Lab testing of CONDUCRETE per IEC 62561-7

Submitted To: **SAE Inc.**
19 Churchill Drive
Barrie, ON L4N 8Z5
Attn: Mr. Rylan Boyd
705-715-7382
rboyd@saeinc.com

Submitted By: **Powertech Labs Inc.**
12388 88th Ave.
Surrey, BC, Canada V3W 7R7
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Prepared By:

Shima Karimi
____
Shima Karimi, EIT
Engineer, Applied Materials
Phone: 604-598-5016
Email: Shima.Karimi@powertechlabs.com

Reviewed By:

Earl Mayuga
____
Earl Mayuga, EIT
Engineer, Asset Management
Phone: 604-590-7406
Email: earl.mayuga@powertechlabs.com

Patrick Ho., P.Eng.
Senior Engineer, Applied Materials
Phone: 604-590-6694
Email: patrick.ho@powertechlabs.com

1.0 INTRODUCTION

SAE requested Powertech Labs Inc. (Powertech) to conduct laboratory tests per IEC 62561-7, "*Lightning protection system components (LPSC) – Part 7: Requirements for earthing enhancing compounds"* on its CONDUCRETE product. Leaching test, ICP, resistivity and LPR measurements were performed. The details of the tests and results are described below.

2.0 EXPERIMENTAL SET-UP

Three samples were prepared for each test. Conducrete was received in a powder form in the 55 lb bags. For resistivity, OCP and LPR measurements, Conducrete was mixed by following these steps:

1- Slowly add 2.497 kg (1/10 of a bag) of Conducrete to 1.135 L (1/10 of 3 US gallons) of deionized water
2- Manually mix them in a bucket with a steel bar until all the powedered conducrete was blended with the water and no dry powder exists.
3- The slurry was mixed so as to avoid the entrenchment of air during the mixing process.
4- Pour the slurry into plastic molds from the bucket. Ladles or other small containers may be used to transfer the slurry from the bucket to the plastic molds.
5- Place electrodes into the slurry for the OCP and LPR tests
6- Let the slurry cure for 21 days before OCP, LPR and resistivity measurements

PH of the slurry was 12.9 at 22.7 °C.

The goal and sample preparation for the leaching test, ICP, resistivity measurements, OCP and ICP analyses are explained as follows.

2.1 Leaching Test

The goal of leaching test was to determine concentrations of leachable ions (Fe (iron), Cu (copper), Zn (zinc), Ni (nickel), Cd (cadmium), Co (cobalt), Pb (lead)). Procedures in EN 12457-2 and EN 12506 standards were followed.

2.2 ICP Analyses

ICP analyses were conducted to measure sulfur concentration utilizing 3050B method. It is similar to ISO 4689-3 or ISO 14869-1 except the tumbler rotation speed was 28 rpm.

2.3 Resistivity Measurements

The electrical resistivity of the Conducrete was measured after 21 days of curing as per the manufacturer's instruction. The test procedure utilized the Wenner 4-pin method as described in ASTM G57-06. The equipment utilized for the resistivity measurements were as follows:

- M.C. Miller Co. Miller 400D Resistance Meter (Calibrated 04/2017);
- Tinker & Rasor SB-1 four-electrode soil box.

As per IEC 62561-7, three samples of Conducrete were prepared for resistivity testing, and were labelled as S1, S2 and S3. The samples were mixed as per the manufacturer's instructions and placed into three Tinker & Rasor SB-1 soil boxes, with dimensions of 4cm x 6cm x 23.75cm. The inside pin spacing was 18.3cm. A Miller 400D resistance meter, most recently calibrated in April 2017, was used to measure the electrical resistance of the sample. Electrical resistivity was calculated as follows:

$$\rho = R\frac{A}{a}$$

Where:

ρ: Sample resistivity (Ω cm)
R: Measured resistance (Ω)
A: Cross sectional area of the container perpendicular to the current flow (cm^2)
A: Inner electrode spacing, measured from inner edges of electrodes (cm)

2.4 Open Circuit Potential (OCP) and Linear Polarization Resistance (LPR) Measurements

OCP measurements were carried out for 5 minutes to monitor the potential-time behavior of the galvanized steel electrode in Conducrete and measure the open circuit potential (E_{ocp}) before LPR tests. The purpose of LPR tests was to find corrosion rate of galvanized steel in Conducrete.

OCP and LPR measurements were performed with Princeton Applied Research PARSTAT 4000 potentiostat/galvanostat using a conventional three-electrode electrochemical as per ASTM G59-97. A rod of zinc-galvanized low-carbon steel with a length of 25 cm and diameter of 1 cm was utilized as a working electrode. The counter electrode was a conductive graphite rod with a length of 15.24 cm and diameter of 1.27 cm. They were purchased from McMaster-Carr. An Ag/AgCl ([Cl-] = 4 M) reference electrode used as a reference electrode.

