Electrochemical Noise Sensors for Detection of Localized and General Corrosion of
Natural Gas Transmission Pipelines

Final Report for the Period July 2001 – October 2002

December 2002

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TABLE OF CONTENTS

1.0 EXECUTIVE SUMMARY

The U.S. Department of Energy, National Energy Technology Laboratory funded a Natural Gas Infrastructure Reliability program directed at increasing and enhancing research and development activities in topics such as remote leak detection, pipe inspection, and repair technologies and materials. The Albany Research Center (ARC), U.S. Department of Energy was funded to study the use of electrochemical noise sensors for detection of localized and general corrosion of natural gas transmission pipelines. As part of this, ARC entered into a collaborative effort with the corrosion sensor industry to demonstrate the capabilities of commercially available remote corrosion sensors for use with the Nation's Gas Transmission Pipeline Infrastructure needs. The goal of the research was to develop an emerging corrosion sensor technology into a monitor for the type and degree of corrosion occurring at key locations in gas transmission pipelines.

Electrochemical noise equipment capable of being used in field installations was acquired from Intercorr International, Inc. The field instrument consisted of hardware that coupled electrochemical noise (EN) with linear polarization resistance (LPR) and harmonic distortion analysis (HDA), and software that provided continuous data analysis and is designed to operate with two or three electrode electrochemical sensors. All of the electrochemical corrosion tests were performed using three electrode electrochemical sensors identical in surface area and made of the same steel. Testing of the electrochemical noise sensors was conducted in the laboratory.

Corrosion rate measurements using EN, LPR, and HDA were made in three types of environments: 1) aqueous solutions typical of those found within gas pipelines in equilibrium with the corrosive components of natural gas (H_2O, NaCl, CO_2, and O_2); 2) biologically-active soils typical of wetlands (soil, H_2O, and NaCl); and 3) a simulated, unpressurized, internal gas/liquid (H_2O, CO_2, O_2, and NaCl,) gas pipeline environment. Gravimetric measurements in aqueous and soil environments were conducted in parallel with the electrochemical measurements. For the aqueous and soil tests, three cylindrical electrode electrochemical sensors were made from API Grade X42 transmission pipe. For the simulated gas pipeline environment, three ASTM A105 steel flanges and three disc electrodes made from No. 12, Grade A615 steel rebar were used as the electrochemical sensors.

The corrosion rates from EN, LPR, and HDA measurements show good agreement with each other and with the gravimetric weight loss. The corrosion measurements in simulated internal aqueous gas pipeline environments showed that the EN corrosion rates are sensitive to small changes in oxygen and carbon dioxide contents present in the aqueous phase. The corrosion measurements in simulated external soil environments showed that the EN corrosion rates are sensitive to the amount of water and salt in the soil and responds to temperature changes. The corrosion measurements in the simulated gas pipeline environment showed that corrosion rates and pitting factors could be measured in a pipeline using different configurations.

Using the sensor configurations described here, EN with LPR and HDA techniques are excellent for monitoring the occurrence of corrosion in gas transmission pipelines. It is recommended that the EN sensors be evaluated in a field test in conditions found in the Gas Transmission Pipeline network.

2.0 BACKGROUND/PROGRAM GOALS

2.1 Background

An analysis[1] of 5872 pipeline incidents in the U.S. from 1970 to 1984 reported that 54 % of the service failures of gas pipelines were attributable to outside forces such as earth movement, weather, and equipment operation by outside parties, 17 % to corrosion, and 17 % to material failures. A later analysis[2] of 12,137 failures in Canada from 1980 to 1997 concluded that 63% of pipeline failures were caused by corrosion with 50% due to internal corrosion and 13% due to external corrosion. Corrosion failures represent a significant proportion of the total number of failures. Sensors that can detect internal and external corrosion in real time before failure occurs will enhance gas transmission pipeline reliability.

Gas transmission pipelines are susceptible to both internal (gas side) and external (soil side) corrosion attack. Internal corrosion is caused by the presence of water, salt, carbon dioxide, oxygen, methane, and hydrogen sulfide in the natural gas. There are certain areas of the U.S. that are more prone to this type of corrosion than others. This is due to the production of moist sour gas in areas such as Louisiana and Texas, for example. This type of attack usually manifests itself as general corrosion, forming either non-protective carbonate scales or semi-protective sulfide scales. The presence of chlorides (salt) in entrained water also can lead to pitting corrosion. Pitting (localized) corrosion can more rapidly penetrate a steel pipeline than general corrosion, resulting in leaks or rupture of the pipe.

External corrosion of gas transmission pipelines is usually controlled by the application of various polymeric coatings augmented with cathodic protection (CP). When corrosion does occur on the outside of the pipeline, the combination of general and localized corrosion with the high stresses in the pressurized pipelines can sometimes lead to stress corrosion cracking (SCC). In cases where CP is inadequate or non-existent, pipelines exposed to ground waters can experience transgranular SCC due to low pH (6.5) carbon dioxide containing water.[3] Even when CP is adequate, gas pipelines may be susceptible to intergranular SCC due to the higher pH external environment generated by CP.[3]

There are a variety of electrochemical techniques that can be used to measure the corrosion rates of metals in almost any process environment. Of all of the electrochemical techniques, electrochemical noise (EN) has the most potential for being used successfully to measure general and localized corrosion rates of equipment. EN is increasingly being applied to field and industrial installations for *in situ* corrosion monitoring.

