at Collbran to two factors: the test linings were applied in a shop setting, presumably providing the opportunity for better surface preparation and improved environmental controls; and consistent service conditions that created less hygrothermal stresses. Of these factors, improving surface preparation and application conditions provides the greatest opportunity for increasing corrosion protection at Reclamation and beyond. There may also be opportunities to incorporate the lessons learned about hygrothermal stresses. For example, future coating developments can focus efforts to improve resistance to damage from cycles of wetting/drying and temperature fluctuation.

Combining the visual inspection results with field EIS data helps to advance understanding of coating performance as well as the field EIS data itself. Table 6 separates lining systems according to the number of its ECM model values above 10<sup>7</sup> ohms or below 10<sup>8</sup> F, for resistive or capacitive elements, respectively. These threshold values are subjectively chosen to separate the linings into two groups, denoting whether the particular ECM element value was among one of the better electrochemical characteristics (marked with an X) or not (left blank). These results are further sorted according to the visual classification of each system so the linings with the best combined outcomes appear at the top of the list. The ranking system highlights 21D (VR-3 vinyl), 1A (chlorinated rubber), and 34G (metal conditioner and VR-3 vinyl) as the top three performers at Collbran, followed by 28F(epoxy mastic) and 25Ea/25Eb (inhibitive primer and VR-3 vinyl).

System ID	Class	R <sub>pore</sub> > 10 <sup>7</sup> ohms	R <sub>ct</sub> > 10 <sup>7</sup> ohms	CPE <sub>coat</sub> < 10 <sup>8</sup> F	CPE <sub>dl</sub> < 10 <sup>8</sup> F
21D	В	Х	Х	Х	Х
1A	А	Х	Х	Х	
34G	А		Х	Х	Х
28F	В		Х	Х	Х
25Ea/25eb	С		Х	Х	Х
3A	А			Х	Х
5B	В			Х	
27E	С				Х
7B	В				

Table 6.— Collbran test linings ranked in order of number of favorable ECM results (denoted with an X) and visual inspection classification

Two of the three test linings at Collbran that presented no visible damage across the entire test section, Class A designation, had ECM results suggesting good corrosion protection:

- 1A (chlorinated rubber)
- 34G (metal conditioner and VR-3 vinyl)

These two Class A linings have excellent capacitive behavior with  $CPE_{coat}$  (near  $10^{-9}$  F) indicating the intact coating material is a strong barrier and  $CPE_{dl}$  (in the range of  $10^{-7}$  F to  $10^{-8}$  F), which may suggest that a high fraction of the linings remain bonded to the substrate [19]. Recall these and the following results are not normalized for the 20-cm<sup>2</sup> test cell area.

Of the Collbran linings rated Class B by visual inspection, three had ECM results suggesting good corrosion protection:

- 21D (VR-3 vinyl)
- 25Ea/25Eb (inhibitive primer and VR-3 vinyl)
- 28F (epoxy mastic)

ECM results of these three linings may help to assess and develop new coating systems with longer service lifetimes. Most notably, the capacitors modeled for these systems had values in the range of  $10^{-8}$  F to  $10^{-9}$  F or lower. The capacitor values increased for the poorest performing systems, with CPE<sub>coat</sub> values greater than  $10^{-8}$  F and CPE<sub>dl</sub> greater than  $10^{-5}$  F. The results suggest the best differentiating factor for coatings performance evaluation may be CPE<sub>dl</sub>, a reflection of the level of bonding between the coating and substrate.

Three of the systems visually rated as Class A or B had ECM results suggesting poor or diminishing corrosion protection:

- 3A (coal tar epoxy)
- 5B (coal tar polyurethane)
- 7B (coal tar epoxy)

As an example, the good visual results for System 7B (coal tar epoxy) are in stark contrast to the ECM results suggesting poor corrosion protection. More research is needed to determine how field EIS testing results should inform coating maintenance decisions.

Another interesting outcome of the Collbran evaluations is the comparison of coal tar epoxybased coatings. Although comprised of similar materials, System 3A (Class A) appeared visually superior to System 7B (Class B) and 6B (Class C) and had better capacitive properties than the ECM results for System 7B. This observation is a reminder of the complex nature of coating formulations and slight changes in components, ratios, or number of coats affects long-term performance.