The galvanized steel rods were washed with a soap solution, rinsed with deionized water, and dried in air. Once the Conducrete slurry was prepared and poured into a plastic mold, the steel and graphite rods were dipped into the slurry to embed them into the Conducrete during the curing time. A 12 cm long plastic test tube with a diameter of 1.5 cm was embedded into the slurry to make a spot for a reference electrode. To keep the distance of electrodes according to ASTM G59-97, wooden jigs were made to secure the electrodes during curing periods. The distance between the reference electrode and working electrode was 2 mm and the distance between the working electrode and the counter electrode was 4 cm. A sample of LPR test is presented in Figure 1.

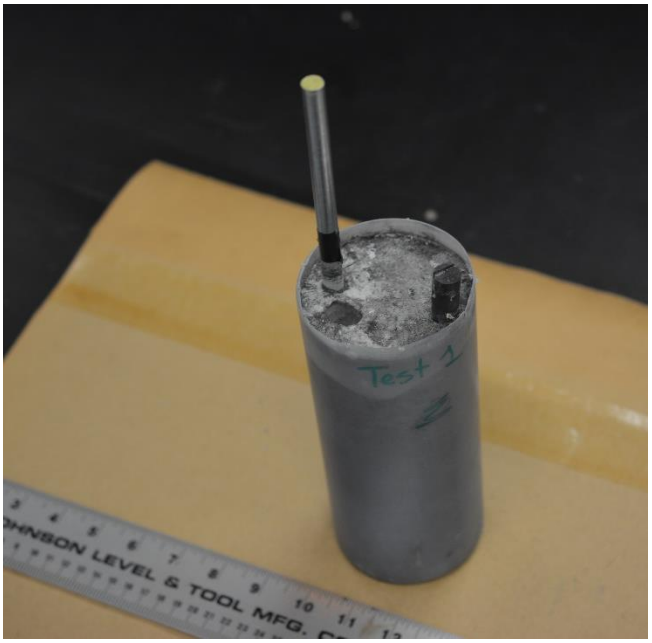


Figure 1: Image of the sample for LPR tests of galvanized steel embedded in Conducrete

Approximately 12 cm of the steel and 2.5 cm of the graphite rods were left exposed out of the slurry for making electrical connections during electrochemical tests.

Electrochemical corrosion tests were started with 5 minutes of OCP measurements followed by LPR tests. Polarization resistance of the samples was measured at ± 30 mV vs. OCP with a potential sweep rate of 0.1667 mV s^{-1}. Also, potentiodynamic polarization tests were conducted to find Tafel constants. Potentiodynamic polarization experiments were conducted in a potential range of -0.2 V vs. OCP to 0.2 V vs. OCP with a scan rate of 0.167 mV s^{-1}. The EIS measurements were performed with the amplitude of 10 mV at OCP in the frequency range of 1 to 10,000 Hz and a sampling rate of 10 points per decade.

3.0 RESULTS

3.1 Leaching Test

The result of the leaching analysis indicates that Aluminum (0.68 mg/L), Barium (1.74 mg/L), and Zinc (0.06 mg/L) are the leachable ions from Conducrete. The leaching test results including DI water leach blank is reported in Appendix A.

3.2 ICP Analyses

The measured concentration of sulphur in Conducrete is less than the detection limit of sulphur by ICP-OES analysis. The detection limit of sulphur is 1.0 mg/kg by the ICP-OES analysis. Therefore, sulphur concentration of Conducrete is less than 1.0 mg/kg.

3.3 Electrical Resistivity

Electrical resistivity measurements were recorded from each sample in a dry state, and in a saturated state. As stated in ASTM G57, the saturated measurement will provide an as approaching minimum resistivity, and can be usefully compared with "as-received" resistivity measurements. Deionized water was added to the sample and resistivity measurements were recorded over time. The samples were considered saturated when the resistivity measurements stabilized. The measured resistivity values are as follows:

Table 1: Electrical Resistivity Measurements

Sample No.	ASTM G57 Resistivity (ohm-cm)	
	Dry	Saturated
S1	1587	367
S2	1692	358
S3	1967	358

The minimum Conducrete resistivity achieved in the dry state was 1,587 ohm-cm. The minimum resistivity achieved in the saturated state was 358 ohm-cm.

3.4 OCP and Linear Polarization Measurements

Samples are labelled as test 1, test 2, and test 3. The surface area of each working electrode were calculated and used to find current density.

The OCP reached to -1.229, -1.246, and -1.34 V vs Cu/CuSo4 for test 1, test 2, and test 3 after five minutes. The variation of OCP over time is shown in Appendix B.

In the LPR tests, ΔE(t) is defined with respect to the corrosion potential ($\Delta E = E - E_{corr}$). The polarization resistance Rp can be defined by plotting ΔE(t) versus current density. The slope of the plot indicates Rp value defined by equation 1.

$$R_P = \left(\frac{\partial \Delta E}{\partial\, i}\right) i = 0 \qquad \text{Equation 1}$$

ΔE versus current plots are presented in Appendix C. Rp values in ohm were determined by the VersaStudio software. They were converted to ohm m^2 by multiplying them to the surface area (m^2) of samples. Noises were detected for the LPR results of the sample 2. The presence of noise due to a bubble trapped in the reference electrode. The rest of electrochemical corrosion experiments were noise free.

