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DEVELOPMENT OF THE IMPROVED CORROSION
RATE METER AND EVALUATION OF COMMERCIALY
DEVELOPED HIGH-TEMPERATURE pH ELECTRODES

Interim Report for
Period Ending September 1981

M. J. Danielson

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Pacific Northwest Laboratory
Richland, WA 99352

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SUMMARY AND CONCLUSIONS

This report discusses the continued development of the improved corrosion rate meter (ICR) at Pacific Northwest Laboratory (PNL) and the initial laboratory evaluation of commercially developed (under subcontract to PNL) pH electrodes.

The ICR is being developed because it is important to have accurate, on-line corrosion information to warn of any chemistry perturbations that could result in catastrophic corrosion rates in a geothermal environment. Commercial equipment currently in use can both underestimate and overestimate corrosion rates. The objective of this work is to develop a more flexible instrument that would respond with correct corrosion rates under a wider range of conditions.

The ICR was successfully tested in the field and in the laboratory. Although commercial corrosion rate meters (CCRs) are adequate for many situations, the ICR is the most versatile instrument and gives more accurate corrosion rate measurements.

Glass and ceramic pH electrodes were tested at 95°C and 200°C. None of the electrodes performed well at 95°C, and the glass pH electrodes either failed or had no pH response at 200°C. The ceramic electrode performed well at 200°C and is worthy of further development.

CONTENTS

SUMMARY AND CONCLUSIONS	iii
INTRODUCTION	1
IMPROVED CORROSION RATE METER	2
BACKGROUND	2
FIELD TEST	4
LABORATORY STUDIES	6
pH ELECTRODE STUDIES	10
ELECTRODE TESTS AT 95°C	10
Glass Electrode Results (Manufacturer A)	11
Glass Electrode Results (Manufacturer B)	14
Ceramic Electrode Results (Manufacturer C)	16
ELECTRODE TESTS AT HIGHER TEMPERATURE (200°C)	16
REFERENCES	22

FIGURES

1	Corrosion Rate Measurements During the Field Test at the East Mesa Test Facility	5
2	Commercial Electrode Mounting Fixture Used in Laboratory Study	7
3	Corrosion Rate Data Using the Iron Pseudo-Reference Electrode	8
4	Corrosion Rate Data Using the PNL External Reference Electrode	9
5	Mercury-Filled Glass pH Electrode (Manufacturer A)	12
6	pH Response of Three Mercury-Filled Electrodes (Manufacturer A) at 25°C	13
7	pH Response of Three Mercury-Filled Electrodes (Manufacturer A) at 95°C	14
8	Glass pH Electrode (Manufacturer B)	15
9	pH Response of Glass (Manufacturers A and B) and Ceramic (Manufacturer C) Electrodes at 95°C	17
10	Ceramic pH Electrode (Manufacturer C)	18
11	pH Response of Glass and Ceramic pH Electrodes at 200°C	19
12	Before and After Views of Aqueous-Filled Glass pH Electrode (Manufacturer A)	20
13	Before and After Views of Glass pH Electrode (Manufacturer B) That Broke During Testing	21

INTRODUCTION

Any system used to control scaling or corrosion in a geothermal power plant must include provisions for monitoring and verifying the fluid chemistry. Current methods (for example, grab-sampling for later analysis) are not entirely satisfactory; a better method would involve inserting in-line or down-hole chemical sensor probes. Using such sensors, continuous on-line data could be produced without the problems associated with sampling, such as infrequency, contamination, or chemistry changes caused by cooling. In-line probes for high-temperature geothermal environments are beyond the current commercial state of the art; however, technological advances have occurred in recent years largely because of the need for such probes in high-pressure nuclear power plants.

This report discusses the continued development of the improved corrosion rate meter (ICR) at Pacific Northwest Laboratory (PNL)^(a) and the initial laboratory evaluation of commercially developed (under subcontract to PNL) pH electrodes. The theoretical development of the ICR and a description of the electronics were discussed in an earlier report (Danielson and Koski 1981).