#### **5.1 Recommendations and Future Work**

The Collbran inspection incorporated the field EIS lessons learned from the Shasta inspection. Further, the Collbran inspection applied the new ASTM D8370 standard test method published in 2022. The result provided a distinct improvement in data quality and validation. Industry validation is needed on how to best apply field EIS data from aged coatings for practical use. Specific areas for future work from this research include:

- System 7B EIS data indicated poor corrosion protection, while a visual examination of the coating surface suggests a good, intact coating. Experimental validation is needed to identify thresholds for EIS data and/or ECM results and to correlate them to corrosion rate, pitting, or other metal loss mechanisms occurring beneath the coating.
- ECM of field EIS testing requires further research to confirm if the traditional R<sub>ct</sub> and CPE<sub>dl</sub> elements are appropriate. The field EIS method utilizes two test cells and no connection to substrate. Therefore, the working electrode is the second test cell instead of the steel substrate, and the ability for the measurement to measure electrochemical reactions at the interface is debatable [8].
- ECM often resulted in a particularly broad range for R<sub>pore</sub>. Experimental validation is needed to verify whether a broad range of data is caused by non-uniform coating degradation. If so, it would be helpful to understand if the damage mechanism can be determined, i.e., cracking, polymer degradation, etc.
- Technologies have advanced since the conception of the Historic Linings project. Scientific equipment now exists that is capable of easily providing combined visual and elemental analysis at the scale of interfaces between coatings and their substrates. Coupling EIS investigations on long-term panels with microscopic assessment of corrosion under the coating would allow researchers to correlate specific ECM results to physical observations using techniques such as quantitative elemental mapping. This work would progress the understanding of how steel corrosion products, which form as the performance of a coating system wanes, may affect long-term corrosion protection of aging coatings.

### 6. Summary and Conclusions

Researchers analyzed data collected during a 2019 inspection which included photographs and field EIS data from six of the original 26 tested lining systems at Shasta. The inspection photos showed good visual coating appearance in the cleaned areas used for field EIS testing. The EIS results showed low to moderate corrosion protection. All surviving Shasta test linings showed some level of capacitive behavior. ECM could help determine whether the capacitive response comes from the coating, the interface, or some combination of both.

Collbran data collection centered around a 2022 inspection which incorporated traditional visual inspection methods, DFT and UT measurements, and field EIS testing. Visual inspections found 13 of 28 lining systems exhibit no or minimal damage after 63 years. EIS data was combined with visual inspection results to investigate how dielectric characteristics might be related to the visual appearance of the test linings.

Researchers analyzed a subset of Collbran field EIS data using ECM. The results showed decreased resistance and increased capacitance values for the poorest performing systems. Of the two system capacitors,  $CPE_{dl}$ , related to disbonded surface area at the coating/steel interface, varied by more orders of magnitude than any other element modeled. This suggests the double layer capacitance results may provide a strong differentiating factor for comparative coating performance evaluation.

The findings from Shasta and Collbran together demonstrate certain formulations containing coal-tar, red lead or vinyl can achieve corrosion protection for over 50 years of service. Additionally, Collbran showed lining systems comprised of chlorinated rubber or epoxy mastic can also provide many decades of defense for immersed steel structures. Before pursuing coating development efforts based on these results, the authors must first determine the material properties and other physical characteristics of the successful linings, then correlate ECM results to the attributes which contributed to longevity.

The preliminary field EIS results presented here show that ECM methods can aid analysis of field EIS data, but correct interpretation and validation is essential to reaching meaningful and actionable conclusions. Expanding ECM proficiencies within Reclamation requires external guidance from experts within the EIS field. Employing modern analytical techniques will be key to verifying ECM results.

Future work includes:

- Validating ECM for two-cell field EIS data.
- Determining material (electrical, thermal, mechanical) properties and physical characteristics (adhesive performance, rust creep resistance, etc.) of successful coatings materials.
- Correlating ECM results with
  - o material properties, physical characteristics, and
  - o corrosion rates and degradation mechanisms.
- Validating ECM results experimentally with quantitative techniques such as elemental mapping of long-term coating test panels.
- Identifying acceptance thresholds for field EIS data and/or ECM results.

# 7. Project Data

- Share Drive folder name and path where data are stored: T:\Jobs\DO\\_NonFeature\Science and Technology\2022-PRG-Historic Linings
- Point of contact: Meredith Heilig, <a href="mailto:mheilig@usbr.gov">mheilig@usbr.gov</a>, 720-467-1735
- Short description of the data: visual inspection records and photographs, DFT measurement data, UT measurement data, EIS data, ECM data
- Keywords: corrosion inspection, protective coatings, field EIS, ECM
- Approximate total size of all files: 9 MB

## 8. References

- [1] J. L. Kiewit, "Field Tests of Water Pipe Linings: A Summary of 15th-year Shasta and 10th-year Collbran Performance; Report No. REC-ERC-72-1," Bureau of Reclamation, Denver, CO, 1972.
- [2] T. M. Tenczar and C. E. Selander, "Eighth Year Evaluation of Experimental Test Linings in Shasta Dam Penstock; Report No. P-72," Bureau of Reclamation, Denver, 1958.
- [3] J. L. Kiewit, "First-Year Laboratory and Field Inspection of Experimental Protective Linings Southside Canal Collbran Project, Colorado; Report No. P-89," Bureau of Reclamation, Denver, CO, 1962.
- [4] F. Mansfield, "Use of electrochemical impedance spectroscopy for the study of corrosion protection by polymer coatings," J. Appl Electrochem, vol. 25, pp. 187-202, 1995.
- [5] D. Loveday, P. Peterson and B. Rodgers, "Evaluation of Organic Coatings with Electrochemical Impedance Spectroscopy. Part 2: Application of EIS to Coatings," JCT Coatings Tech, no. October, pp. 88-93, 2004.
- [6] D.-H. Xia, C.-M. Deng, D. MacDonald, S. Jamali, D. Mills, J.-L. Luo, M. G. Strebl, M. Amiri, W. Jin, S. Song and W. Hu, "Electrochemical measurements used for assessment of corrosion and protection of metallic materials in the field: A critical review," J. Mater. Science Technology., vol. 112, pp. 151-183, 2022.
- [7] A. Trentin, A. Pakseresht, A. Duran, Y. Castro and G. Dusan, "Electrochemical Characterization of Polymeric Coatings for Corrosion Protection: A Review of Advances and Perspectives," Polymers, vol. 14, no. 12, p. 2306, 2022.