It is stated in ASTM G102-89 that the effect of solution resistance on the linear polarization resistance can be corrected by Equation 2 where Ra is the obtained polarization resistance (ohm m^2), ρ is the electrolyte resistivity (ohm m) and l is the distance between the specimen electrode and the reference electrode.

$$R_P = R_a - \rho l \qquad \text{Equation 2}$$

Electrolyte (Conducrete) resistivity is 16.92 ohm m (from soil resistivity measurment) and l is 0.002 m. The value of Ra, Rp, and its deviation are summarized in Table 2.

Table 2: Values of surface area, obtained polarization resistance from I-E curves, and polarization resistance for sample 1, 2 and 3 tested at room temperature

Test	Surface area (cm2)	Ra (ohm m2)	Ra Deviation	RP (ohm m2)
1	81.68	4.00	6.74E-06	3.97
2	79.80	3.89	2.84E-05	3.85
3	81.05	1.74	9.08E-06	1.71

To find corrosion current density, i_{corr}, Stern-Geary coefficient, B, is required (Equation 3). The Stern-Geary coefficient can be obtained from Equation 4.

$$i_{corr} = \frac{B}{R_P} \qquad \text{Equation 3}$$

$$B = \frac{\beta_a \cdot \beta_c}{2.303\,(\beta_a + \beta_c)} \qquad \text{Equation 4}$$

The anodic, βa, and cathodic, βc, Tafel slopes were determined by conducting potentiodynamic polarization tests. The units of the Tafel slopes are V/decade. The potentiodynamic polarization curves for sample 1, 2 and 3 along with their anodic and cathodic Tafel slopes are presented in Appendix D.

Table 3: The calculated values of anodic Tafel slope, cathodic Tafel slope, their deviations, Stern-Geary coefficient, and corrosion current density

Test	βa (V/decade)	βa deviation	βc (V/decade)	βc deviation	B (V)	i_{corr} (μA cm^{-2})
1	0.294	0.083	0.084	0.048	0.028	0.707
2	0.244	0.073	0.149	0.057	0.040	1.032
3	0.060	0.091	0.041	0.036	0.011	0.604

4.0 SUMMARY OF RESULTS

The detected leaching ions are Aluminum, Barium, and Zinc. The amount of the released Barium, Aluminum, and Zinc is 0.000174 %, 0.000068 %, and 0.000006 %. The amount of sulphur is less than 0.0001% in Conducrete.

The polarization resistance after 21 days of curing is 3.97, 3.85, and 1.71 ohm m^2 for sample 1, 2 and 3, respectively.

The minimum Conducrete resistivity achieved in the dry state was 1,587 ohm-cm. The minimum resistivity achieved in the saturated state was 358 ohm-cm.

Revision History:

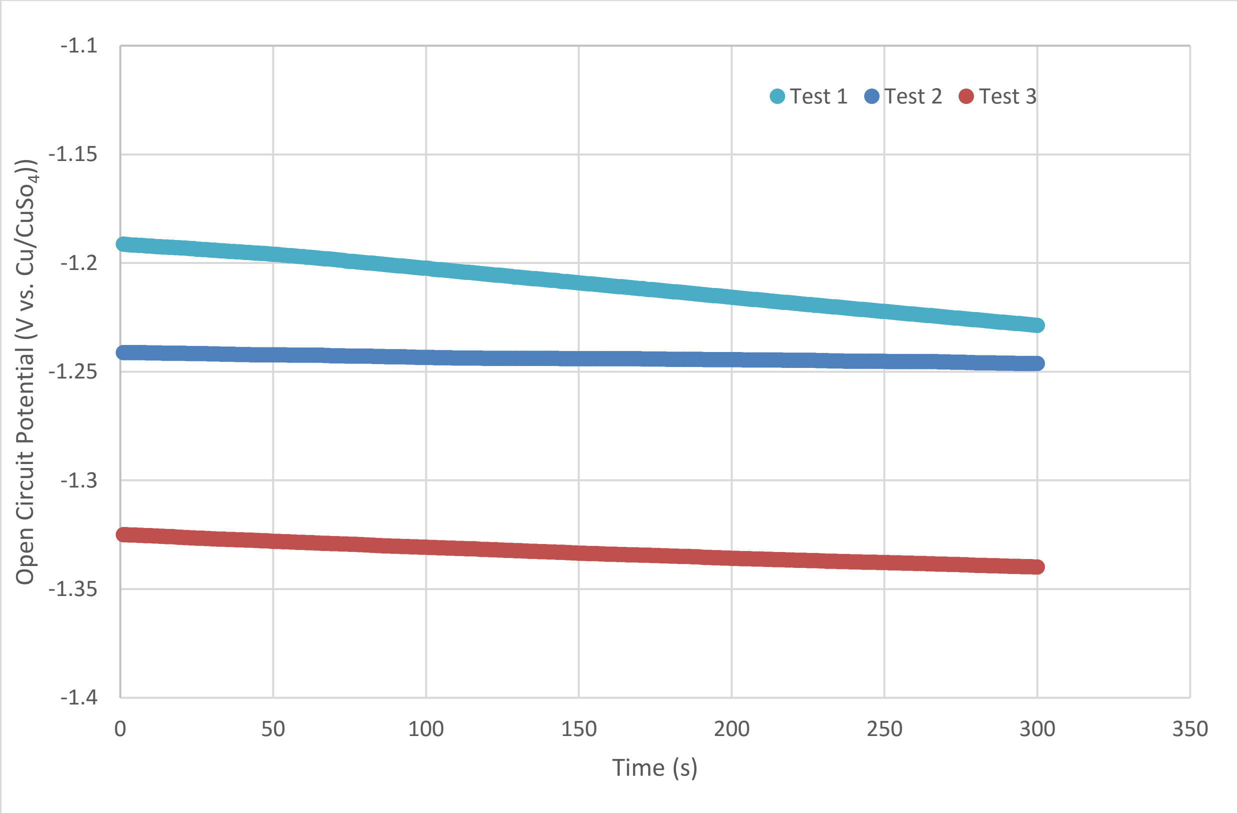
Revision number	Document Date	Description of Change
0	2017-06-09	New document.