2.2 Research Objectives

In this investigation, EN was evaluated to determine its suitability for monitoring internal and external corrosion damage on gas transmission pipelines. The objective of this research was to demonstrate the use of electrochemical noise sensors for measuring the internal and external corrosion of natural gas transmission pipelines. The results of the studies were used to determine

if EN could properly quantify corrosion rates in natural gas pipelines and make a recommendation for a field test of the equipment.

3.0 RESEARCH PROGRAM

3.1 Electrochemical Noise Equipment

Electrochemical noise equipment capable of being used in field installations was acquired from Intercorr International, Inc., the world leader in EN technology. The field instrument (Figure 1) consisted of a three-electrode sensor with hardware that couples electrochemical noise with linear polarization resistance (LPR) and harmonic distortion analysis (HDA), and software that can provide continuous data analysis.

Figure 1 - Electrochemical noise equipment.

The electrochemical noise technique can differentiate general from localized corrosion and provide estimates of corrosion rates without external perturbation of the corroding system. EN measurements are based on fluctuations in electrochemical potential and corrosion current that occur naturally during corrosion. Electrochemical potential is related to the driving force (thermodynamics) of the reaction, while corrosion current is related to the rate (kinetics) of the reaction. Electrochemical events on the surface of a corroding metal will generate fluctuations (noise) in the overall potential and current signals. Each type of corrosion (for example general corrosion, pitting corrosion, crevice corrosion, cavitation attack, and stress corrosion cracking) will have a characteristic "fingerprint" or "signature" in the signal noise. This "fingerprint" can be used to identify the type and severity of corrosion that is occurring.[4]

Electrochemical noise measurements refer to the measurements of the fluctuations in current or potential that occur between two coupled identical electrodes at the open circuit corrosion potential. The fluctuations in the corrosion current and potential occur because of changes in anodic and cathodic areas.

When general corrosion is occurring on the metal surface, the electrochemical noise has a relatively smooth appearance (Figure 2). For general corrosion, it is possible to relate the potential noise and current noise mathematically to calculate the noise resistance (the polarization resistance derived from EN) for the system.[4] The noise resistance, R_n, is defined as the ratio of the standard deviation of the electrochemical noise potential signals to the standard deviation of the electrochemical noise current signals. These signals are generated spontaneously by corroding electrodes in the probe.
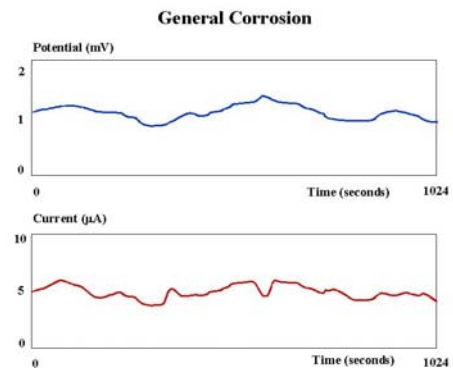
Figure 2 - General corrosion graphs.[4]

$$R_n = \sigma_{V_n} / \sigma_{i_n} \qquad\qquad (1)$$

σ_{V_n} = Electrochemical potential noise (standard deviations).

σ_{i_n} = Electrochemical current noise (standard deviations).

The corrosion current density (i_{corr}) is obtained from the Stern-Geary approximation,

$$i_{corr} = B / R_n \qquad\qquad (2)$$

where B is the Stern-Geary constant. The Tafel coefficients are calculated from Harmonic Distortion Analysis and B is calculated using the Tafel coefficients. The corrosion current density is used to calculate the general corrosion rate by means of Faraday's law.

When pits begin to form on the metal surface or initiate, occasional sharp increases and decreases in the amplitude of both the potential and current noise data occur (Figure 3). The amplitude can change from a few millivolts to hundreds of millivolts, depending on the metal or alloy being studied. When the existing pits continue to grow or propagate, the amplitude of both the potential and current noise continues to increase (Figure 3).
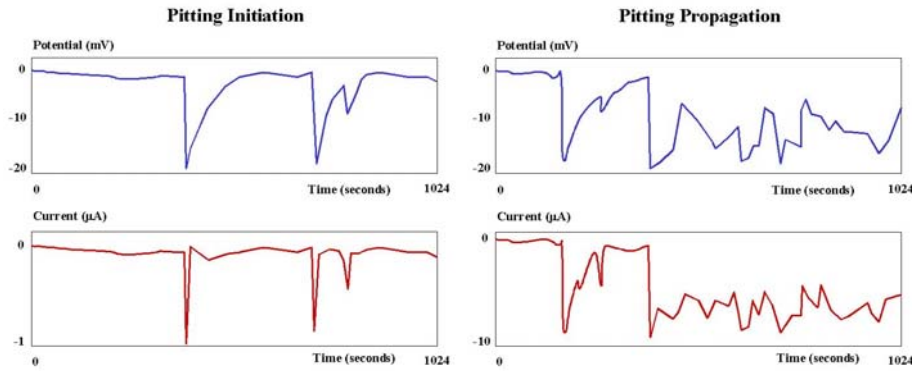