- [8] ASTM D8370-22, "Standard Test Method for Field Measurement of Electrochemical Impedance on Coatings and Linings," ASTM International, West Conshohocken, PA, 2022.
- [9] F. L. Floyd, S. Avudaiappan, J. Gibson, B. Mehta, P. Smith, T. Provder and J. Escarsega, "Using electrochemical impedance spectroscopy to predict the corrosion resistance of unexposed coated metal panels," Progress in Organic Coatings, vol. 66, no. 1, pp. 8-34, 2009.
- [10] D. Loveday, P. Peterson and B. Rodgers, "Evaluation of Organic Coatings with Electrochemical Impedance Spectroscopy Part 1: Fundamentals of Electrochemical Impedance Spectroscopy," JCT CoatingsTech, no. August, pp. 46-52, 2004.
- [11] B. Merten, "Electrochemical Impedance Methods to Assess Coatings for Corrosion Protection, Report No. 8540-2019-03," Bureau of Reclamation, Denver, 2019.
- [12] J. L. a. U. H. K. Kiewit, "Fifteenth Year Evaluation of Experimental Test Linings in Shasta Dam Penstock Final Summary Report; Report No. ChE-59," Bureau of Reclamation, Denver, 1966.
- [13] A. Skaja, "Shasta Dam Unit 5 Penstock Historic Linings Field Test Site; Report No. ST-2022-22024-01," Bureau of Reclamation, Denver, 2023.
- [14] C. Paulson and C. Gulsvig, "Shasta Dam Unit 5 Penstock Internal Inspection; Report No. 8540-2019-11," Bureau of Reclamation, Denver, 2019.
- [15] M. Cleveland and B. Merten, "Temperature effects on two-cell field impedance testing of epoxy and epoxy polysulfide coatings," CORROSION, vol. 78, no. 11, pp. 1079-1086, 2022.
- [16] J. L. Kiewit, "Application of Experimental Protective Linings Southside Canal -Collbran Project Colorado; Report No. P-83," Bureau of Reclamation, Denver, CO, 1960.
- [17] B. J. Merten, M. Heilig and M. Jermyn, "Laboratory Investigation of Coating Impedance in Controlled Environments Using a Two-Cell Field Test Method," in AMPP Annual Conference + Expo, Denver, CO, 2023.
- [18] J. R. Scully, "Electrochemical Impedance of Organic-Coated Steel: Correlation of Impedance Parameters with Long-Term Coating Deterioration," Journal of the Electrochemical Society, vol. 136, no. 4, pp. 979-990, 1989.
- [19] H. Xiao and F. Mansfield, "Evaluation of Coating Degradation with Electrochemical Impedance Spectroscopy and Electrochemical Noise Analysis," Journal of The Electrochemical Society, vol. 141, pp. 2332-2337, 1994.
- [20] J. L. Kiewit, "First-year laboratory and field inspection of experimental protective linings - Southside Canal - Collbran Project, Colorado; Report No. P-89," Bureau of Reclamation, Denver, 1962.
- [21] J. L. Kiewit, "Application of Experimental Protective Linings Southside Canal -Collbran Project, Colorado; Report No. P-83," Bureau of Reclamation, Denver, 1960.

- [22] J. L. Kiewit and H. K. Uyeda, "Fourth-year laboratory and field inspection of experimental protective linings - Southside Canal - Collbran Project, Colorado; Report No. ChE-58," Bureau of Reclamation, Denver, 1966.
- [23] R. C. Bacon and J. J. Smith, "Electrolytic Resistance in Evaluating Protective Merit of Coatings on Metals," Industrial & Engineering Chemicals, vol. 40, no. 1, pp. 161-167, 1948.