Appendix A: Results of leaching tests and ICP-OES analysis to determine leachable ions and sulfur concentration of Conducrete

LAB #					1700333-01	1700333-02	-	-
SAMPLED DATE					05/16/2017	05/25/2017	-	-
MATRIX					Soil	Soil	-	-
SAMPLE ID	Method	Detection Limit	Units	Analysis Date	Powder Sample	Leachate Blank	-	-
Metals - Leachable								
Aluminum	BC MOE	0.20	mg/L	2017-05-26	0.68	<0.20	-	-
Antimony	BC MOE	0.20	mg/L	2017-05-26	<0.20	<0.20	-	-
Arsenic	BC MOE	0.20	mg/L	2017-05-26	<0.20	<0.20	-	-
Barium	BC MOE	0.05	mg/L	2017-05-26	1.74	<0.05	-	-
Beryllium	BC MOE	0.01	mg/L	2017-05-26	<0.01	<0.01	-	-
Boron	BC MOE	1.00	mg/L	2017-05-26	<1.00	<1.00	-	-
Cadmium	BC MOE	0.01	mg/L	2017-05-26	<0.01	<0.01	-	-
Chromium	BC MOE	0.04	mg/L	2017-05-26	<0.04	<0.04	-	-
Cobalt	BC MOE	0.02	mg/L	2017-05-26	<0.02	<0.02	-	-
Copper	BC MOE	0.01	mg/L	2017-05-26	<0.01	<0.01	-	-
Iron	BC MOE	0.20	mg/L	2017-05-26	<0.20	<0.20	-	-
Lead	BC MOE	0.05	mg/L	2017-05-26	<0.05	<0.05	-	-
Manganese	BC MOE	0.02	mg/L	2017-05-26	<0.02	<0.02	-	-
Mercury	BC MOE	0.10	mg/L	2017-05-26	<0.10	<0.10	-	-
Molybdenum	BC MOE	0.03	mg/L	2017-05-26	<0.03	<0.03	-	-
Nickel	BC MOE	0.02	mg/L	2017-05-26	<0.02	<0.02	-	-
Selenium	BC MOE	0.20	mg/L	2017-05-26	<0.20	<0.20	-	-
Silver	BC MOE	0.02	mg/L	2017-05-26	<0.02	<0.02	-	-
Tin	BC MOE	0.30	mg/L	2017-05-26	<0.30	<0.30	-	-
Uranium	BC MOE	10.0	mg/L	2017-05-26	<10.0	<10.0	-	-
Vanadium	BC MOE	0.03	mg/L	2017-05-26	<0.03	<0.03	-	-
Zinc	BC MOE	0.03	mg/L	2017-05-26	0.15	0.09	-	-
Metals by ICP								
Sulphur	EPA 3050/6010	1.0	mg/kg	2017-05-18	<1.0	-	-	-

Special Notes

Appendix B: OCP values of galvanized steel embedded in Conducrete for 21 days- 5 minutes in room temperature