Figure 3 - Pitting initiation and propagation graphs.[4]

For this form of localized corrosion, the risk of pitting on the metal surface is derived from the electrochemical noise and the harmonic distortion analysis data. This value is termed the Pitting Factor (PF).[4] PF refers to the risk of localized attack (pitting) on the metal surface, and is always examined together with corrosion rate. The PF has a value between 0 and 1. As the value approaches 1, the system will be in a pitting regime rather than a regime of general corrosion.

The calculation for the PF is as follows:

$$PF = \sigma_{i_n} / (A * i_{corrHDA}) \qquad\qquad (3)$$

σ_{i_n} = Electrochemical current noise (standard deviations)

A = Electrode surface area, cm^2

$i_{corrHDA}$ = Corrosion current density from Harmonic Distortion Analysis, A/cm^2

Table 1 shows that when the PF is between 0 and 0.1 pitting does not occur. If the PF is between 0.1 and 0.2, then pitting may occur on the metal surface. If the PF is greater than 0.2, then pitting is occurring on the metal surface.[4]

Table 1 - Risk of pitting on the metal surface[4]

Pitting Factor	Form of Corrosion
0 – 0.1	General Corrosion
0.1 – 0.2	Tendency towards localized corrosion or pitting
0.2 – 1.0	Localized Corrosion / Pitting Regime

Linear polarization measurements involve the in-phase measurement of the current response to a small amplitude (± 25mV) sinusoidal polarization of the electrodes under potentiostatic control. The polarization resistance, R_p, is then calculated from 100 pairs of data points using Fourier transform techniques. The corrosion current density (i_{corr}) is obtained from the Stern-Geary approximation, equation 2. The general corrosion rate is calculated from the corrosion current density by means of Faraday's law.

Harmonic distortion is a measure of the non-linear current distortion arising during the linear polarization measurement. The data is analyzed (using Fast Fourier Transform analysis) to provide a measurement of the corrosion current, and to provide an on-line calculation of the Tafel and Stern-Geary constants. The general corrosion rate is calculated from the corrosion current density by means of Faraday's law. Harmonic distortion analysis measurements become unstable under pitting corrosion conditions (or other localized attack). However, this phenomenon, together with an increase in the PF can be used to alert the operator to pitting of the metal surface.

3.2 Research Approach

3.2.1 Electrochemical Noise Tests

All of the electrochemical corrosion tests were performed using three-electrode electrochemical sensors identical in surface area and made of the same material. The first electrode was used as the working electrode, the second as the counter electrode, and the third as the reference electrode. Electrochemical corrosion rate measurements were made in three types of environments: 1) aqueous solutions typical of those found within gas pipelines in equilibrium with the corrosive components of natural gas (H_2O, NaCl, CO_2, and O_2), 2) biologically-active soils typical of wetlands (soil, H_2O, and NaCl); and 3) a simulated, unpressurized, internal gas/liquid (H_2O, CO_2, O_2 and NaCl,) gas pipeline environment. Gravimetric measurements in aqueous and soil environments were conducted in parallel with the electrochemical measurements.

For the aqueous and soil tests, cylindrical electrodes were made from X42 transmission pipe. The chemical composition of the cylindrical electrodes is shown in Table 2. The electrodes were 5 cm in length and 0.79 cm in diameter. The exposed area was 12.5 cm^2. The cylindrical

electrodes finished to 600 grit with SiC paper. Prior to the test the cylindrical electrodes were cleaned and degreased in methanol.

Table 2 - Chemical composition of the electrodes

Electrodes	Standard	Chemical composition, wt %					
		C	Mn	P	S	Si	Fe
Cylindrical	API Grade X42	0.27	1.25	0.03	0.03	NA	Bal
Flange	ASTM A105	0.35	0.60	0.04	0.05	0.35	Bal
Disc	No. 12, Grade A615 rebar*	0.31	1.36	0.013	NA	0.25	Bal

NA – not applicable
* also Cu, 0.41; Ni, 0.14; Cr, 0.081; Mo, 0.27

The electrochemical tests used for measuring the corrosion of gas transmission pipeline metal in aqueous solutions (type 1) typical of those found within gas pipelines were performed in the corrosion cell shown in Figure 4. Two lexan containers sealed with silicone were used; one for the corrosion cell (Figure 4) and one for a trap to minimize carry-over of liquid in the off-gas stream. In a previous study,[6] where the internal corrosion behavior of gas transmission pipeline material was measured in solutions representative of those found in the field, analyses showed that these solutions contained chloride in the form of calcium, potassium, magnesium and sodium chlorides. The Cl^- concentration ranged from approximately 0.006 wt % to 0.13 wt %. For the aqueous electrochemical tests, the solution used was 3.5 wt % NaCl solution (a standard laboratory solution) with 1000 mL/min N_2, 0-2 mL/min O_2 and 0-20 mL/min CO_2. The test was performed at room temperature.