### **Appendix A – Shasta EIS Bode Plots**

#### Shasta Section 5 – VR-3 vinyl

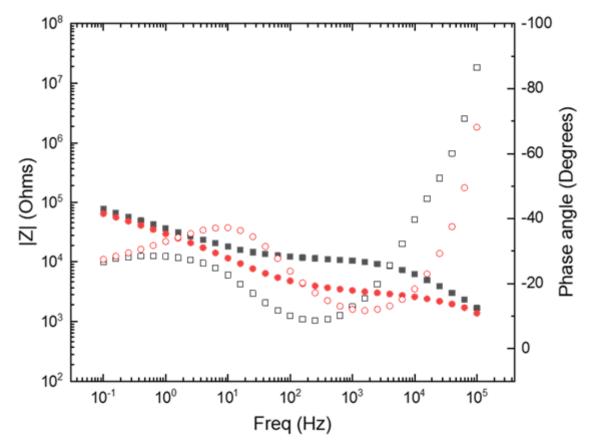
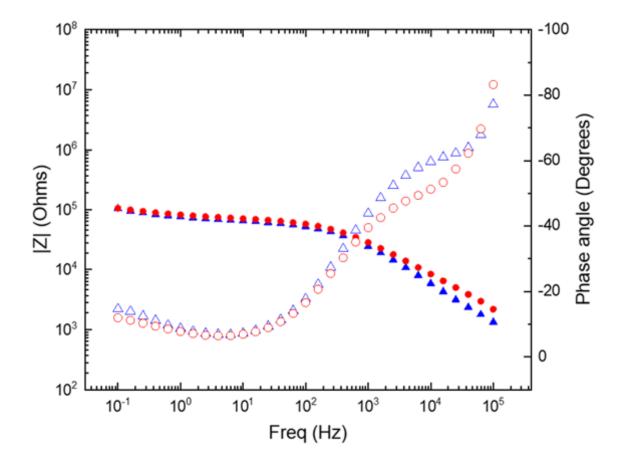
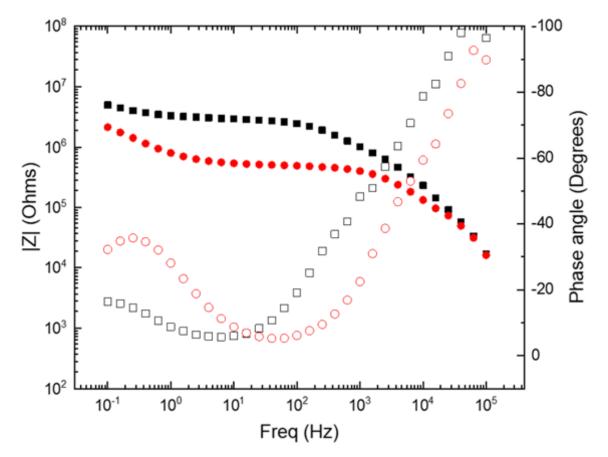


Figure A-1.— EIS Bode plot from Shasta Section 5 - VR-3 vinyl. |Z| values are solid markers and  $\theta$  values are open markers.



#### Shasta Section 6 – VR-6 vinyl

Figure A-2.—EIS Bode Plot from Shasta Section 6 – VR-6 vinyl. |Z| values are solid markers and  $\theta$  values are open markers.



#### Shasta Section 7 – coal tar CA-50 or CTE

Figure A-3.—EIS Bode plot for Shasta Section 7 - coal tar CA-50 or CTE. |Z| values are solid markers and  $\theta$  values are open markers.

# Shasta Section 8-1 – red lead phenolic with zinc-based inhibitive pigments direct to metal

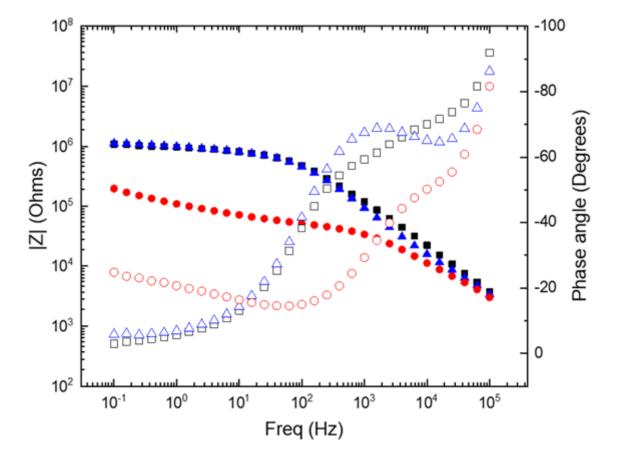


Figure A-4.— EIS Bode plot for Shasta Section 8-1 - red lead phenolic with zinc-based inhibitive pigments direct to metal. |Z| values are solid markers and  $\theta$  values are open markers.

