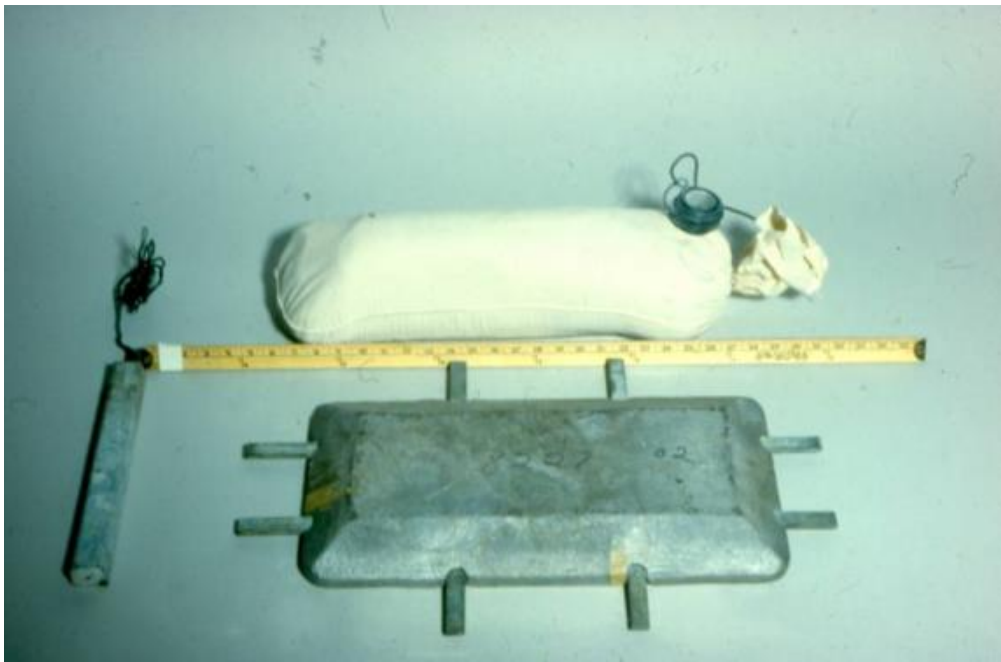


Potentiodynamic Polarization Testing to Confirm the Suitability of Zinc Anodes in Natural Waters Specific to Reclamation

Research and Development Office
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**U.S. Department of the Interior
Bureau of Reclamation
Research and Development Office**

April 2016

Mission Statements

The U.S. Department of the Interior protects America's natural resources and heritage, honors our cultures and tribal communities, and supplies the energy to power our future.

The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

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Potentiodynamic Polarization Testing to Confirm the Suitability of Zinc Anodes in
Natural Waters Specific to Reclamation

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Technical Memorandum No. 8540-2016-07

**Potentiodynamic Polarization Testing to
Confirm the Suitability of Zinc Anodes in
Natural Waters Specific to Reclamation**


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Abbreviations and Acronyms

°C	degrees Celsius
ASTM	ASTM International, formerly known as the American Society for Testing and Materials
CSE	copper sulfate reference cell
MCL	Materials and Corrosion Laboratory
mg/L	milligrams per liter
mV	millivolts
SCE	saturated calomel reference cell

Definitions

autogeneous corrosion	self-corrosion or corrosion not related to providing galvanic current
Cl ⁻	chloride ion
current density	current per unit surface area
driving potential	voltage available from an anode for cathodically polarizing the metal to be protected
HCO ₃ ⁻	bicarbonate
K ₂ SO ₄	potassium sulfate
NaCl:	sodium chloride
NaHCO ₃	sodium bicarbonate
NaNO ₃	sodium nitrate
NO ₃ ⁻	nitrate
Noble potential shift	shift to more positive or less negative potential
SO ₄ ⁻²	sulfate
Zn(OH) ₂	zinc hydroxide
ZnNH ₄ PO ₄	zinc ammonium phosphate
ZnO	zinc oxide

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Executive Summary

Cathodic protection protects a metal electrochemically by making it a cathode (i.e., location where reduction reactions predominate). Employed in conjunction with a bonded dielectric coating, it is an economical way to mitigate corrosion of structures, such as those within Reclamation. Galvanic anode cathodic protection, in which a more active metal is electrically coupled to a less active one, is a form of cathodic protection requiring no outside power source. Zinc and magnesium are two such commonly used anode materials.

In soil, zinc anodes are bedded in a sulfate-rich backfill that helps keep them active. This is not practical, however, in immersed situations, where the anode must be boldly exposed. As a result, immersed zinc anodes sometimes undergo a surface reaction called “passivation” that decreases or eliminates their ability to protect a ferrous structure. Because it is important to know situations when this will happen, the Materials and Corrosion Laboratory (MCL) undertook a research project to develop a rapid test to determine when zinc anodes would not be effective in natural waters.

Anodic potentiodynamic polarization is a common electrochemical research technique used to study the active and passive behavior of a metal in a particular environment. This technique forces the electrochemical potential of the metal across a range of potentials, while measuring the current density associated with a given potential. Passivating metals demonstrate a characteristic potentiodynamic polarization curve; the current required to shift the potential anodically beyond some point does not increase and often decreases. The current associated with metal in the passive range is characteristically small, indicating a very low level of metal corrosion. In this study, potentiodynamic polarization was attempted and, in fact, showed passivation of zinc in some artificial laboratory solutions. However, the technique requires a sophisticated piece of equipment called a “potentiostat,” as well as meticulous procedures.

Passivation causes a metal to demonstrate a more noble or electropositive behavior than it would otherwise. Therefore, the voltage difference between zinc anode material and a standard reference electrode in test solutions was monitored over time to look for unanticipated noble potential shifts. Although some researchers indicated it could take considerable time to reach a final stable potential, this technique indicated that zinc anodes would trend towards passivation in some solutions in a matter of hours. Furthermore, this method requires only a standard reference cell and a typical high impedance voltmeter. The method also requires very little training or experience.

First, the MCL recommends that zinc Type II anodes not be used in immersed applications unless experience or testing shows they will remain active in that

particular water. MCL also recommends that when testing is required, monitoring the electrochemical potential of zinc anode material in the water of interest over the course of hours, or even overnight, be done before using this material. Given the possible variability in water chemistry, monitoring the potential in waters taken in different seasons would provide added confidence. Testing might even be performed by site personnel with direction from the Technical Service Center. Alternatively, potentiodynamic polarization scans are also useful for demonstrating passivation of zinc anode material, but they are perhaps more useful for detailed research.

Introduction

Although they develop only a small driving potential, zinc galvanic anodes have some advantages over magnesium anodes. First, they generally do not cause cathodic disbondment, even when directly mounted on an appropriately coated surface of a structure to be cathodically protected. Second, they are much more efficient (90 to 95 percent) compared to magnesium anodes (50-percent efficiency), suffering much less autogenous corrosion. They are, therefore, useful when small amounts of current are needed, especially in low resistivity soil where they are buried in a sulfate-rich backfill or in seawater where chloride and sulfate levels are relatively high.

Some metals, when immersed in particular solutions, react to form a surface film that will prevent further significant corrosion. This mechanism, called passivation, is the method whereby stainless steels and many other alloys resist attack. While passivation is a desirable feature of engineering alloys, it is undesirable for galvanic anode materials; if galvanic anodes passivate, they can no longer protect a structure against corrosion. Hence, to prevent passivation, buried zinc galvanic anodes are surrounded by sulfate rich backfill that keeps them active; however, surrounding zinc anodes with sulfates is not possible when they are used in immersion service because the backfill would wash away. The environment itself must keep the anode active. The literature, as indicated in the following paragraphs show that passivation of zinc is impacted by various chemical constituents of an environment. Temperature can accelerate the rate of passivation (Slunder and Boyd, 1986).

Water Chemistries

The zinc anode material recommended for burial and fresh water applications is high purity zinc specified as American Society for Testing and Materials (ASTM)¹ B-418, Type II. However, the NACE International² CP-3 Manual (Cathodic Protection Technologist Course Manual) warns that waters high in such components as carbonates, bicarbonates, or nitrates can cause a noble shift in zinc potential, even at room temperature, and that higher temperatures accelerate this passivation effect (NACE, 2004). That warning is based on earlier research, some of which is mentioned below. The literature also indicates that dissolved chemicals, such as sulfates and chlorides, can aid in preventing zinc passivation. However, natural waters typically contain a mixture of competing species.

In his 1979 Materials Performance magazine article, George Kurr refers to unpublished studies by Asarco Research indicating that when the amount of

¹ The American Society for Testing and Materials is now known as ASTM International.

² NACE International was formerly known as the National Association of Corrosion Engineers.

sulfates or chlorides exceeds the amount of bicarbonates, carbonates, nitrates, and phosphates, the potential of zinc will not be “seriously” affected, regardless of temperature (Kurr, 1979). He follows up that statement by saying that bare zinc anodes should not be used until tests in solutions similar to the intended environment have shown compatibility. In addition, later articles cast doubt on the feasibility of relying on a simple excess of chlorides and sulfates. For instance, a recent paper presented at the NACE Corrosion 2010 Conference described experiments in Lake Maracaibo water in which various anode materials were tested; zinc anode materials were susceptible to passivation even though the water contained almost 1,500 parts per million chloride ions, far in excess of carbonates and bicarbonates (de Rincon et al., 2010). Other studies are also worth considering.

In a 1982 article published in *Materials Performance* magazine, Elwood Haney, of Asarco Inc., adds borates, dichromates, tungstates, permanganate, and ferricyanates to the list of passivating anions under certain conditions (Haney, 1982). However, as he indicates, these, along with phosphates, are less common in natural waters. He also mentions that oxygen helps to ennoble the zinc, though it appears that oxygen concentration would need to be very low to keep zinc at its usual protective potential.