(a) Operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute.

IMPROVED CORROSION RATE METER

BACKGROUND

A brief review of how commercial equipment measures corrosion rates and a possible solution to its limitations are outlined in this section. The basic kinetic equation (Vetter 1967) that describes many electrochemical processes is:

$$I = I_{\text{corr}} \left[\exp \left(\frac{2.3}{b_a} E \right) - \exp \left(- \frac{2.3}{b_c} E \right) \right] \quad (1)$$

where I = measured current

I_{corr} = corrosion current (also called exchange current)

b_a, b_c = anodic and cathodic Tafel parameters

$E = E_{\text{applied}} - E_{\text{corr}}$ = potential perturbation from the open-circuit corrosion potential.

By taking a partial derivative of Equation (1) with respect to E and then setting it equal to zero at $E = 0$, the basic equation under which commercial equipment operates is derived as:

$$I_{\text{corr}} = \frac{\Delta I}{\Delta E} \cdot B \quad (2)$$

where $B = \frac{b_a \cdot b_c}{2.3(b_a + b_c)}.$

B is a constant that is dependent upon the Tafel slope parameters and generally falls between 10 to 45 mV for iron (Callow, Richardson, and Dawson 1976). Some commercial equipment has a hard-wired B value of about 30 mV, but a problem arises at elevated temperatures when oxygen enters the system and the cathodic reaction (oxygen consumption) becomes mass transport-controlled (independent of potential so that $b_c \rightarrow \infty$). Equation (2) then becomes:

$$I_{\text{corr}} = \frac{\Delta I}{\Delta E} \left(\frac{b_a}{2.3} \right) \quad (3)$$

If the iron is partially passive, the anodic Tafel value (b_a) can be relatively large (considerably larger than the 30-mV value used in commercial equipment). In one case at 150°C in oxygenated brine (Danielson 1980), the actual corrosion rate was six times greater than the rate measured by commercial equipment. Since B is a function of the corroding metal, time of exposure, and chemical environment, the commercial equipment cannot recognize the new conditions when B changes. The ICR will overcome these limitations by determining a B value at the same time the corrosion rate is measured.

There are three unknowns in the fundamental kinetic equation (b_a , b_c , and I_{corr}) that require a minimum of three data points for solution. Barnartt (1970) has investigated this technique and calls it the "three-point method." He found a closed-form solution for Equation (1) when the perturbing potentials have a certain relationship to each other: $|\Delta E|$, $2|\Delta E|$, and $-2|\Delta E|$ (ΔE is some potential perturbation typically in the range of 5 to 30 mV). The corrosion rate from Barnartt's work is:

$$I_{\text{corr}} = \frac{I_2 |\Delta E|}{r_2 [r_2^2 - 4(r_1)^{1/2}]^{1/2}} \quad (4)$$

$$\text{where } r_1 = \frac{I_2 |\Delta E|}{I_{-2} |\Delta E|}$$

$$r_2 = \frac{I_2 |\Delta E|}{I_{|\Delta E|}}.$$

Tafel slope parameters can also be calculated; but the major effect of this method is that the corrosion rate can be calculated even when B values are not constant, which results in a more flexible corrosion rate meter. The potential perturbation pattern is not limited to Barnartt's; several others were discussed in an earlier report (Danielson and Koski 1981).

FIELD TEST

The ICR was field tested at East Mesa, California, in early fiscal year (FY) 1981. It was installed in the PNL Bypass Test Facility, which uses brine provided by Magma Electric. Brine fluid flowed past the electrodes at 350 psig, 175°C, and 10 gal/min. Weight loss coupons and electrodes for the ICR were installed in a 2-in. pipe tee, and a commercial corrosion rate meter (CCR) and the ICR were used to take readings two or more times per day for the 4-1/2 day test. A longer test would have been preferred, but the Magma power plant shut down and brine was not available at the test facility.