# Shasta Section 8-2 (or 8A-2) – red lead phenolic (or red lead phenolic with zinc-based inhibitive pigment) with oil primer

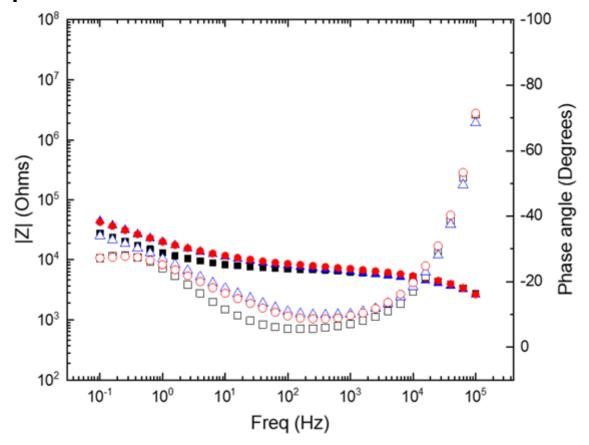
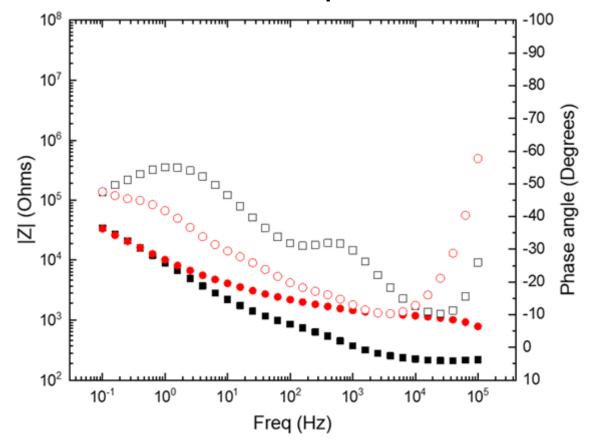


Figure A-5.—1EIS Bode Plot for Shasta Section 8-2 (or 8A-2) – red lead phenolic (or red lead phenolic with zinc-based inhibitive pigment) with oil primer. Solid markers are |Z| and open markers are  $\theta$ .



Shasta Section 8A-1 – red lead phenolic direct to metal

Figure A-6.—EIS Bode plot for Shasta Section 8A-1 - red lead phenolic direct to metal. Solid markers are |Z| and open markers are  $\theta$ .

### **Appendix B – Collbran EIS Bode Plots**

#### **Collbran System 1A – Chlorinated Rubber**

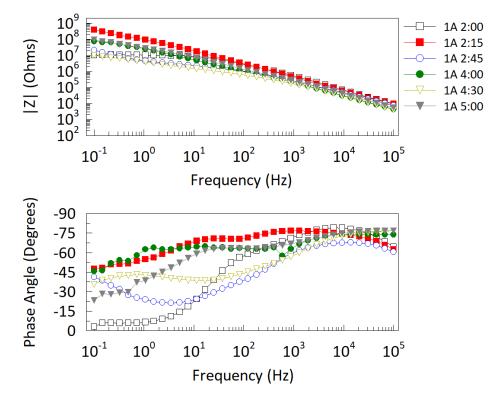
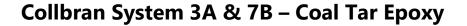


Figure B-1.—EIS Bode plot from Collbran System 1A – chlorinated rubber. The recorded lining surface temperature was 54 °F during the measurements.



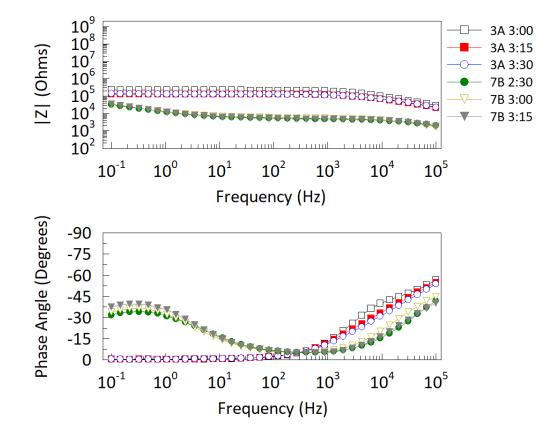


Figure B-2.—EIS Bode plot from Collbran Systems 3A and 7B – coal tar epoxy. The recorded lining surface temperatures were 55 °F and 52 °F, respectively, during the measurements.



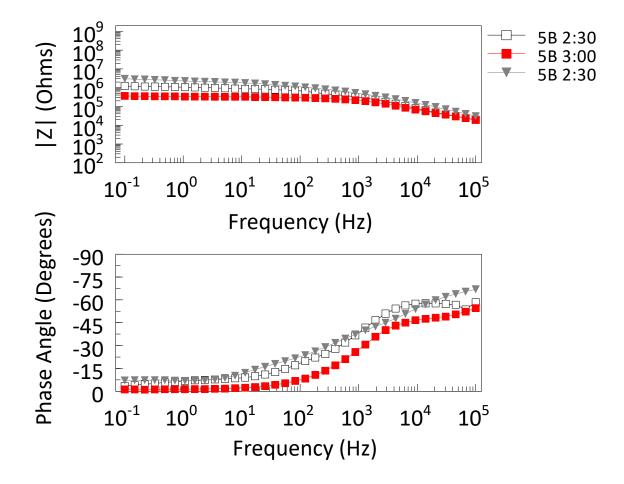


Figure B-3.—EIS Bode plot from Collbran System 5B – coal tar polyurethane. The recorded lining surface temperature was 58 °F during the measurements.