Haney also mentions that at least one study showed that calcium and silicates can help keep zinc anodes active (Haney, 1982). He also found that sulfates were more effective than chlorides in keeping zinc active, although both were helpful in that respect. He indicates that increasing the concentration of inhibiting anions does not necessarily have an additive impact. Furthermore, Haney was able to show that the zinc electrochemical potential was adversely affected in a solution of 600 milligrams per liter (mg/L) of HCO_3^- , even at room temperature; thus, elevated temperature is not necessary for ennoblement of the zinc, although elevated temperatures accelerate the effect.

In their *Handbook of Cathodic Corrosion Protection*, von Beckmann et al. state that “the use of zinc anodes in fresh water is very limited because of their tendency to passivate” (von Baeckmann, 1997). These authors specifically warn that in waters low in salts and with low loading of the anode, less soluble basic salts, such as basic zinc chloride, are formed. They further indicate that waters containing phosphates can form the very insoluble ZnNH_4PO_4 . However, these compounds will only precipitate in a narrow pH range around pH 7. Furthermore, weakly acidic, flowing, salt rich media can help solubilize surface films. They also state that at a temperature of 60 degrees Celsius ($^{\circ}\text{C}$), the structure of a surface film can change from $\text{Zn}(\text{OH})_2$ to ZnO , causing a shift to a more positive potential in common oxygen bearing fresh water. The result can be a potential reversal in which the rest potential of zinc becomes more positive than the potential of iron. Under such conditions, zinc would not protect, but would rather accelerate, corrosion of iron or steel.

The various chemistries that can induce passivation are, therefore, complicated and can be interrelated with pH, temperature, etc. The literature does not give specific guidance as to the composition of waters that determines when zinc anode material will passivate and when it will stay active. It only provides some general guidelines. Nonetheless, we can summarize chemical species that will tend to passivate versus those that will tend to maintain activity in table 1.

Table 1. —Chemical species that passivate Zn versus those that keep Zn active

Passivating Species	Activating Species
Carbonates	Sulfates
Bicarbonates	Chlorides
Nitrates	Calcium
Phosphates	Silicates
Borates	—
Dichromates	—
Tungstates	—
Permanganates	—
Ferricyanates	—
Oxygen	—

Zinc Anode Electrochemical Potential Response

Frank Ansuini, of EDI, Inc., supplied an unpublished summary of test results for zinc material his company had once contemplated using as zinc reference cells in waters (Ansuini, personal communication, 2016). Zinc, either pure or as an anode alloy, should have an uncoupled potential of approximately -1,100 millivolts (mV) with respect to a copper/copper sulfate reference cell (CSE), or about -1,025 mV with respect to a saturated calomel reference cell (SCE) which, as in this report, is often used for laboratory experiments. Zinc is a solid; therefore, it should serve as a rugged reference electrode if the potential is stable in natural waters. Zinc surrounded by a sulfate-rich backfill is, in fact, used as a permanent reference cell in soil applications. The EDI test waters consisted of well water from Parkman, Ohio; two surface waters (one from Belmont, Massachusetts [Metropolitan District Commission], and one from Lincoln, Rhode Island [Providence Water Supply Board]); and an artificial ASTM seawater mixture. Ansuini's results showed that the potential of bare 99.99-percent zinc in the two surface waters shifted almost 300 mV to a stable potential of -800 mV (CSE) in about 4 to 5 months. The same zinc material in the test well water stabilized at a potential of -975 mV_(CSE), a shift of about 125 mV from the anticipated -1,100 mV_(CSE) in about 2 weeks. The pure zinc anode material in seawater

stabilized at $-1,100 \text{ mV}_{(\text{CSE})}$ in about 2 weeks. More detailed water chemistry information was not available.

While it took weeks or months to reach a stable potential in Ansuini's tests, determining whether zinc will trend towards passivation or remain stable may be simpler because it is not critical to know the final potential. One must only know that zinc will not have the expected active potential. While the literature indicates that electrochemical methods have been used to study the general passivation behavior of zinc anodes, no indication was found that a specific screening test had been developed.

Potentiodynamic polarization is an electrochemical research method that passes current between a metal sample of interest and an inert electrode in a given solution to alter the electrochemical potential (V) of the test sample. The current density passed for a given potential is then recorded. This procedure progresses over the range of potentials of interest. Because the current density correlates with the ongoing oxidation or corrosion of the metal, passivation behavior of the metal, or lack thereof, can be displayed graphically. The display of a passivating situation typically shows up as a drastic drop in oxidation current once the potential is sufficiently noble. Figure 1 shows a typical passivation curve.

Active metals, such as zinc and aluminum, in a passivating solution will sometimes not display the "nose" shown in figure 1. Rather, a change in potential may demonstrate a low current density or corrosion rate until a transpassive region is reached.

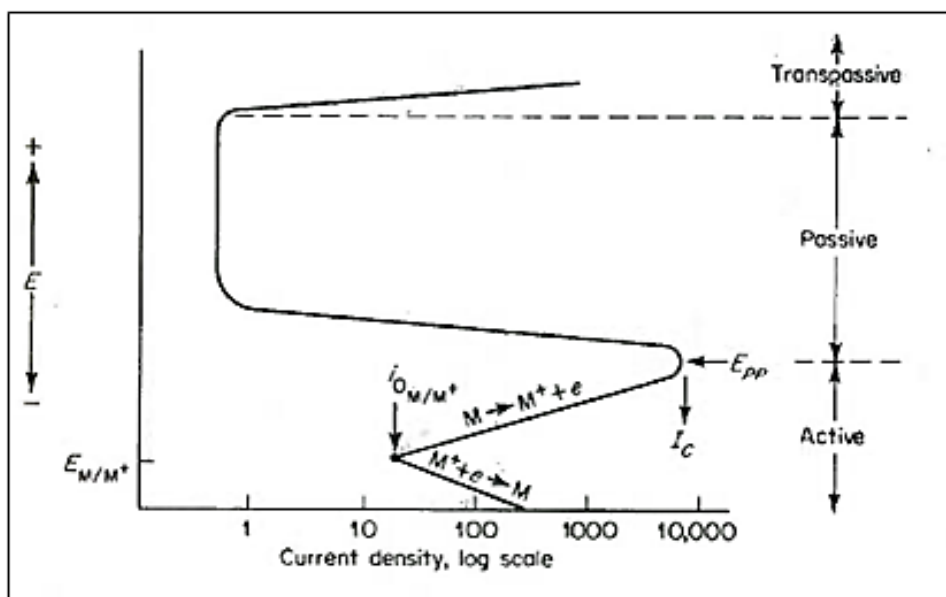


Figure 1.—Typical Evans diagram for a passivating metal (Source: Fontana, 1986).

Experimental Setup and Procedures

Potentiodynamic scans were performed using a Princeton Applied Research Parstat 2273 potentiostat (see figure 2).



Figure 2.—Parstat 2273 potentiostat used in these experiments.

A standard glass test cell was used, and the cell was placed in a Faraday cage (see figure 3) to minimize electronic noise. The test sample was centered in the cell with two graphite working electrodes on either side and electrically jumpered together to pass current between the test sample and themselves. A saturated calomel reference electrode in a salt bridge filled with the solution of interest faced the metallic sample on one side in order for the potentiostat to monitor the potential being held. High purity nitrogen was bubbled through the solution using a sintered glass sparger on the side of the cell opposite the salt bridge. The nitrogen was released from the cell by bubbling through a beaker partly filled with water to seal out air.

Solid zinc rods 5/8 inch in diameter were obtained through Farwest Corrosion Control Company. The rods were of a high purity or Type II zinc anode composition (see table 2). The rods were machined to size to fit the holders described below.



Figure 3.—Test cell and test cell in Faraday cage.

Table 2.—Composition of zinc material

Element	% Supplied	Maximum ASTM Limits
Cd	0.0001	0.003
Fe	0.0002	0.0014
Cu	0.0012	0.002
Pb	0.0018	0.003
Sn	0.0001	—
Al	<0.0001	0.005
Zn	99.9966	Remainder

The machined zinc samples were either cylindrical or flat circular type and were held in sample holders (see figure 4). The area of exposed surface on cylindrical samples was measured with Vernier calipers before exposure, and the information was fed to the program, directing the potentiostat in order to graph the amperage per area on the X-axis. Cylindrical samples consistently exposed 1 square centimeter due to the size of the opening on the flat sample holder.

Test surfaces of zinc samples were ground through 600 grit paper to ensure a consistent surface and current density comparisons. Following surface grinding, samples were thoroughly rinsed using methanol and allowed to dry before they were mounted in a sample holder and immersing in a test solution.