Appendix C: LPR results for galvanized steel embedded in Conducrete for 21 days

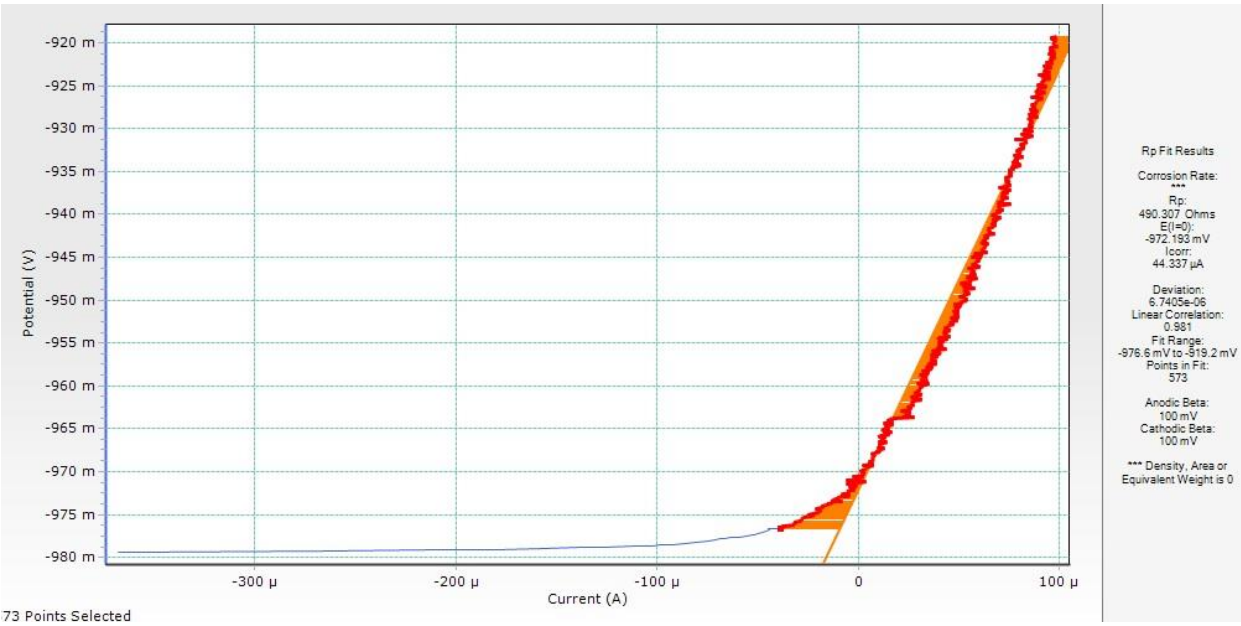


Figure 2: Linear polarization curve and the polarization resistance (Rp) fits for Test 1 for a galvanized steel rod embedded in Conducrete for 21 days-LPR tests were conducted at room temperature

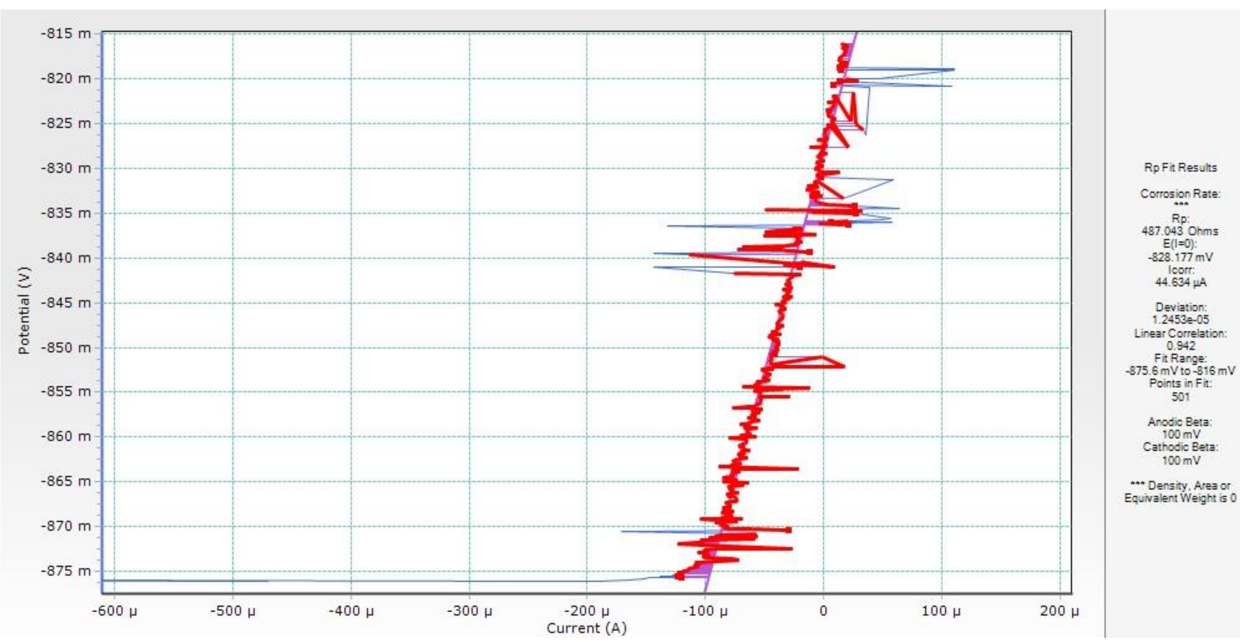


Figure 3: Linear polarization curve and the polarization resistance (Rp) fits for Test 2 for a galvanized steel rod embedded in Conducrete for 21 days-LPR tests were conducted at room temperature