The weight loss coupons and test electrodes (1018 steel) were mounted on a commercial electrode holder and installed in the bypass test loop. Weight loss corrosion rates were 9.1 ± 0.8 mils per year (mpy); ICR values, which were integrated over the experiment period, were 10.6 mpy; and integrated CCR values were 24.9 mpy. ICR and weight loss results agreed quite closely while the CCR system overestimated the corrosion rate by approximately 2-1/2 times. CCR results were larger than ICR results for the first 2 days of the experiment followed by closer agreement, which may imply that the kinetic parameters are changing with time until the corrosion film has reached a steady-state composition (see Figure 1). One of the principal improvements of the ICR over commercial equipment currently in use is that it recognizes these changes in kinetic parameters.

In a corroding system, the initial corrosion rates of a bare metal electrode are usually quite high until a protecting corrosion product film is established. For a test of short duration, it is particularly important that initial corrosion rate data be taken since these high corrosion rates act as a weighting factor on the integrated corrosion rate (as measured by weight loss coupons). After 24 h, ICR data showed a corrosion rate of <5 mpy; but the weight loss value was 9.1 mpy, indicating the influence of the early high corrosion rates.

The ICR used cathodic polarization currents measured at -20, -40, and -60 mV from the corrosion potential. As mentioned in a previous report (Danielson and Koski 1981), this pattern results in a more rapid achievement of a steady-state current and fewer imaginary solutions for the corrosion current.

FIGURE 1. Corrosion Rate Measurements During the Field Test at the East Mesa Test Facility

Since the corrosion potential was changing during the earliest part of the experiment (~ 4 mV/h), a 4-min cycle was set on the ICR (4 min per data period). After 24 h, an 8-min cycle was substituted. A rapidly changing open-circuit corrosion potential creates a problem for both the ICR and the CCR since they must assume that the corrosion potential is constant and make potential perturbations on that basis. If the corrosion potential is shifting, the potential perturbation is larger or smaller than assumed and the measured corrosion rates will be in error. For the ICR, the set of three measurements (needed to calculate a corrosion rate) must be completed before the corrosion potential shifts more than a few millivolts. Fortunately, these shifting potential conditions were only present at the start of the experiment when the corrosion rate was large. Generally, the higher the corrosion rate, the faster the currents reach a steady state; therefore, a shorter cycle time permitted adequate data to be taken.

After 24 h, other anodic potential variation schemes were tried; but they never worked as well as the -20 , -40 , and -60 mV polarizations. Imaginary solutions to Equation (4) with the ICR calculations were often the result. The all-cathodic polarization always gave real solutions that were of reasonable magnitude.

In general, the ICR performed better than the CCR in the field when their integrated corrosion rates were compared to weight loss measurements.

LABORATORY STUDIES

After the field test, further laboratory studies were conducted to determine the effect of oxygen—a difficult condition to control in the field. The presence of oxygen usually results in enhanced corrosion rates and can result in severe corrosion damage when undetected in field equipment.

The test solution composition consisted of 25 g/l of NaCl and 1.5 g/l of $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ stored in a 200-l polyethylene drum. Oxygen content was controlled at 1.0 ppm by mixing CO_2 and air in a flow ratio of 6 to 1. With this large supply of solution, experiments could be run at 200°C for 7 days with a refreshed autoclave rate of 20 ml/min. A 1-l titanium autoclave with

three lid penetrations was used, and two commercial electrode mounting fixtures were threaded into the bottom of the autoclave lid (see Figure 2). One specimen was used as the perturbed electrode, and the other was used as a pseudo-reference electrode. The autoclave was used as the counter electrode with the ICR electronics package and the CCR. The perturbed electrode and the weight loss coupons were both 1018 steel. The three weight loss coupons were mounted inside the autoclave using the fitting shown in Figure 2 except that the end with the wire lead was plugged to prevent contact with the solution.