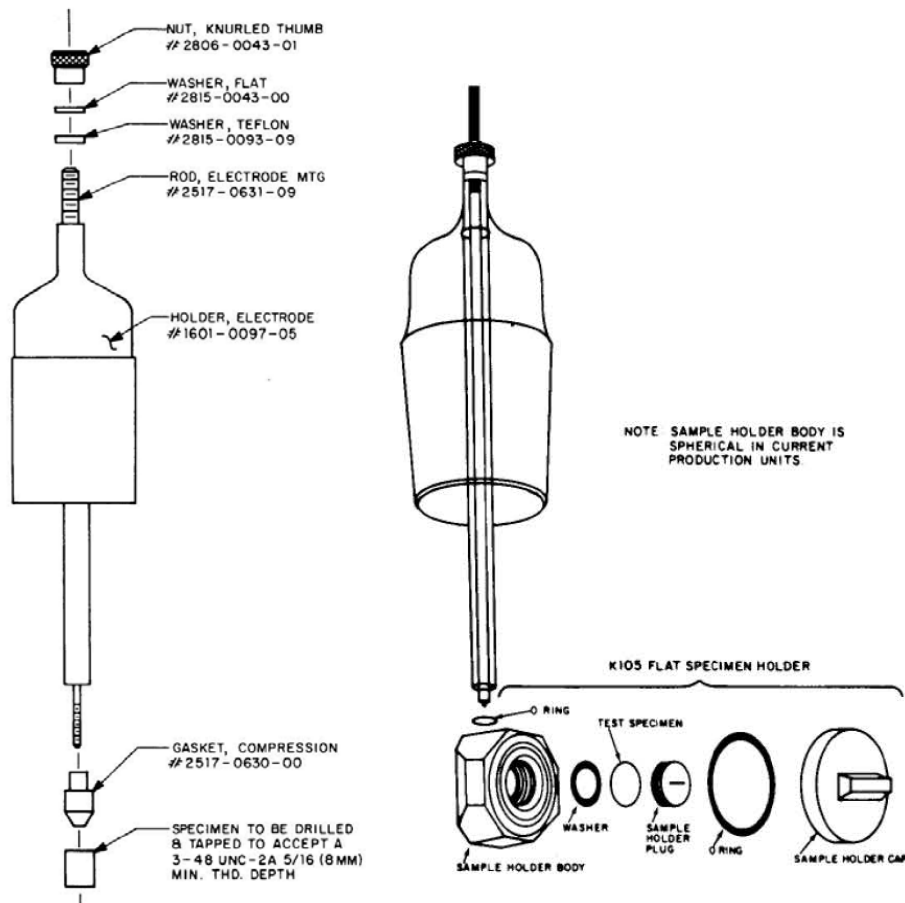


Figure 4.—Two styles of sample holders used.

Nitrogen was sparged through the test solution in the cell for about 1 hour to remove as much dissolved air as possible. With dissolved oxygen present, metal oxidation readings may be hidden by oxygen reduction at the more strongly negative potentials. Following de-aeration, the prepared sample was immersed in the test solution, and the solution was sparged with nitrogen for an additional hour before a scan was begun in the anodic direction (i.e., from more negative to more positive). The scan rate was 0.166 millivolts per second, and work was performed at ambient room temperatures (approximately 21 to 25 °C).

To verify the integrity of the equipment, experimental setup, and procedures, scans of 430 stainless steel were first performed in accordance with ASTM G5. Appendix A, figure A-1 shows several final repeated calibration scans.

Test solutions were made up using deionized water and reagent grade chemicals in standard volumetric flasks. Scans of zinc anode material were performed in solutions containing ions known to keep the zinc active, as well as in solutions of ions known to passivate zinc. Solutions of mixtures of these conflicting ions were also used in these experiments. Scans were performed at least twice to help ensure repeatability of the data; crevices between the zinc sample and holder, and other minor abnormalities, can significantly alter test results. Table 3 lists the solutions used in the study.

Table 3.—Solutions used and study results

Solution Composition	Zinc Behavior
200 mg/L SO_4^{-2} (K_2SO_4)	Active
200 mg/L Cl^- (NaCl)	Active
600 mg/L HCO_3^- , 73 mg/L NO_3^- (NaHCO_3 and NaNO_3)	Passive
600 mg/L HCO_3^- , 73 mg/L NO_3^- , 800 mg/L SO_4^{-2} (NaHCO_3 , NaNO_3 , K_2SO_4)	Passive
600 mg/L HCO_3^- , 73 mg/L NO_3^- , 800 mg/L Cl^- (NaHCO_3 , NaNO_3 , NaCl)	Passive
60 mg/L HCO_3^- , 7.3 mg/L NO_3^- , 80 mg/L Cl^- (NaHCO_3 , NaNO_3 , NaCl)	Passive

In a separate series of experiments, samples of zinc anode material were immersed in open beakers of the same solutions as were used in the potentiodynamic polarization scans along with a SCE reference cell. The sample and reference were connected to the potentiostat, which was then programmed to operate in an open circuit potential reading mode, reading the potential of the zinc sample at defined time intervals. These tests were often allowed to run overnight (see appendix A).

Results and Discussion

Appendix A shows results of potentiodynamic scans and of potential readings over time for the same solution. Table 3 summarizes the general behavior of the zinc anode material (active or passive) in the various solutions. Note that only a typical scan of a set is shown for each solution.

Figures A-2 and A-4 in appendix A show the characteristic polarization scans for a nonpassivating situation. Increasingly positive potentials result in an increasing corrosion rate (increasing anodic current density) until limited by polarization affects (i.e., reaction kinetics limit increases in anodic current as potential becomes more positive), at which point the curve tends to go near vertical.

Figures A-6, A-8, A-10, and A-12 in appendix A show a characteristic passivation behavior in which current density remains quite low as potentials are forced to more positive values, until a transpassive region is reached. After reaching the transpassive region, changes in potential in the positive direction cause the zinc to increasingly corrode (current densities increase). Test solutions with comparatively high concentrations of passivating ions (see figures A-6, A-8, and A-10) showed a very distinct passive region and a transpassive transition point at a significantly positive potential, even when a significant concentration of activating ions was added. When, as seen in figure A-12, concentrations of both passivating and activating ions were kept in the same ratio, but reduced to a tenth of that shown in figure A-10, the transpassive point was observed at a less positive potential.

Figures A-3 and A-5 show the electrochemical potential over time for zinc anode material in the activating solutions used in figures A-2 and A-4. As suggested by the private communication from Ansuini, these may not be the final stable potentials. Nevertheless, they clearly indicate no tendency for the zinc anode material to become passive. On the other hand, figures A-7, A-9, A-11, and A-13, using the same solutions as for scans shown in figures A-6, A-8, A-10, and A-12, clearly indicate a tendency for the zinc anode material to seek a more positive potential after even a few hours.

As indicated in the literature, solutions high in sulfates or chlorides maintained zinc anode material in the active state. Solutions high in bicarbonates and nitrates produced passivation of the zinc Type II anodes. A simple excess of sulfates or of chlorides above the concentration of passivating anions was not sufficient in these tests to keep the zinc anodes active.

Conclusions

Potentiodynamic polarization is a valuable tool for corrosion research. Trained personnel can garner significant corrosion information by using this technique. This present research has demonstrated that potentiodynamic polarization can show when zinc Type II anode material will exhibit passivation in natural waters. However, it requires expensive equipment and somewhat complex, detailed procedures, as well as an understanding of electrochemistry.

On the other hand, simply immersing a clean sample of anode material in the natural water of interest and monitoring the changes in potential with respect to a standard reference cell (e.g., copper/copper sulfate, saturated calomel electrode, etc.) over time appear to be capable of indicating in a matter of hours whether anode passivation may take place. Although readings in this study were taken

automatically using features of the potentiostat, the same determination could be made more simply. A potential measurement could be manually taken when a sample of anode is first immersed in the water of interest and then in several hours (or overnight). This should indicate whether the electrochemical potential is shifting to an unexpectedly more positive potential, indicating the zinc is passivating, or if the potential remains in the range normally expected of the anode. This is not only simpler, but could also give some indication of the magnitude of potential shift.

Recommendations

In view of the study findings the MCL recommends that:

- Zinc Type II anodes be boldly exposed in a site-specific water sample if it has been confirmed, either by testing or by prior experience, that they will not passivate in that particular water.
- Testing of zinc Type II anodes cover a range of seasonal water variations to account for chemical changes.
- Testing take place in coordination with a corrosion professional.
- Testing consist of monitoring the corrosion potential of uncoupled zinc anode material boldly exposed to the natural water in question; this may be done in the lab or in the field.
- Potentiodynamic polarization be considered an alternative to monitoring potentials and for more detailed studies.

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Appendix A

Potentiodynamic Scans on Zinc Anode Material and Potential Versus Time

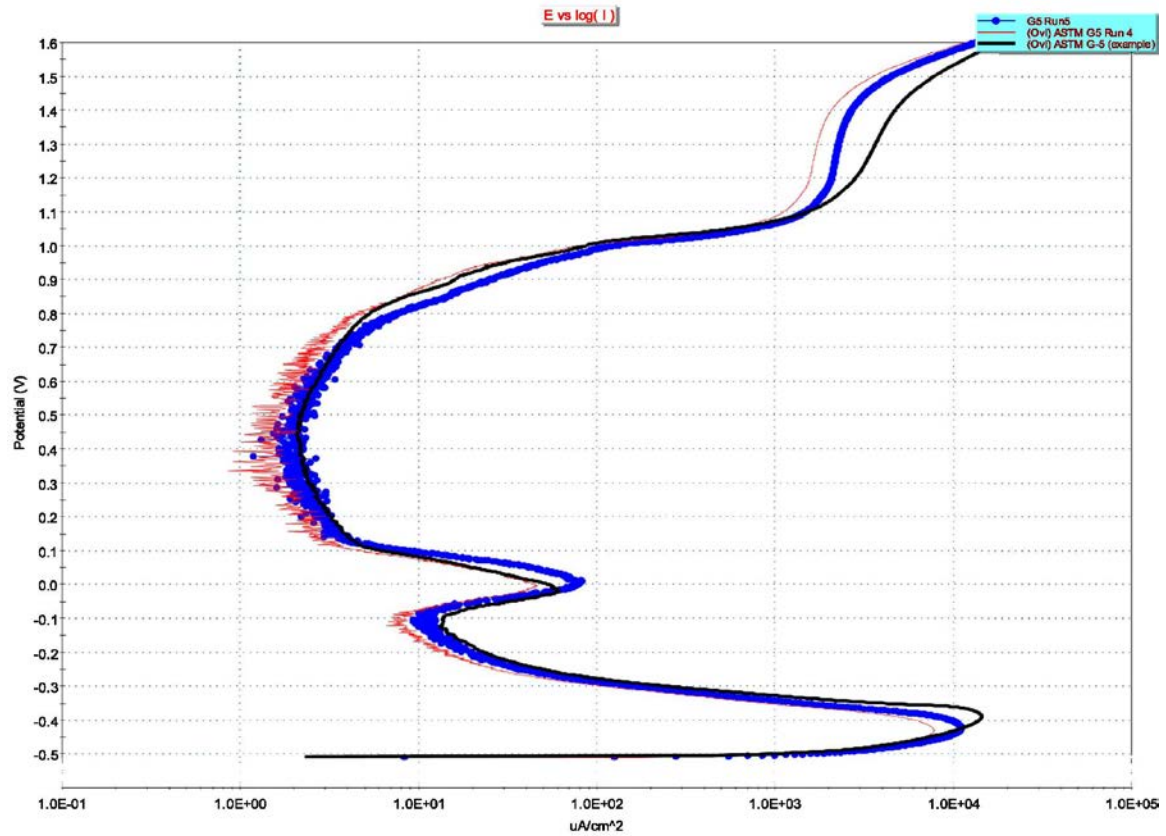


Figure A-1.—Calibration scans on 430 stainless steel in sulfuric acid solution per ASTM G5.