#### **Collbran System 8B – Phenolic with Mica Primer and Phenolic**

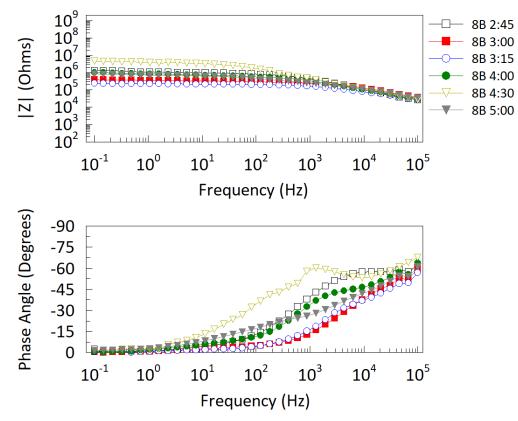


Figure B-4.—EIS Bode plot from Collbran System 8B – phenolic with mica primer and phenolic. The recorded lining surface temperature was 52 °F during the measurements.

#### Collbran System 9C – Aluminum metal and Vinyl Butyral Primer and Vinyl Alkyl Aluminum

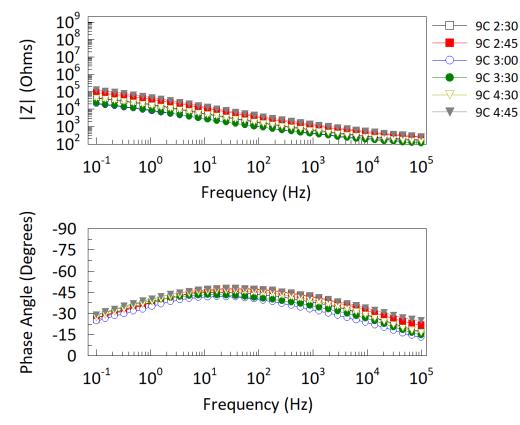


Figure B-5.— EIS Bode plot from aluminum metal and vinyl butyral primer and vinyl alkyl aluminum. The recorded lining surface temperature was 52 °F during the measurements.

#### Collbran System 10C – Zinc Metal and Vinyl Butyral Primer and Vinyl Alkyl Aluminum

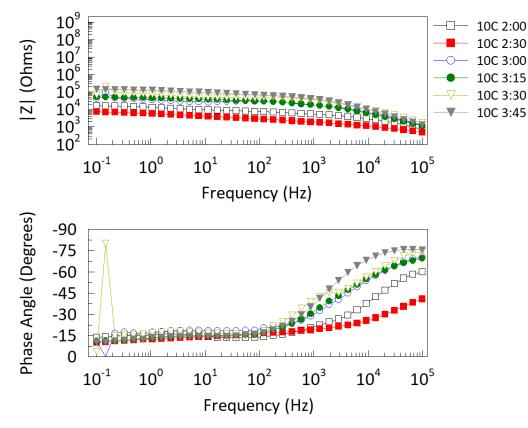


Figure B-6.—EIS Bode plot from Collbran System 10C – zinc metal and vinyl butyral primer and vinyl alkyl aluminum. The recorded lining surface temperature was 52 °F during the measurements.

# Collbran System 11C – Zinc Metal and Thinned VR-3 Vinyl and VR-3 Vinyl Seal

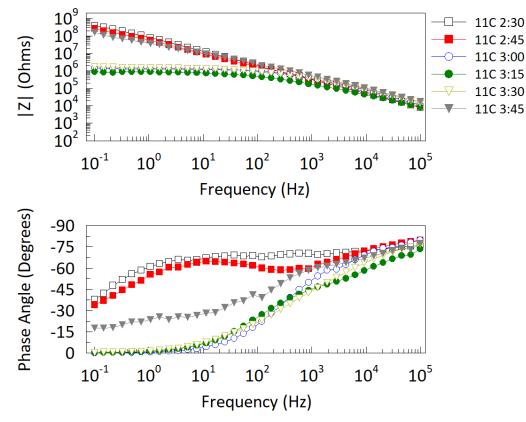


Figure B-7.— EIS Bode plot from Collbran System 11C – zinc metal and thinned VR-3 vinyl and VR-3 vinyl seal. The recorded lining surface temperature was 51 °F during the measurements.

#### Collbran System 21D – VR-3 Vinyl

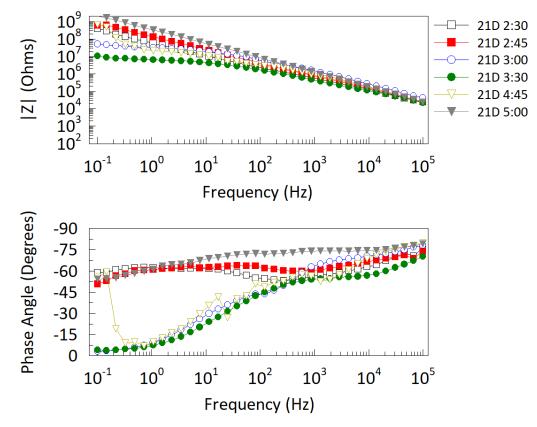


Figure B-8.— EIS Bode plot from Collbran System 21D – VR-3 vinyl. The recorded lining surface temperature was 51 °F during the measurements.