Six 7-day experiments were conducted; many were done to work out experimental "bugs." There were two problems: 1) the rapid cycling of the corrosion potential with time, which made corrosion rate measurements difficult and 2) the wide divergence of individual corrosion rates for the weight loss coupons, which were sensitive to sample location inside the autoclave. Input levels of oxygen were 1 ppm; but outlet levels were about 0.10 ppm, indicating that corrosion processes were consuming most of the oxygen. The corrosion potential was cycled about ± 100 mV (measured between the working electrode and the iron pseudo-reference electrode) around the corrosion potential with a period of about 1.5 h. These observations tend to indicate that convection cells were created inside the autoclave with the result that nonuniform concentrations of oxygen were present and tend to imply that corrosion rates were under oxygen mass transport control.

FIGURE 2. Commercial Electrode Mounting Fixture Used in Laboratory Study

The magnitude and rate of the corrosion potential cycling were decreased to about ± 50 mV for 6 h by installing the PNL external reference electrode (Danielson 1979). With this change, corrosion measurements did not have to be taken on top of a moving open-circuit corrosion potential.

Two sets of data with identical chemistries are shown in Figures 3 and 4. The data in Figure 3 were taken using an iron pseudo-reference electrode, and those in Figure 4 were taken using a PNL external reference electrode. The integrated corrosion rates for the ICR and the CCR were 33 and 34 mpy, respectively; and weight loss measurements were 44 ± 1 mpy for two coupons and 12.5 mpy for the other coupon. The data from both rate meters agreed quite closely but were between the values for the weight loss coupons. However, a value of 33 mpy is obtained if the weight loss data are simply averaged.

FIGURE 3. Corrosion Rate Data Using the Iron Pseudo-Reference Electrode

FIGURE 4. Corrosion Rate Data Using the PNL External Reference Electrode

Using the PNL external reference electrode, ICR and CCR values were 13 and 21 mpy, respectively; and weight loss measurements were 8.6 ± 0.3 mpy for two coupons and 5.2 mpy for the other coupon. In this case, both the rate meters overestimated the corrosion rate although the ICR was the closer of the two.

These two experiments are interesting because they demonstrate the problem of carrying out a mass transport-controlled process in an autoclave with natural convective processes. With identical chemistries and flow rates, the weight loss measurements varied by about a factor of five. It is important to note that the the corrosion rate meters correctly "tracked" this phenomenon. Although agreement between the ICR and CCR was quite good in the laboratory, measurements made in the field and those reported earlier (Danielson and Koski 1981) indicate that the ICR was superior.

pH ELECTRODE STUDIES

Two commercial manufacturers have been under subcontract to PNL to develop a glass pH electrode that can operate at 250°C and 5000 psi. Both manufacturers have submitted final reports, and electrodes based on their designs arrived during the last half of FY 1981 and have been tested. Neither manufacturer was able to meet the pH response criteria of the original Request for Proposal (RFP); both electrodes survived 5000 psi, but pH response was not demonstrated at elevated temperatures. In fact, the manufacturer's performance criterion was to autoclave the electrodes and then test the pH response when returned to room temperature. When this criterion was used, both manufacturers noted pH sensor failure when autoclaved above 200°C.

Another company that has been pursuing high-temperature pH electrode development for nuclear applications has verified that their ZrO_2 -based electrodes have a near-Nernstian pH response at 285°C using a dilute acid and base as pH standards. When their internally funded research and development ended, PNL subcontracted with them to complete the project. This contract entails testing the pH response with buffers closer to neutrality (the pH region of interest in geothermal applications), working out the technique of pH standardization at temperature, and optimizing the composition of the pH-sensing membrane.

PNL's specific role is to test the pH response at elevated temperature to verify the response, examine problems with standardization at temperature, and generally become familiar with the electrodes so that they can be successfully used in a field test. The most important variable in water chemistry is pH, and successful demonstration of these devices would be an important milestone in the general field of process control instrumentation for geothermal systems.