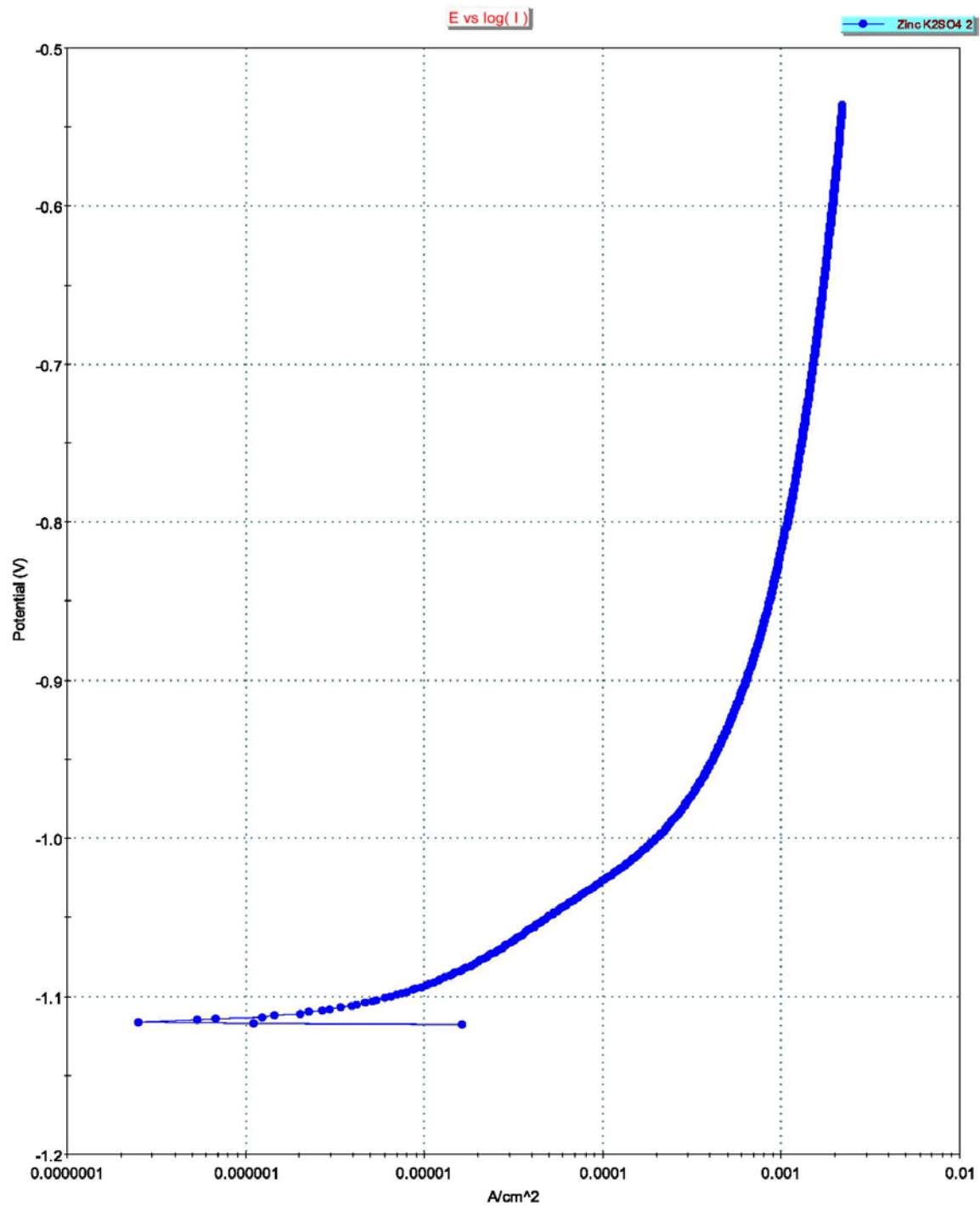


Figure A-2.—Scan of active zinc anode (200 mg/L SO_4^{2-} solution (K_2SO_4)).

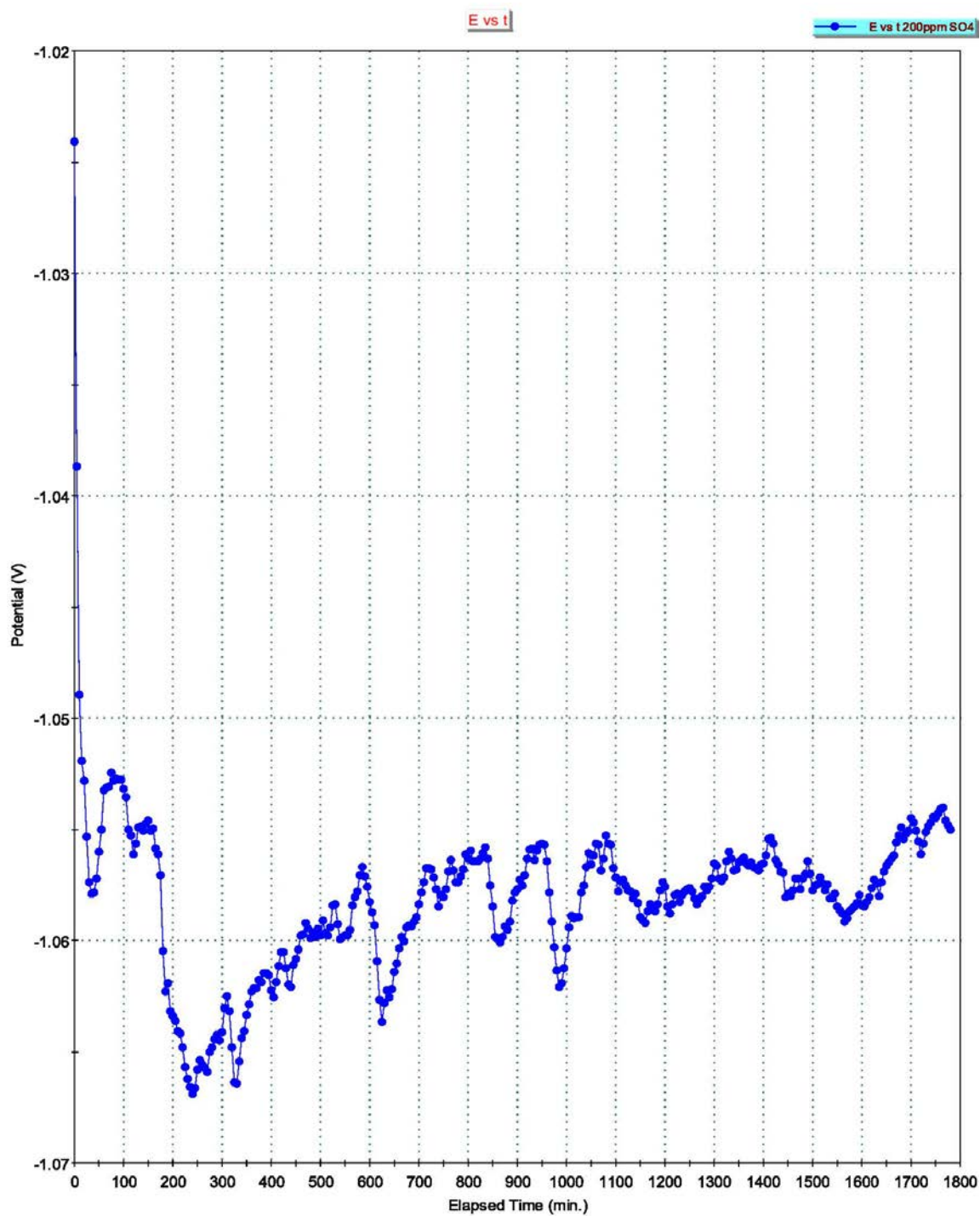


Figure A-3.—Potential versus time of active zinc anode (200 mg/L SO_4^{-2} solution (K_2SO_4)).