Figure 4 - Test cell for aqueous solutions with three electrodes.

For measuring the external corrosion of gas transmission pipelines (type 2), electrochemical noise tests were performed in soil. The test cell (Figure 5) was a typical soil box as specified in ASTM G57, "Field Measurement of Soil Resistivity Using the Wenner Four-Electrode Method." Three cylindrical electrodes of X42 gas transmission pipeline steel were inserted into the soil box as the corrosion sensors and four stainless steel electrodes were inserted for measuring soil resistivity. The soil resistivity was measured on a daily basis and the pH measured before and after each test.

Figure 5 - Soil test cell with three electrodes.

The soil used was Dayton type soil, which is defined as fine, montmorillonitic, mesic Typic Albaqualf, and is poorly drained soil. It was taken

from the well-characterized Lake Creek site in the southern Willamette Valley in Western Oregon near an oil pipeline. The soil was air dried and crushed prior to placing it in the test cell. Table 3 shows that the air dried soil contained 12.6 wt % water, 0.129 wt % Cl^- and had a pH of 6.00.

Two electrochemical tests were performed. In the first experiment, 2488 mL (16 wt %) of additional water was added to the "as received" soil over a period of time in increments of 340 mL. In the second experiment, 3000 mL (20 wt %) of additional water in increments was added to soil containing 1 wt % of NaCl in increments of 300 mL. Table 3 shows that after NaCl was added, the soil contained 7.6 wt % water, 0.655 wt % Cl^- and had a pH of 5.72. The test was performed at room temperature, with no gases added, and left open to the atmosphere.

Table 3 - Soil analyses

Soil	pH	Cl^-, wt %	H_2O, wt %
"As received"	6.00	0.129	12.6
+ 1 wt % NaCl	5.72	0.655	7.6

For measuring the internal corrosion of gas transmission pipelines in simulated internal gas/liquid pipeline environments (type 3), electrochemical measurements were performed in a pipeline cell. The apparatus for this phase of the research cons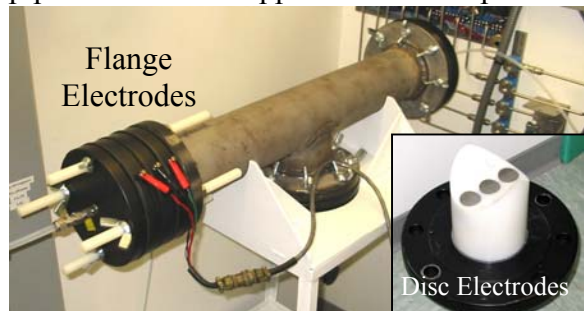isted of a section of 4" (10.2 cm) diameter X42 gas transmission pipe, with $^3/_8$" wall thickness, containing two different sensor configurations (Figure 6). The first sensor configuration consisted of three insulated carbon steel flanges in series with the pipe. The chemical composition of the flange electrodes is shown in Table 2. The flange electrodes were cleaned and degreased and the surface area left as received. The total exposed area was 60.76 cm^2 per flange electrode. The second sensor configuration consisted of three solid disc electrodes (Figure 6 inset) inserted at the bottom of the pipe in a tee adapter. The disc electrodes were machined from No. 12, Grade A615 rebar (Table 2). The exposed area of each of the disc electrodes was 6.5 cm^2. The disc electrodes were finished to 600 grit with SiC paper. Prior to the test the disc electrodes were cleaned and degreased in methanol. The electrochemical noise experiment was performed in an environment that consisted of water saturated 1000 mL/min N_2; 0, 5, and 10 mL/min CO_2; 0, 2, and 4 mL/min O_2 gas phase; and 800 mL of 3.5 wt % NaCl added over time.

Figure 6 – Pipeline test cell with three flange electrodes and three disc electrodes (inset) in the side arm.

3.2.2 Weight loss measurements

Weight loss (gravimetric) measurements were made only on the cylindrical X42 electrodes in types 1 and 2 media environments. The areas were measured and the electrodes degreased in methanol, and weighed. After the corrosion tests, the electrodes were chemically cleaned (similar to ASTM G1-72) of the corrosion product in a 12 volume % H_2SO_4 solution containing