ELECTRODE TESTS AT 95°C

A temperature of 95°C was chosen for the initial test for several reasons: 1) neither glass pH electrode vendor was able to demonstrate pH response at any temperature above 50°C because of experimental problems, 2) National Bureau of

Standards (American Society for Testing Materials 1957) buffer data are available at this temperature, and 3) a number of experimental problems can be worked out in this environment before moving to the more difficult autoclave environment.

Glass Electrode Results (Manufacturer A)

Manufacturer A delivered two styles of glass pH electrodes: two aqueous-filled models (phosphate buffer with KCl, $\text{Ag}^\circ/\text{AgCl}$ electrode) and 15 mercury-filled models (platinum electrodes). The mercury-filled model (see Figure 5) was developed after it was discovered that the aqueous-filled model suffered decreased pH response when autoclaved above 200°C . A hydrofluoric acid etch would restore ambient pH response, but aqueous-filled models suffered both internal and external damage to the pH sensing membrane after autoclaving.

Three buffers were chosen for these studies: two commercially available products with pHs of 7.12 and 4.21, respectively, at 95°C ; and one (American Society for Testing Materials 1957) 0.01-m sodium tetraborate with a pH of 8.83 at 95°C . These experiments were run in beakers inserted in a constant temperature bath, and large rubber stoppers with holes for the pH and reference electrodes were inserted into the beakers. The PNL external reference electrode (Danielson 1979) and a commercial off-the-shelf Ag, AgCl reference electrode (consisting of a combination pH-reference electrode) were used and both gave similar results.

Typical results are shown in Figure 6 for three mercury-filled electrodes at 25°C fitted using least squares. The slope was -55 to -50 mV/pH (theoretical Nernstian slope was -59 mV/pH), and the dc resistance of the electrodes was about 10^9 ohms. As a precaution, special shielded connections and wires were used to make electrical measurements. Noise was a problem, and all mercury-filled electrodes were extremely vibration sensitive at ambient and 95°C . An electrometer of $>10^{14}$ ohms input resistance was used to measure potentials; and even at 95°C , the measured potential asymptotically approached the final steady-state value as though it was charging a capacitor. Switching the input to the electrometer off and on would restart the process, and several minutes were required to reach a new steady state. Typical results at 95°C are shown in Figure 7; the theoretical Nernstian slope is -73.1 mV/pH. All of these

FIGURE 5. Mercury-Filled Glass pH Electrode (Manufacturer A)

FIGURE 6. pH Response of Three Mercury-Filled Electrodes (Manufacturer A) at 25°C

electrodes are significantly sub-Nernstian, which indicates that something is not functioning properly. A commercial pH electrode that was not intended for temperatures above 95°C was included in one test (Figure 7); and its performance was 97% of theoretical, which indicates that the buffers and other details of the experimental setup were satisfactory.

It was hypothesized that the noise and vibration sensitivity of the mercury-filled electrodes were due to the lack of a common charge carrier between the mercury (electronic conductor) and the glass (ionic conductor). The typical behavior of a mercury-filled electrode that had been electrolyzed (made cathodic) for 3 h at about 0.5 μ A is also shown in Figure 7. Although the pH response did not improve, the electrode lost its vibration sensitivity

FIGURE 7. pH Response of Three Mercury-Filled Electrodes (Manufacturer A) at 95°C. The response of a commercial pH electrode is included for comparison.

and the potential reached a steady-state value much quicker, losing that slow asymptotic response seen with the standard mercury electrode. Electrolyzing for longer than 3 h did not seem to improve the performance significantly.

Glass Electrode Results (Manufacturer B)

Ten glass pH electrodes (see Figure 8) were received from Manufacturer B late in the fiscal year, and a limited number of tests were performed. The manufacturer had not demonstrated Nernstian pH response at any temperature above ambient.

Two electrodes were separately tested at 95°C. The first electrode was tested twice and demonstrated no pH response, and it was concluded that this particular unit was defective. The second unit (#5) was tested; and it was determined by a least squares fit to have a pH response of 150% of theoretical

FIGURE 8. Glass pH Electrode (Manufacturer B)

(see Figure 9). There was a great amount of scatter in the data. Approximately 1 h was spent in each buffer, and this electrode suffered from potential drift. A commercial pH electrode (off-the-shelf model) used in the same test is also shown in Figure 9 for comparison, and its potential is "rock" steady and noise free.