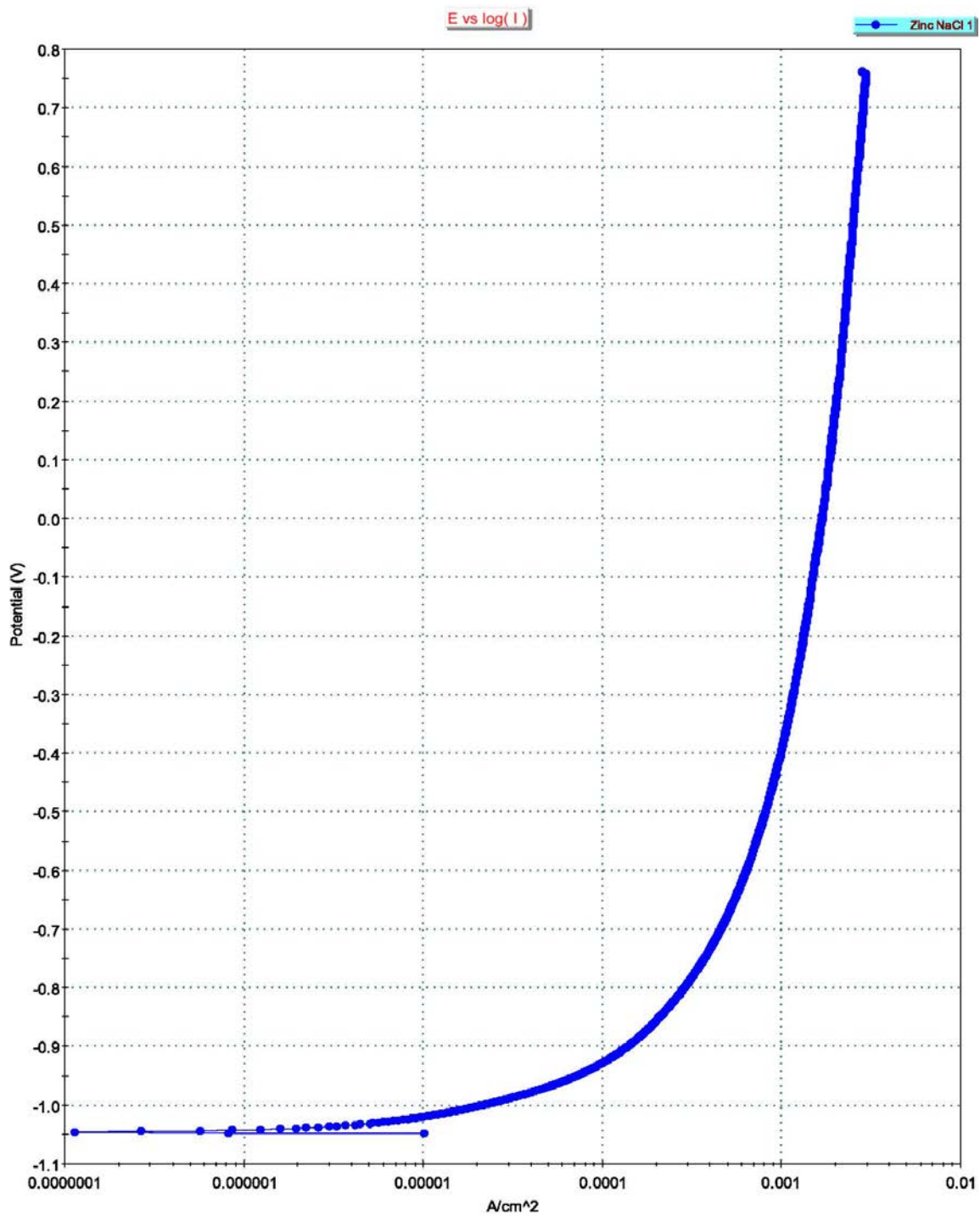


Figure A-4.—Scan of active zinc anode (200 mg/L Cl⁻ solution (NaCl)).

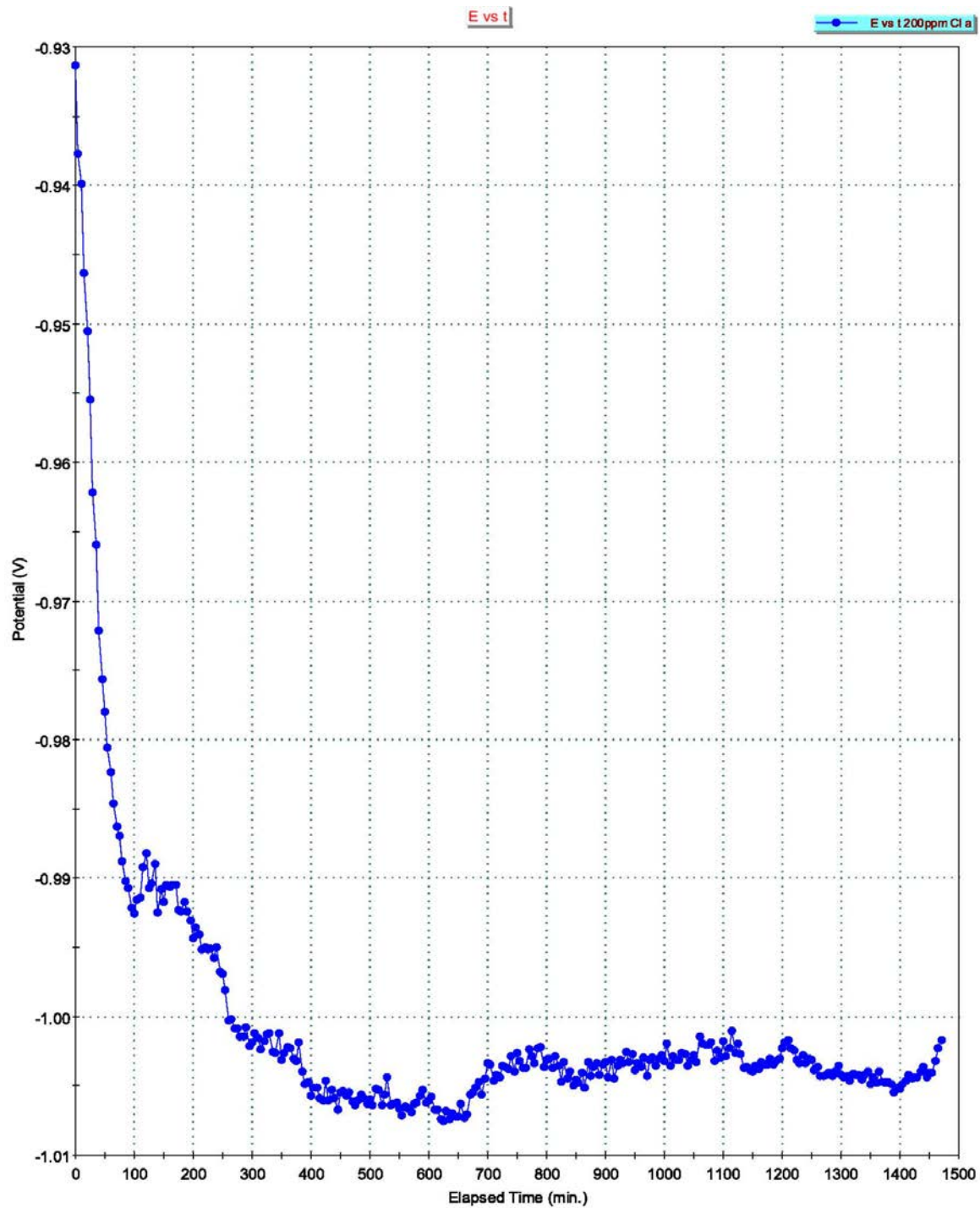


Figure A-5.—Potential versus time of active zinc anode (200 mg/L Cl⁻ solution (NaCl)).

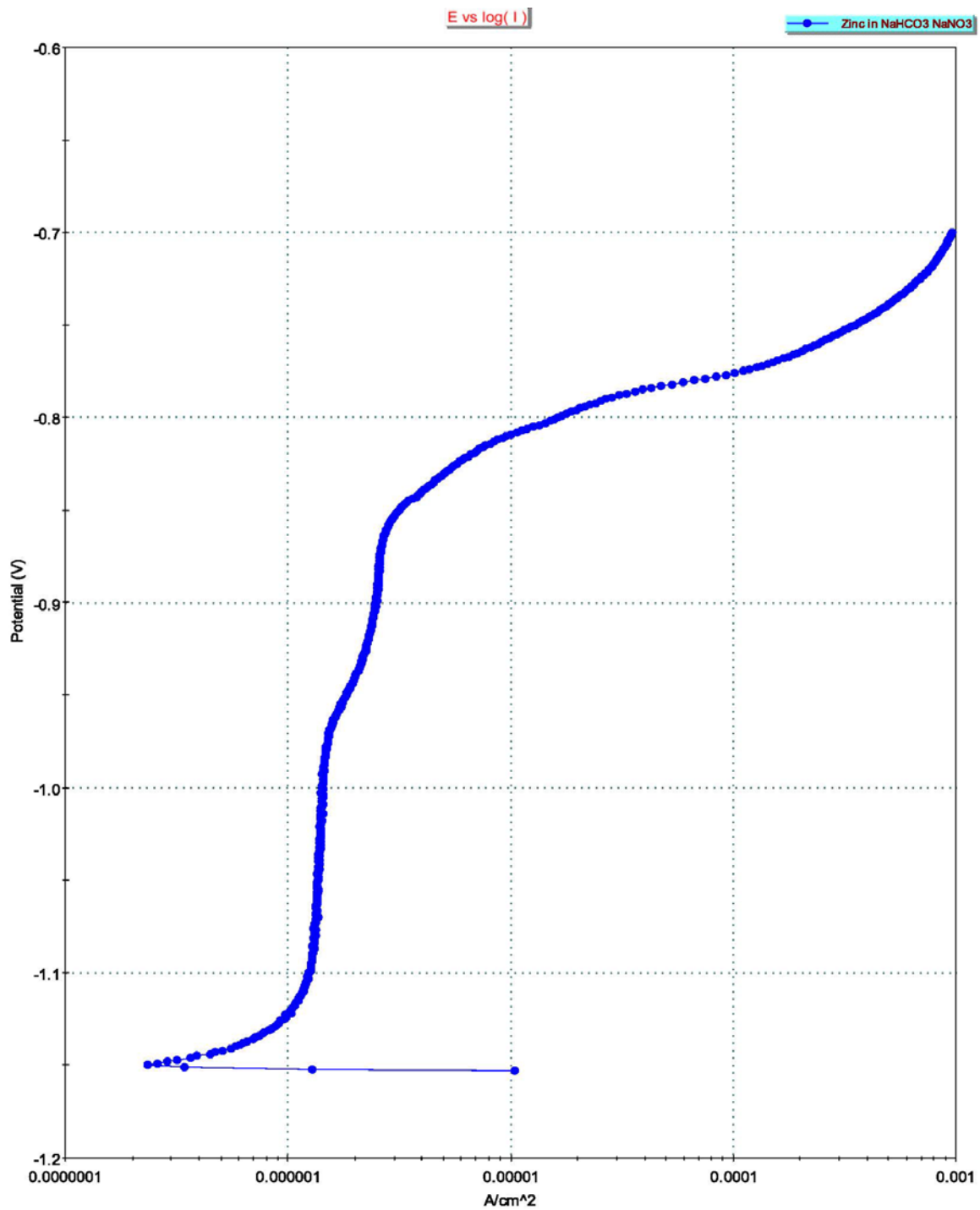


Figure A-6.—Scan of passivated zinc anode (600 mg/L HCO_3^- and 83 mg/L NO_3^- solution (NaHCO_3 and NaNO_3)).