2.5 mL of Rodine 95 per liter of solution. After cleaning and drying, the electrodes were weighed and the mass loss corrosion rates in millimeters per year (mmpy) were calculated.
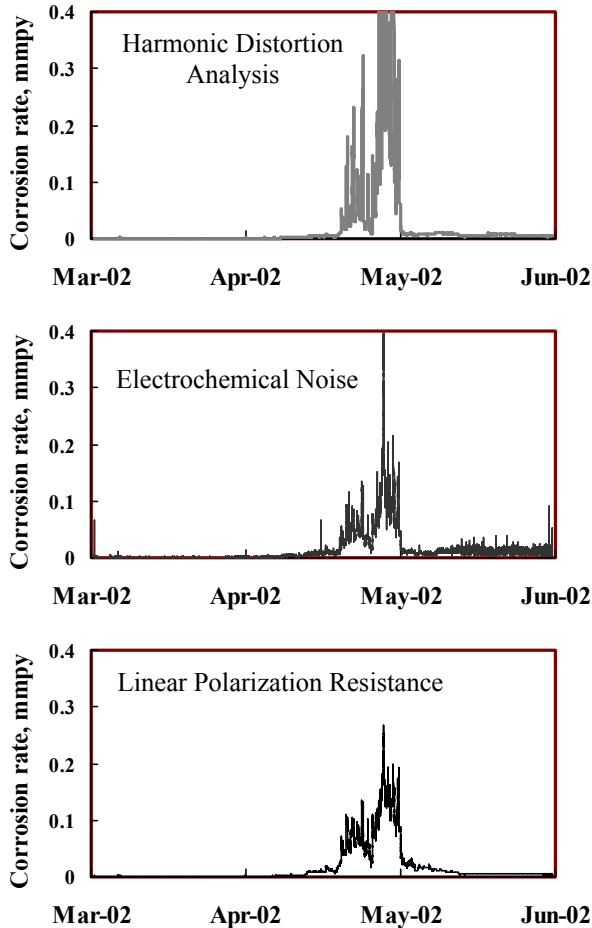
4.0 RESULTS

Figure 7 – Comparison of corrosion rates from harmonic distortion analysis, electrochemical noise, and linear polarization resistance measurements.

The X42 gas transmission pipe cylindrical electrodes were exposed to a 3.5 wt % NaCl solution (type 1) for 6 weeks. Figure 8 shows the EN corrosion rate as a function of time.

The EN corrosion rate stayed at 0.1 mmpy for a month, rose briefly to 0.4 mmpy, and then dropped back to 0.1 mmpy. During this 6-week period, O_2 and CO_2 levels were varied and responsible for these changes in corrosion rate. Figure 9 shows the same EN corrosion rates

The instrument used in this research has multi-technique capabilities for combined electrochemical monitoring. The comparison of the corrosion rates determined from harmonic distortion analysis, electrochemical noise, and linear polarization measurements of the cylindrical electrodes in soil is shown in Figure 7. There was good agreement between the three techniques.
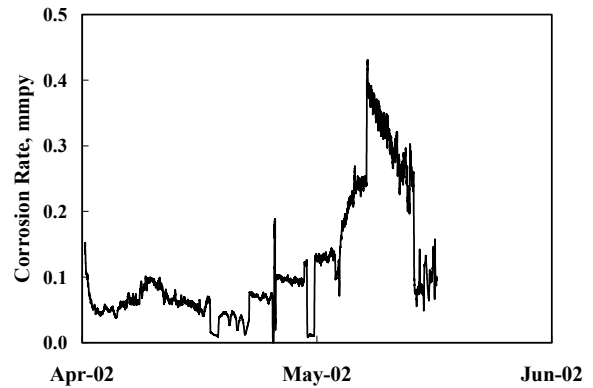

Figure 8- EN Corrosion rate of X42 transmission pipeline electrode in 3.5 wt % NaCl aqueous solution.
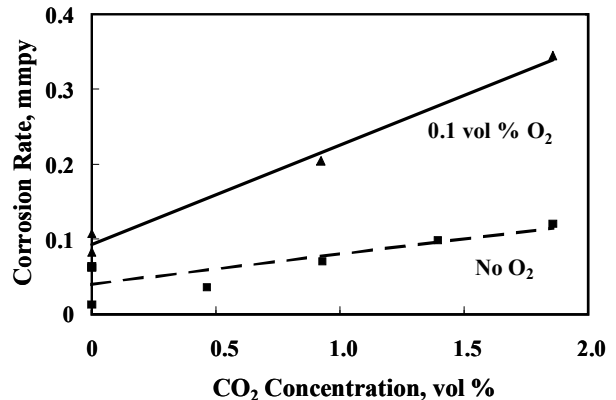

Figure 9 – The effect of O_2 and CO_2 on the EN corrosion rate of X42 transmission pipeline electrode in 3.5 wt % NaCl aqueous solution.

11

plotted as a function of O_2 and CO_2 concentration in volume per cent. The results showed that increasing the O_2 and CO_2 concentrations increased the corrosion rates.