# Collbran Systems 22E / 27E – Chlorinated Rubber Primer and Neoprene / Neoprene

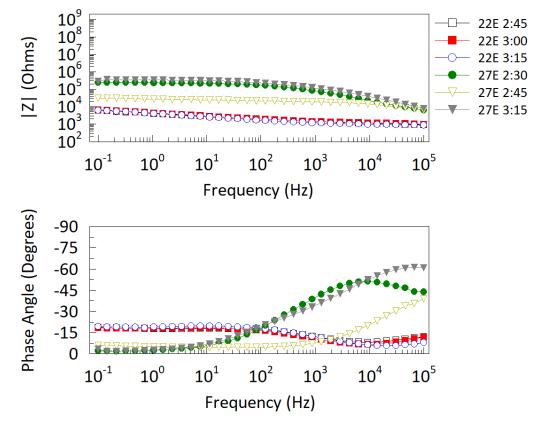


Figure B-9.—EIS Bode plot from Collbran Systems 22E / 27E – chlorinated rubber primer and neoprene or neoprene. The recorded lining surface temperature was 50 °F during the measurements.

#### Collbran System 24E – Chlorinated Rubber Primer with Inhibitive Pigment and Liquid Neoprene and Chlorosulfonated Polyethylene (Aluminum)

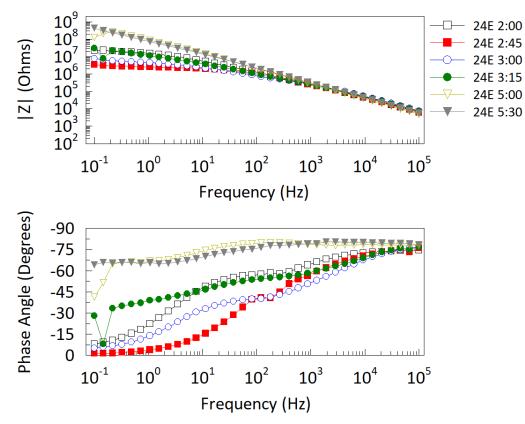


Figure B-10.—EIS Bode plot from Collbran System 24E – chlorinated rubber primer with inhibitive pigment and liquid neoprene and chlorosulfonated polyethylene (aluminum). The recorded lining surface temperature was 51 °F during the measurements.

#### Collbran System 25Ea & 25Eb – Inhibitive Primer and VR-3 Vinyl

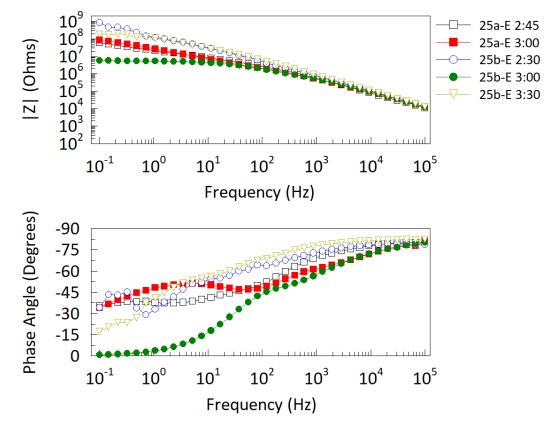


Figure B-11.—EIS Bode plot from Collbran System 25Ea & 25Eb – inhibitive primer and VR-3 vinyl. The recorded lining surface temperature was 51 °F during the measurements.

#### **Collbran System 26E – Vinyl Wash Primer and Neoprene**

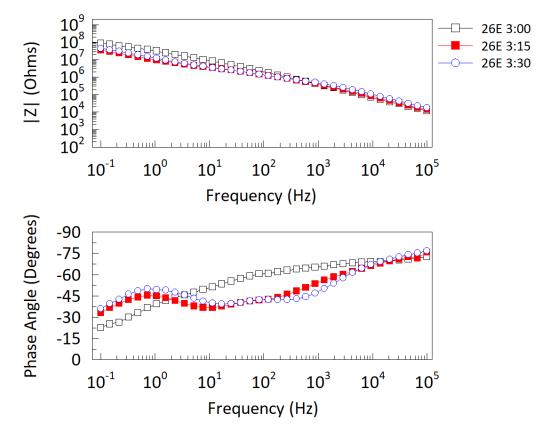
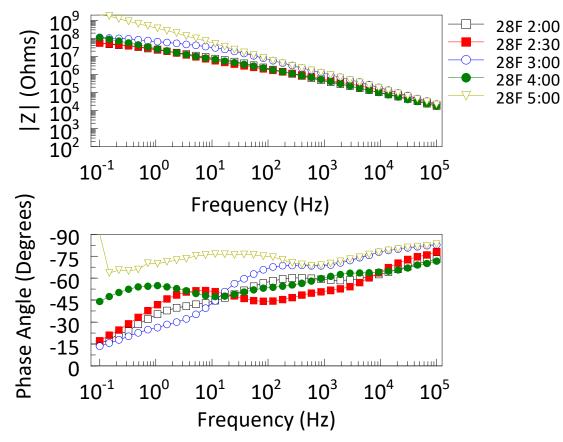


Figure B-12.—EIS Bode plot from Collbran System 26E – vinyl wash primer and neoprene. The recorded lining surface temperature was 51 °F during the measurements.