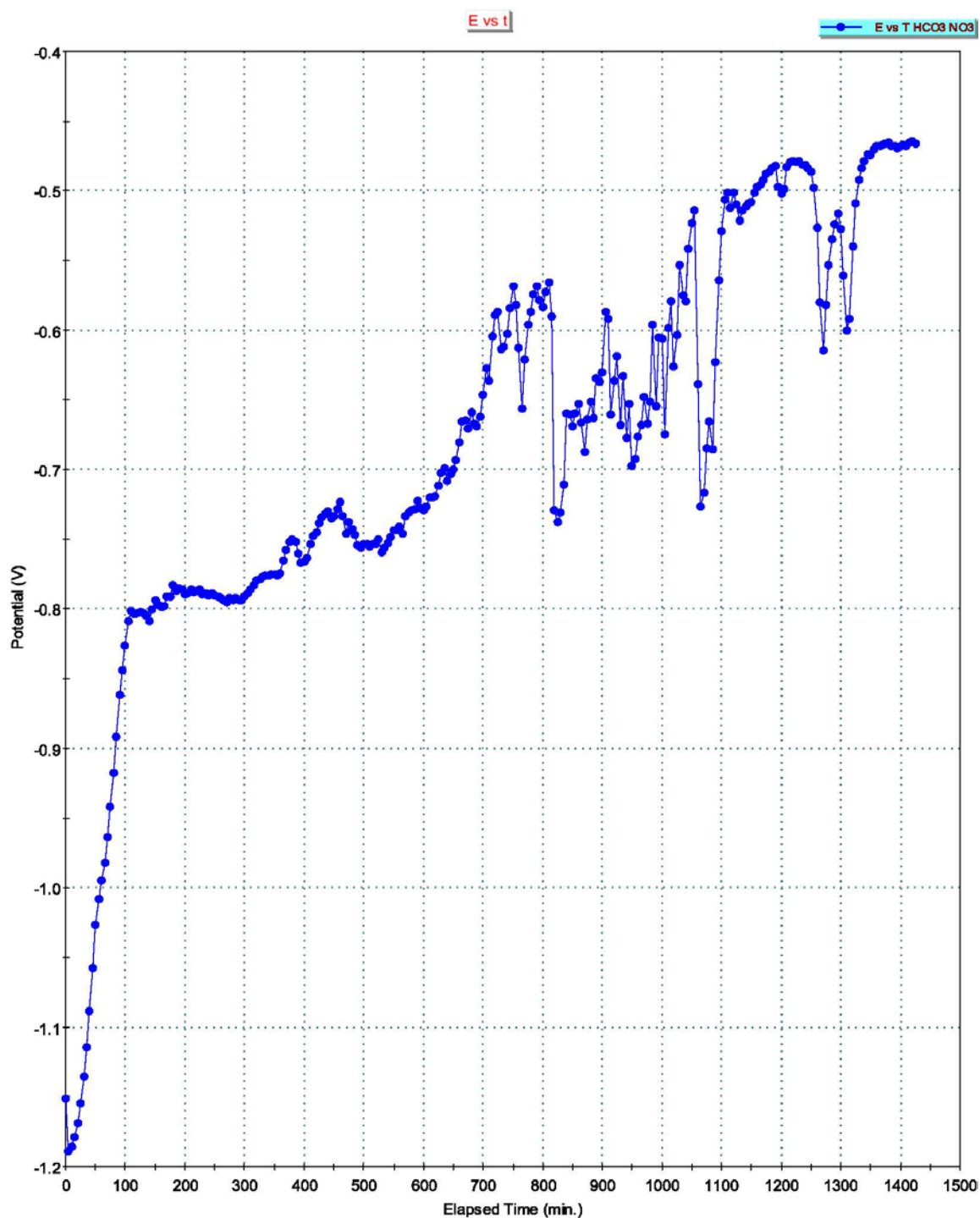


Figure A-7.—Potential versus time of passivated zinc anode (600 mg/L HCO_3^- and 73 mg/L NO_3^- solution (NaHCO_3 and NaNO_3)).

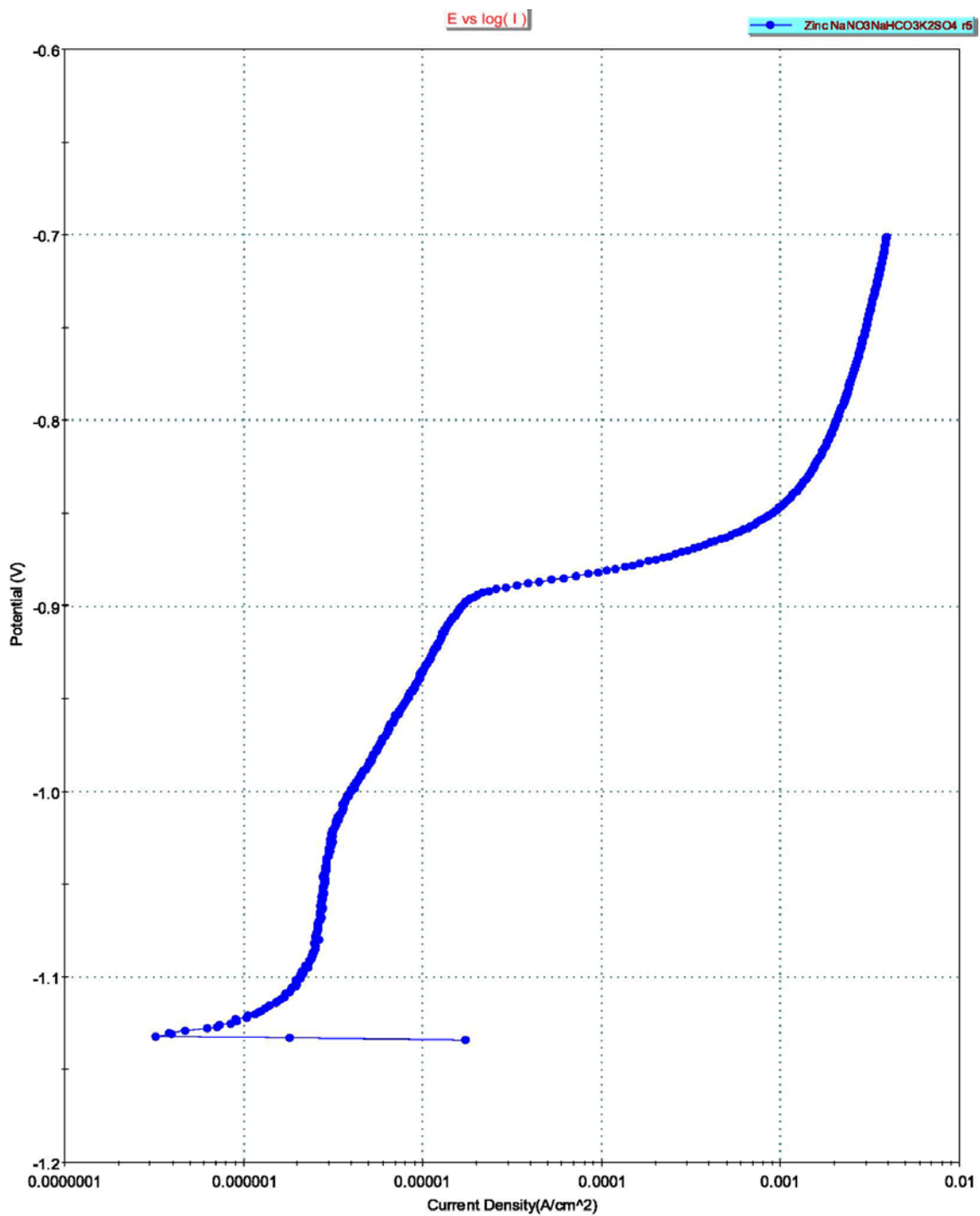


Figure A-8.—Scan of passivated zinc anode (600 mg/L HCO_3^- , 73 mg/L NO_3^- , and 800 mg/L SO_4^{2-} solution (NaHCO_3 , NaNO_3 , K_2SO_4)).