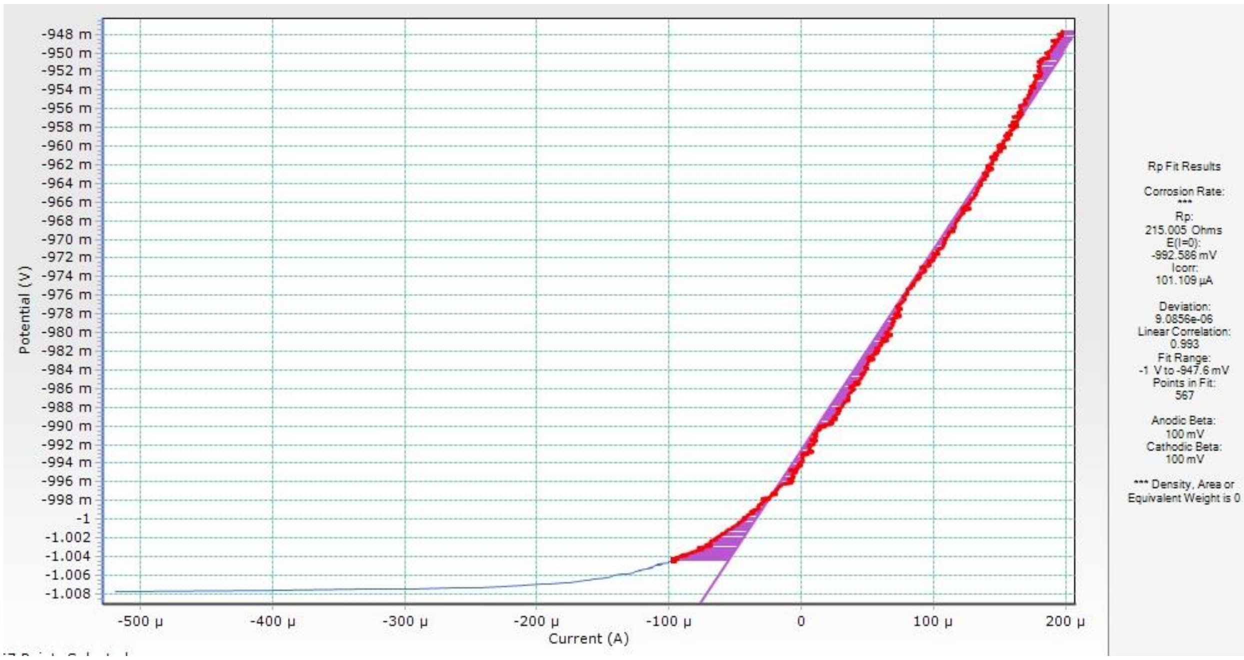


Figure 4: Linear polarization curve and the polarization resistance (Rp) fits for Test 3 for a galvanized steel rod embedded in Conducrete for 21 days-LPR tests were conducted at room temperature

Appendix D: Potentiodynamic polarization results for galvanized steel embedded in Conducrete for 21 days