The corrosion rate of the X42 gas transmission pipeline steel cylindrical electrodes in "as received" soil (type 2), volume (mL) of water added, and the resistivity of the soil as a function of time are shown in Figure 10. For soil to be corrosive, there must be: oxygen, moisture, and soluble salts.[6] Table 3 shows the moisture and salt contents before and after the tests. In the "as
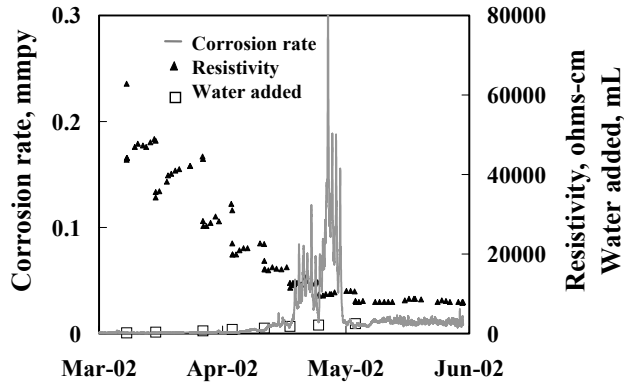

Figure 10 – Effect of resistivity and water on the EN corrosion rate of X42 transmission pipeline electrodes in "as received" soil. The squares represent mL of water added. The triangles represent the resistivity of the soil.

received" soil experiment, as 2488 mL (16 wt %) of additional water was added to the soil over a period of 2.5 months, the resistivity decreased from relatively less corrosive[6] (> 60,000 ohms-cm) to moderatively corrosive[6] (8000 ohms-cm). Figure 10 shows that beginning at 1028 mL (8 wt %) added water and a resistivity of 10,000 ohms-cm, the corrosion rate increased to 0.15 mmpy. At 2488 mL (16 wt %) additional water and a resistivity of 8000 ohm-cm, the corrosion rate decreased to <0.01 mmpy, possibly due to the lack of availability of oxygen in the water-saturated soil.
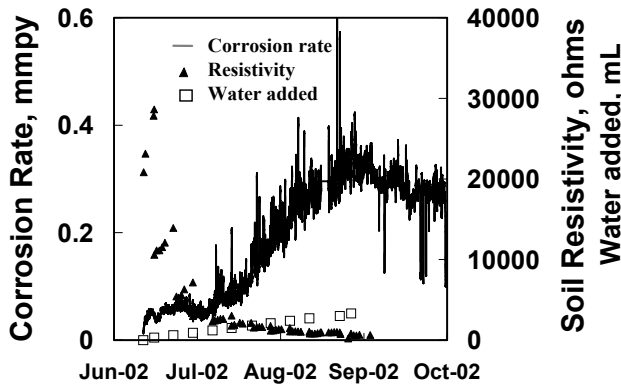

Figure 11 – Effect of resistivity and water on the EN corrosion rate of X42 transmission pipeline electrodes in soil + 1 wt % NaCl. The squares represent the mL of water added. The triangles represent the resistivity of the soil.

Figure 11 shows the corrosion rate of the X42 gas transmission pipe cylindrical electrodes in soil +1 wt % NaCl (type 2), and the volume (mL) of water added, and the resistivity of the soil as a function of time. In this environment, after the addition of only 300 mL (2 wt %) additional water, the resistivity dropped from 35,000 to 10,000 ohms-cm and the corrosion rates began increasing. At approximately 2400 mL (~16 wt %) of added water, the resistivity dropped to 1000 ohms-cm and the corrosion rates became constant possibly due to the lack of availability of oxygen in the water-saturated soil.
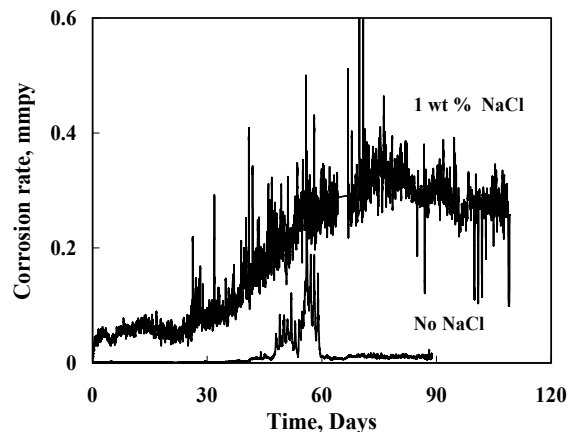

Figure 12 – Effect of NaCl on the EN corrosion rate of X42 transmission pipe cylindrical electrodes in soil.

The effect of NaCl on the corrosion rate of the X42 gas transmission pipe cylindrical electrodes in soil is shown in Figure 12. NaCl increased the corrosion rate 35 fold in soil. The response of these soil corrosion rates to increased room temperature and moisture is shown in Figure 13. In the soil experiments, when the water and air temperatures increased, the corrosion rates increased.

Figure 14 shows the effect of NaCl on the pitting factor of the X42 transmission pipe electrodes in soil. The electrodes in the "as received soil" showed pitting factors between 0.0001 and ~0.01 indicating that there was no
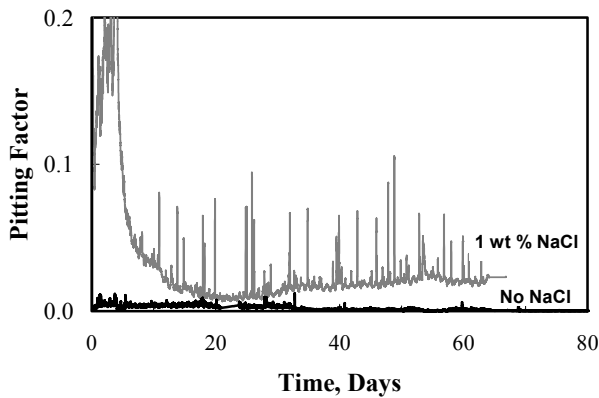
Figure 13 – Effect of water and temperature on the EN corrosion rate of X42 transmission pipeline electrodes in soil.

pitting. The electrodes in the soil with 1 wt % NaCl showed an initial spike between 0.1 and 0.2 indicating that pitting may have initiated on the metal surface, then decreased, possibly due to pit repassivation or to a transition to general corrosion. After 20 days the pitting factor remained at approximately 0.02 until the end of the experiment. Examination of these electrodes after removal from the soil showed a rough general corrosion of the majority of the surface and some pitting of the ends.