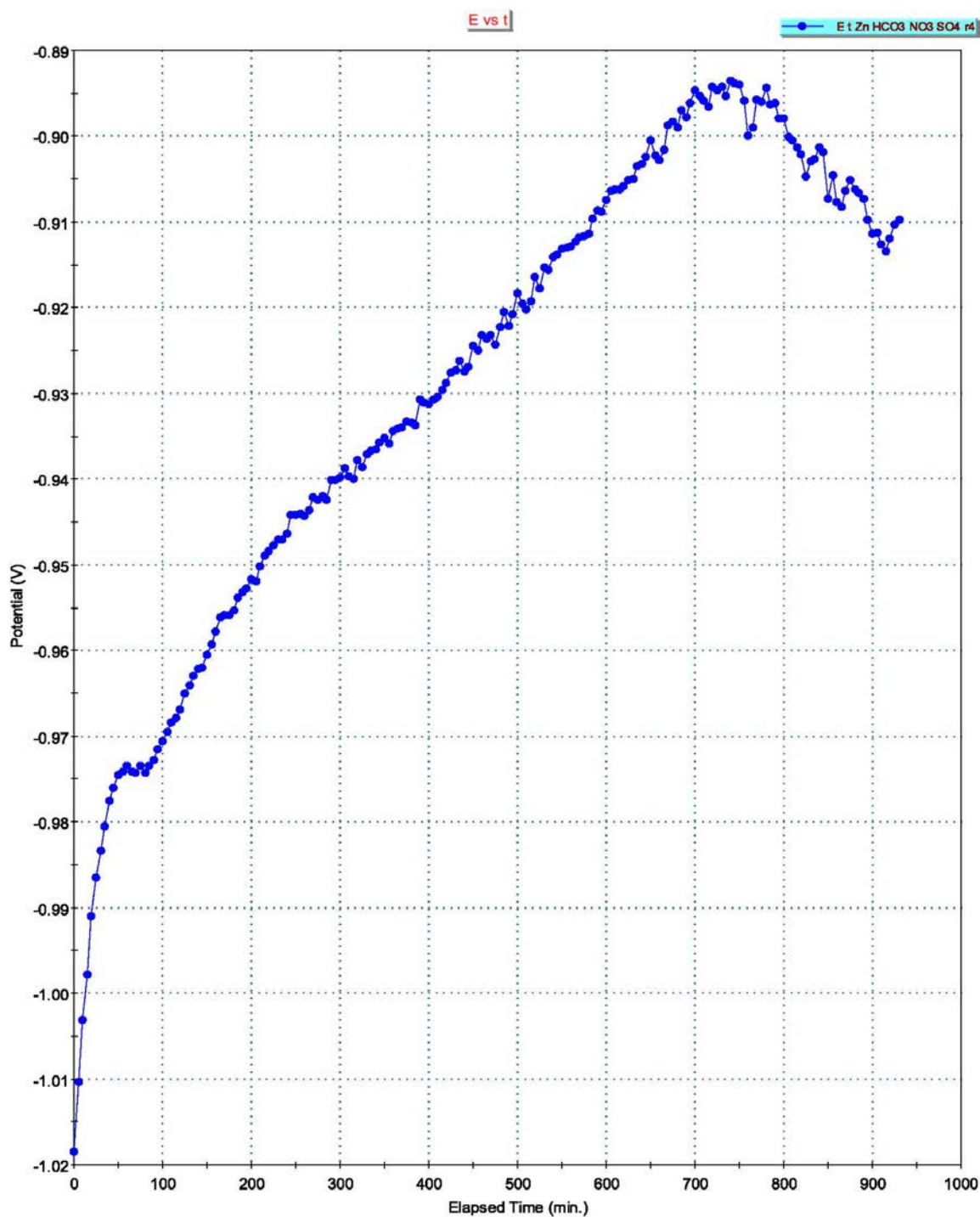


Figure A-9.—Potential versus time of passivated zinc anode (600 mg/L HCO_3^- , 73 mg/L NO_3^- , and 800 mg/L SO_4^{2-} solution (NaHCO_3 , NaNO_3 , K_2SO_4)).

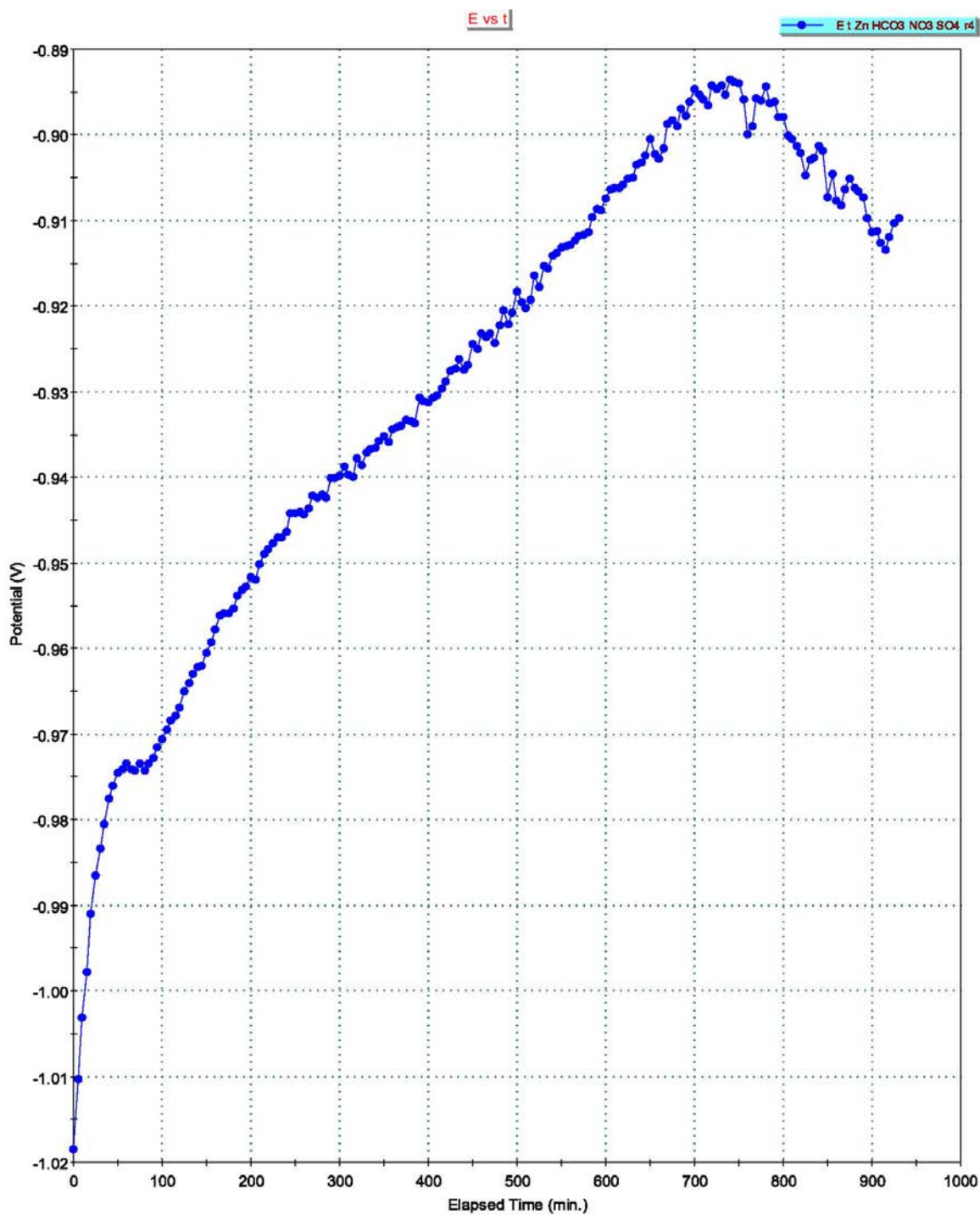


Figure A-10.—Scan of passivated zinc anode (600 mg/L HCO_3^- , 73 mg/L NO_3^- , and 800 mg/L Cl^- solution (NaHCO_3 , NaNO_3 , NaCl)).