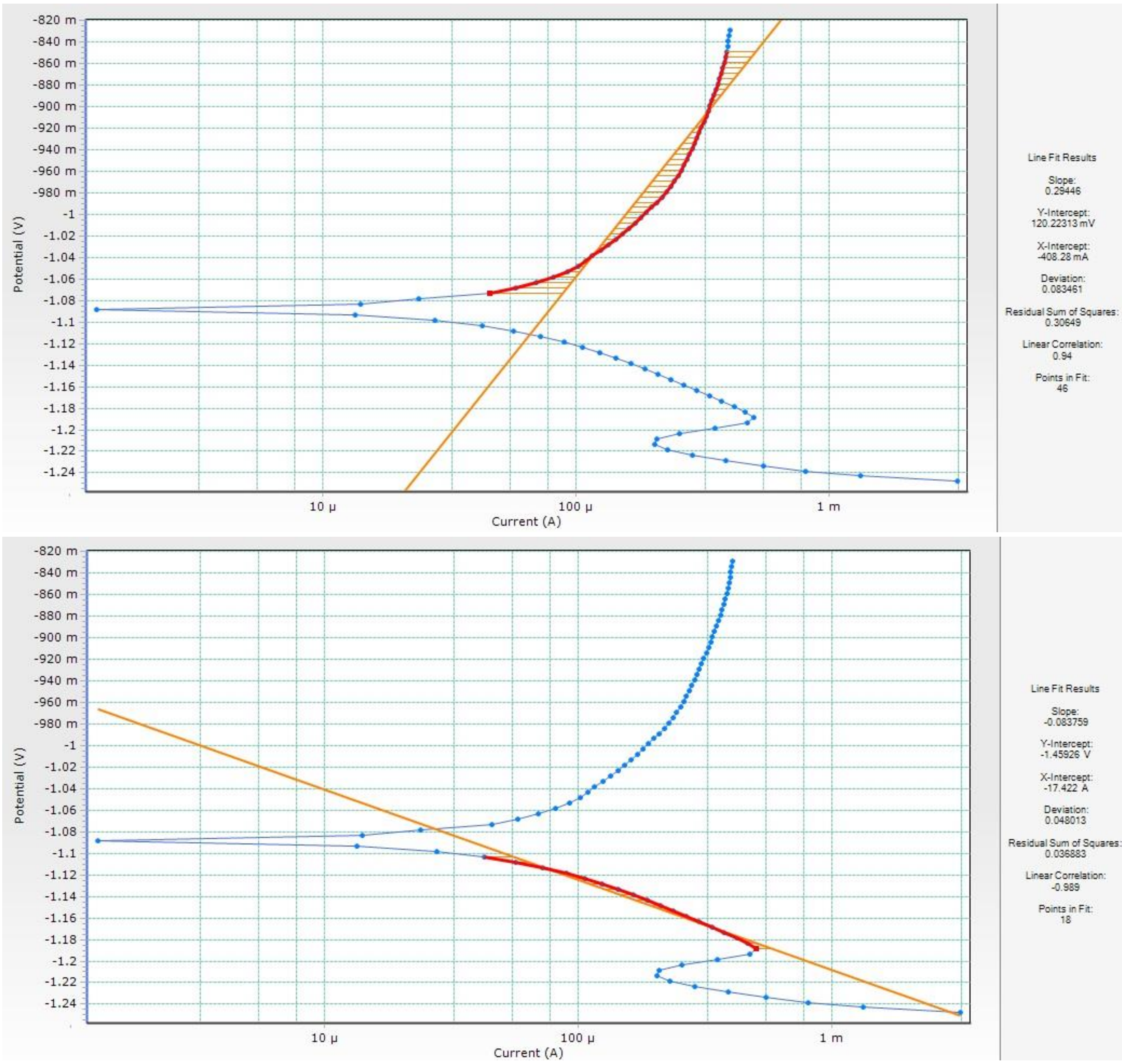


Figure 5: Potentiodynamic polarization curve, line fit results for the anodic Tafel and cathodic Tafel regions of Test 1 for a galvanized steel rod embedded in Conducrete for 21 days