#### **Collbran System 28F – Epoxy Mastic**

Figure B-13.—EIS Bode plot from Collbran System 28F – epoxy mastic. The recorded lining surface temperature was 50 °F during the measurements.

#### **Collbran System 29F – Vinyl Mastic**

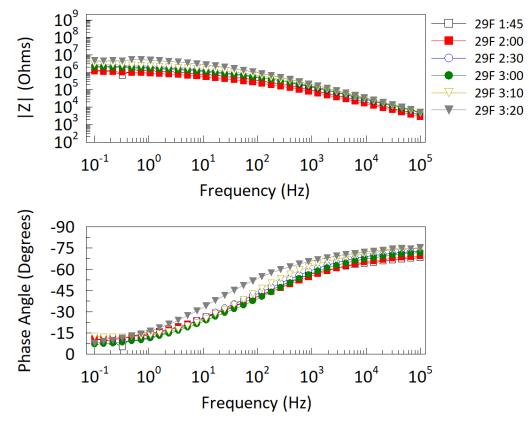


Figure B-14.—EIS Bode plot from Collbran System 29F – vinyl mastic. The recorded lining surface temperature was 50 °F during the measurements.

#### **Collbran System 30F – Vinyl Wash Primer and Vinyl Red Lead**

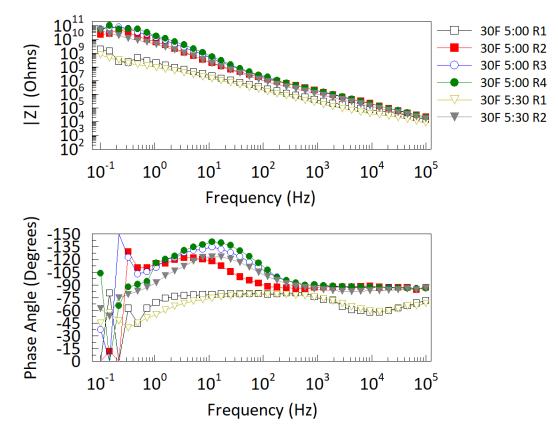


Figure B-15.—EIS Bode plot from Collbran System 30F – vinyl wash primer and vinyl red lead—note the expanded y-axis to show all data. The recorded lining surface temperature was 48 °F during the measurements.

# Collbran Systems 31F / 33G – Vinyl Wash Primer / Metal Conditioner and Phenolic Red Lead

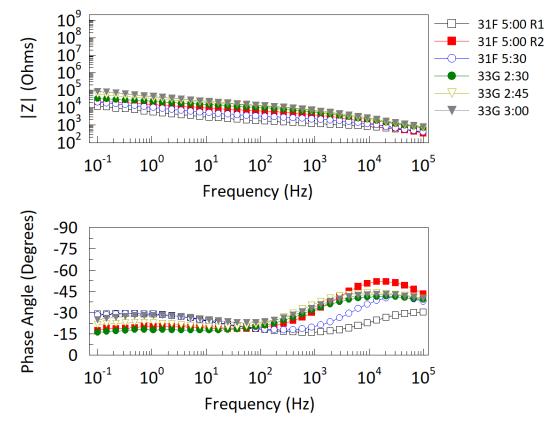


Figure B-16.—EIS Bode plot from Collbran Systems 31F or 33G – vinyl wash primer or metal conditioner and phenolic red lead. The recorded lining surface temperature was 48 °F during the measurements.



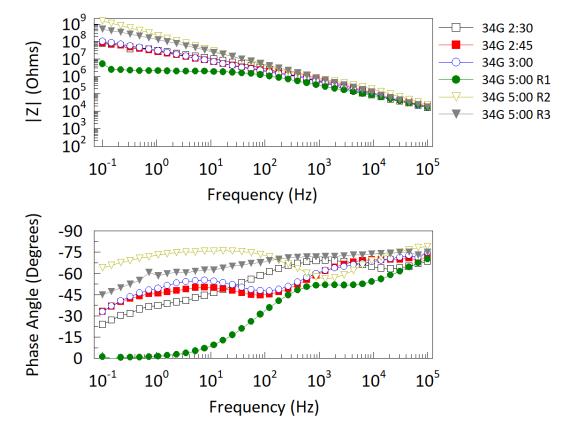


Figure B-17.— EIS Bode plot from Collbran System 34G – metal conditioner and VR-3 vinyl. The recorded lining surface temperature was 49 °F during the measurements.