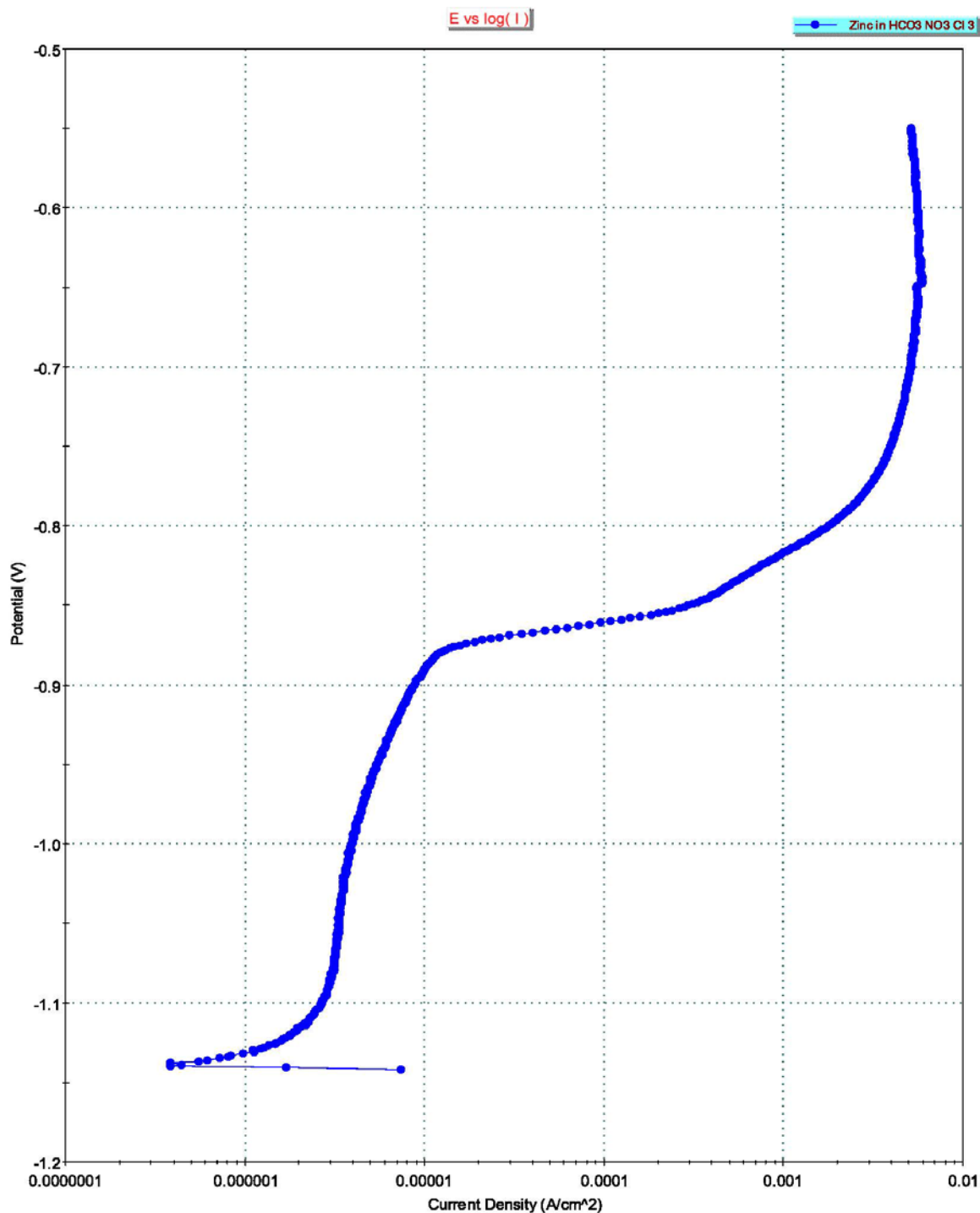


Figure A-11.—Potential versus time of passivated zinc anode (600 mg/L HCO_3^- , 73 mg/L NO_3^- , and 800 mg/L Cl^- solution (NaHCO_3 , NaNO_3 , NaCl)).

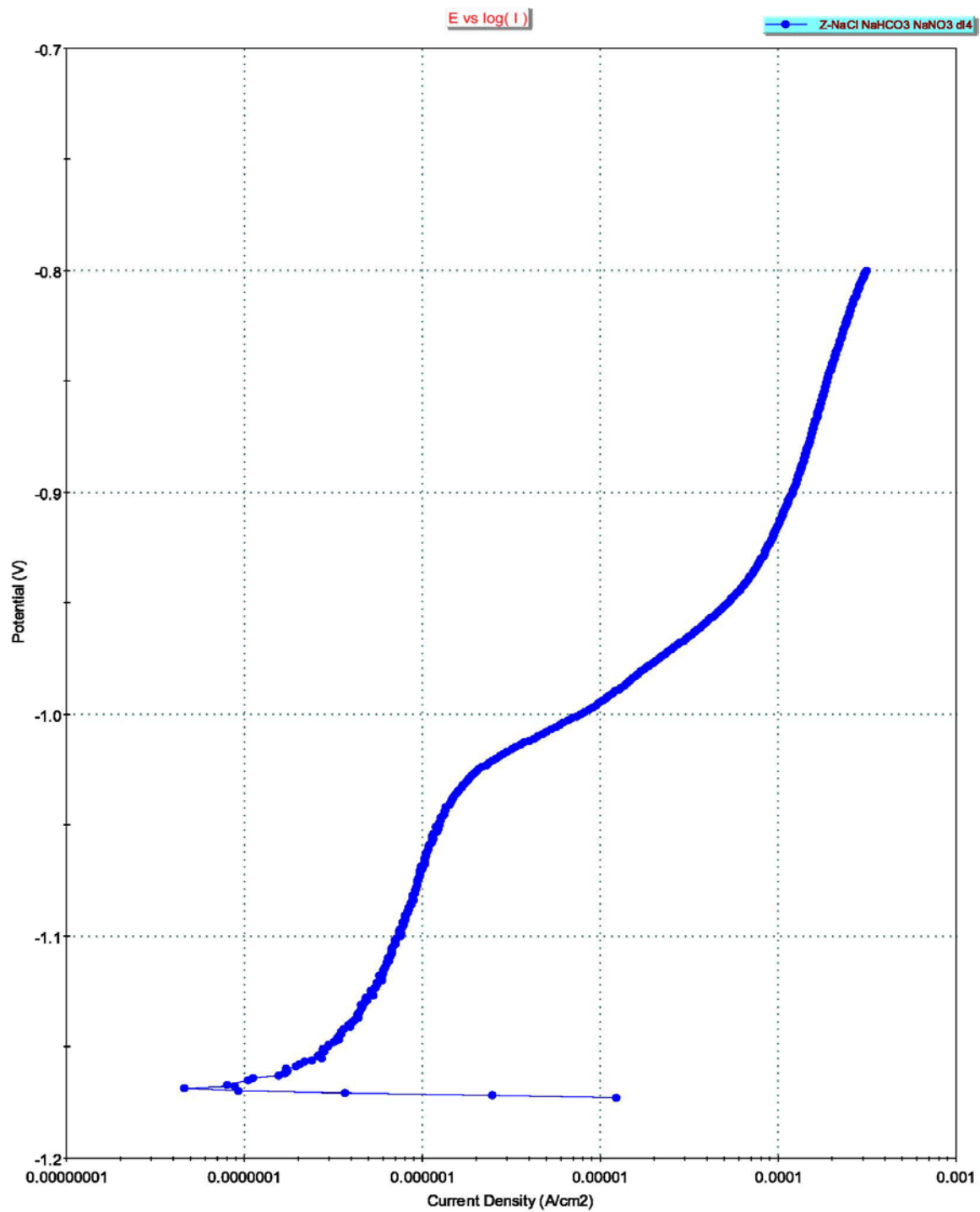


Figure A-12.—Scan of passivated zinc anode (60 mg/L HCO_3^- , 7.3 mg/L NO_3^- , and 80 mg/L Cl^- solution (NaHCO_3 , NaNO_3 , NaCl)).

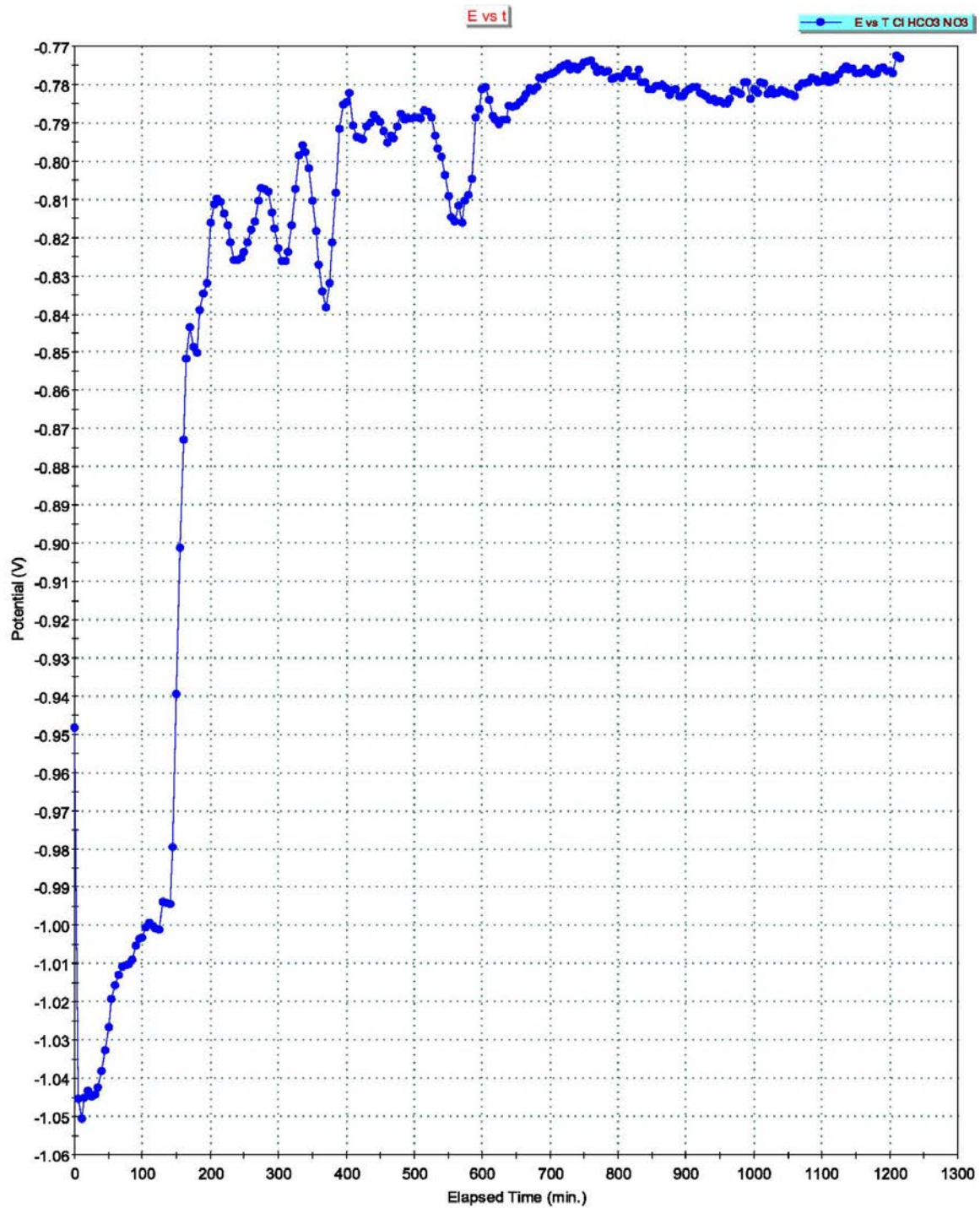


Figure A-13.—Potential versus time for passivated zinc anode (60 mg/L HCO_3^- , 7.3 mg/L NO_3^- , and 80 mg/L Cl^- solution (NaHCO_3 , NaNO_3 , NaCl)).