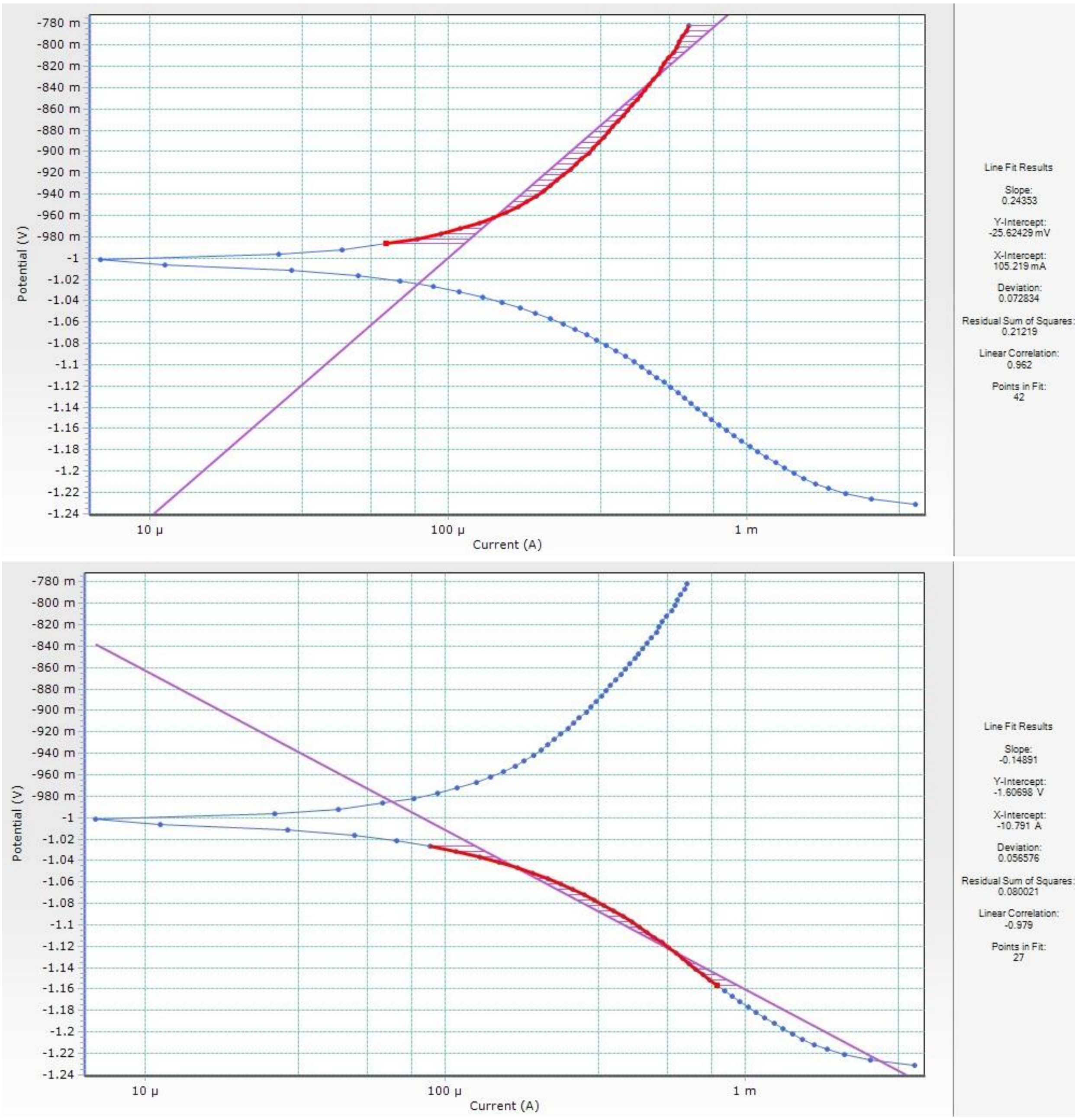


Figure 6: Potentiodynamic polarization curve, line fit results for the anodic Tafel and cathodic Tafel regions of Test 1 for a galvanized steel rod embedded in Conducrete for 21 days

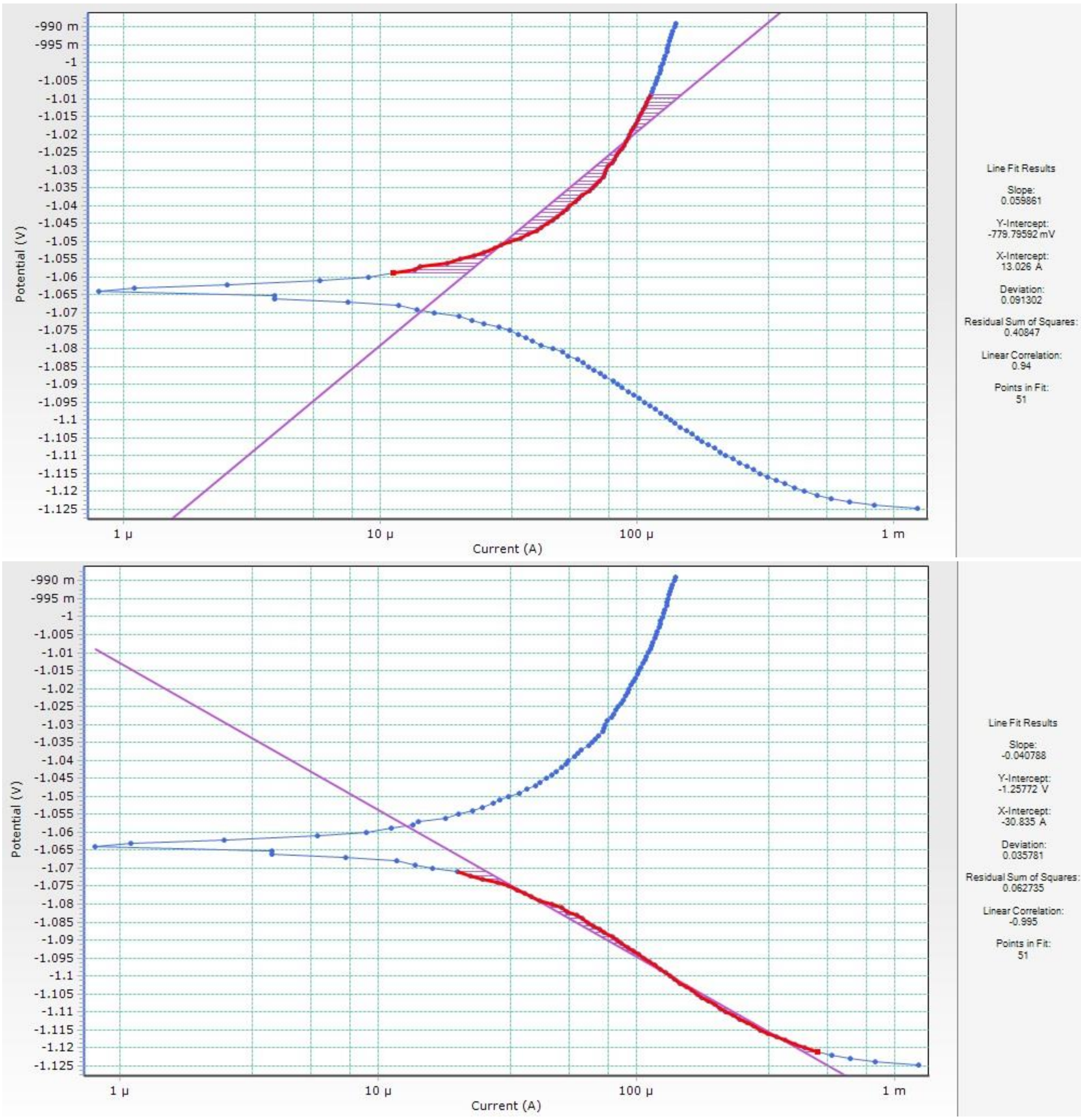


Figure 7: Potentiodynamic polarization curve, line fit results for the anodic Tafel and cathodic Tafel regions of Test 1 for a galvanized steel rod embedded in Conducrete for 21 days