Ceramic Electrode Results (Manufacturer C)

One ZrO_2 -based pH electrode was received late in the fiscal year from Manufacturer C (see Figure 10). This particular electrode had been autoclave tested by the manufacturer at 285°C and found to have an acceptable pH response. Three tests were conducted at 95°C with the typical response shown in Figure 9. The performance was always considerably less than Nernstian (20% of theoretical in Figure 9), and drift of the measured potential was a continual problem. At 95°C , the dc resistance was about 10^9 ohms. Conversations with the manufacturer indicated that they were aware of the sluggish response at lower temperatures and believe that it is due to voids in the slip-cast ZrO_2 where the pH buffers are trapped and slowly exchanged with the external environment. Consequently, the voids act as electrical shunts to the environment and give rise to a "mixed response." The manufacturer is currently developing a higher density product.

In conclusion, neither the glass nor the ceramic pH electrodes had a satisfactory response at 95°C .

ELECTRODE TESTS AT HIGHER TEMPERATURE (200°C)

Two tests were carried out in a refreshed titanium autoclave at 200°C . Two buffers (Macdonald, Wentrcik, and Scott 1980) were chosen for each test and were based on boric acid and lithium hydroxide-boric acid to give pHs of 4.99 and 9.06, respectively, at 200°C . Each buffer was maintained at 200°C for approximately 36 h to evaluate the steady state. When the buffer was changed, the autoclave volume had to be changed about 10 times before the potential readings were stable. The PNL external reference electrode (Danielson 1980) was used with 1.0-N KCl to minimize the liquid junction potential. Due to a limited number of autoclave lid penetrations, only two pH electrodes could be tested at a time. The ZrO_2 -based electrode was included in both tests.

FIGURE 9. pH Response of Glass (Manufacturers A and B) and Ceramic (Manufacturer C) Electrodes at 95°C. The response of a commercial pH electrode is included for comparison.

FIGURE 10. Ceramic pH Electrode (Manufacturer C)

The results of these tests are shown in Figure 11. The ZrO_2 electrode performed very well as evidenced by the reproducibility of the two experiments and on 80% of theoretical Nernstian response. Potentials were steady within 5 mV for periods of 18 h, and the electrode appeared undamaged after the tests.

The before and after condition of the aqueous-filled glass electrode of Manufacturer A is shown in Figure 12. It had a -2% of theoretical response, and potentials were fairly steady; however, the electrode was heavily etched and would not be expected to have a long life.

FIGURE 11. pH Response of Glass and Ceramic pH Electrodes at 200°C

The glass electrode of Manufacturer B had a response that was 63% of theoretical. The potential response of this electrode was noisy. When the autoclave was disassembled upon cooldown, it was discovered that the electrode had broken (see Figure 13). Unfortunately, it is impossible to determine when the electrode broke. The glass envelope was heavily etched, and enough material may have dissolved to weaken the envelope to the autoclave pressures.

In conclusion, none of the glass pH electrodes behaved satisfactorily at 200°C. They suffered from excessive dissolution attack of the pH membrane and poor pH response. However, the ZrO₂-based electrode performed quite well with an 80% of theoretical Nernstian response, no evidence of dissolution attack, and excellent reproducibility of potentials.

FIGURE 12. Before and After Views of Aqueous-Filled Glass pH Electrode (Manufacturer A)

BEFORE

AFTER

FIGURE 13. Before and After Views of Glass pH Electrode (Manufacturer B) That Broke During Testing

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Office of Geochemistry and
Geophysics
345 Middlefield Road
Menlo Park, CA 94025

O. Vetter
Vetter Associates
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Costa Mesa, CA 92626

R. C. Sones
Westec Services, Inc.
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