Figure 14 – Effect of NaCl on the Pitting Factor of X42 transmission pipe electrodes in soil.

The cylindrical X42 transmission pipe electrodes for the aqueous and soil tests were measured and weighed before each test and cleaned and weighed after the test. A gravimetric corrosion rate in mmpy was calculated for each electrode. Table 4 shows the comparison between the electrochemical noise and gravimetric corrosion rates. The corrosion rates measured by the two different techniques, show good agreement.

Table 4 - Corrosion rate comparison		
Test	EN CR, mmpy	Gravimetric CR, mmpy
3.5 %NaCl sol'n	0.095	0.039
Soil	0.005	0.004
Soil +1 wt % NaCl	0.349	0.364

For the corrosion tests in simulated internal gas/liquid pipeline environments (type 3), one sensor configuration consisted of three insulated steel flanges in series with the pipe. The flange electrodes were exposed to both the liquid and gas phases. The second sensor configuration
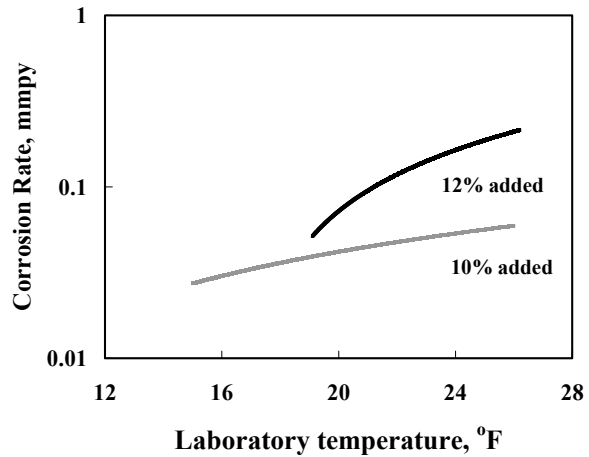
consisted of three solid disc electrodes inserted at the bottom of the pipe in a tee adapter. These three disc electrodes remained submerged in the liquid phase throughout the experiment.

Figure 15 shows the corrosion rates of the flange and disc electrodes as a function of time. The corrosion rates of the disc electrodes were approximately 0.01 mmpy throughout the test. The corrosion rates of the flange electrodes increased from 0.15 to 0.35 after two months. The higher corrosion rate of the flange electrodes compared to the disc electrode could be due to corrosion at the interface between the liquid and gas phases for the flange electrodes as opposed to corrosion in the liquid phase only for the disc electrodes. The flange and disc electrodes were also made from different materials (Table 2) and had different surface finishes, commercial finish (usually 120 to 180 grit) for the flange electrodes and 600 grit for the disc electrodes. Figure 16 shows the pitting factors of the flange and disc electrodes as function of time. Initially the Pitting Factor for the flange electrode was above 0.2 (in the pitting regime). The Pitting Factor for the flange electrode dropped to 0.001 possibly due to pitting repassivation and remained at 0.001 throughout the remainder of the test. The Pitting Factor for the disc electrodes remained below
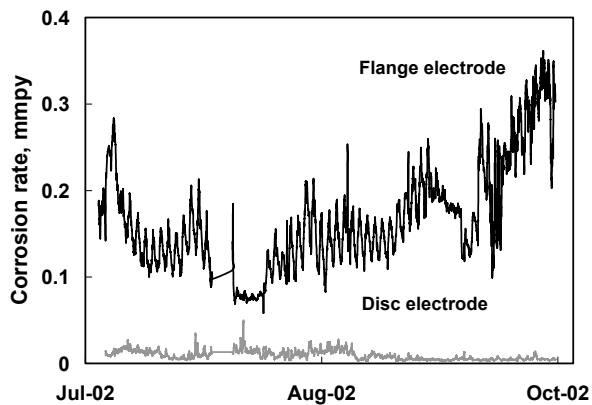
Figure 15 – EN Corrosion rates of flange and disc electrodes as a function of time.
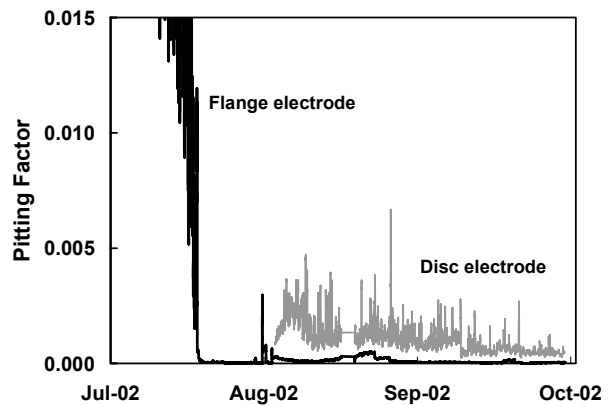
Figure 16 – Pitting Factors of flange and disc electrodes as a function of time.

0.1 throughout the test, indicating no pitting. electrodes showed no pits.

Visual examination of the flange and disc

5.0 PROJECT SUMMARY

EN corrosion measurements were made in three types of environments: 1) aqueous solutions typical of those found within gas pipelines in equilibrium with the corrosive components of natural gas (water, salt, carbon dioxide, and oxygen); 2) biologically-active soils typical of wetlands (soil, water, and salt); and 3) in simulated, unpressurized, internal gas/liquid (carbon dioxide, oxygen, and water) gas pipeline environment. Gravimetric measurements were conducted in parallel with the electrochemical noise measurements to validate the results.

- Using the sensor configurations described here, EN with LPR and HDA techniques are excellent for monitoring the occurrence of corrosion inside and outside natural gas transmission pipelines.

- Electrochemical noise equipment was acquired from Intercorr International,Inc. The equipment consisted of a three-electrode sensor with hardware capable of using EN, LPR, and HDA, and software that can provide continuous data analysis and can be used for field measurements.
- The corrosion rates from electrochemical noise, linear polarization, and harmonic distortion analysis measurements are in good agreement.
- Corrosion rates are increased by the presence of oxygen and carbon dioxide in the liquid phase.
- Corrosion rates and Pitting Factors are increased by moisture and salt in the soil and by increasing temperature.
- EN corrosion rate values are in good agreement with the gravimetric corrosion rate values.
- General corrosion is higher in the presence of a gas/liquid interface in the pipeline environment.
- No pitting occurred in the pipeline environment.
- Corrosion rates and Pitting Factors can be measured in a pipeline using different configurations.

Evaluation: EN sensors are sensitive enough to detect corrosion in and around natural gas transmission pipelines and they can be used to detect corrosive conditions and process changes. EN sensors could be placed at regular intervals inside a natural gas transmission pipeline and will be able to detect the presence of corrosive conditions and process changes. EN sensors could be placed outside a natural gas transmission pipeline at critical/corrosive sites and detect the presence of corrosive conditions as groundwater compositions and levels change. EN noise sensors are shown to be suitable candidates for internal corrosion sensors and for soil corrosion sensors for natural gas transmission pipeline enviroments.

Recommendation: EN sensors should be evaluated in a field test in conditions found in the Natural Gas Transmission Pipeline network.

6.0 COOPERATOR/ACKNOWLEDGEMENTS

A Memorandum of Understanding was signed with RMOTC (Rocky Mountain Oil Testfield Center). Initial arrangements for a future field test of the EN technology were initiated.

NW Natural, Portland, OR provided samples of X42 gas transmission pipeline pipe for electrodes, coupons, and a gas transmission pipeline test cell for research.

Professor John Baham, Department of Crop and Soil Science, Oregon State University, Corrvallis, OR, provided the soil and soil analyses.

ALRC is a research partner with David Eden, InterCorr International Ltd., Houston, TX to demonstrate the capabilities of commercially available remote corrosion sensors for use with the Nation's Gas Transmission Pipeline Infrastructure needs.

Sean M. Garrett, student at Oregon State University, Corvallis, OR, for drafting the disc electrodes and electrode holder

Publications and Presentations

1) Gordon R. Holcomb (DOE), Bernard S. Covino, Jr. (DOE), David Eden (InterCorr International Ltd) "State-of-the-Art Review of Electrochemical Noise Sensors," http://www.netl.doe.gov/scng/publications/t&d/tsa/ENStateoftheArt.pdf

2) Bernard S. Covino, Jr., and Gordon R. Holcomb, "The Use of Electrochemical Noise Technology to Measure Corrosion of Gas Transmission Pipelines" RMOTC presentation, Casper, WY, July 5, 2001

3) Sophie Bullard, Bernard Covino, Stephen Cramer, Gordon Holcomb, James Russell, Margaret Ziomek-Moroz "Electrochemical Noise Sensors for Detection of Localized and General Corrosion of Natural as Transmission Pipelines" Albany Research Center FY 2002 On-site Review, August 20-21, 2002

4) G. R. Holcomb, S. J. Bullard, B. S. Covino, Jr., S. D. Cramer, J. H. Russell, and M. Ziomek-Moroz, "Electrochemical Noise Sensors for Detection of Localized and General Corrosion of Natural Gas Transmission Pipelines," U.S. Department of Energy, Natural Gas Infrastructure Reliability Industry Forums, Morgantown, WV, Sept 16-17, 2002

5) Sophie J. Bullard, Bernard S. Covino, Jr., James H. Russell, Gordon R. Holcomb, Stephen D. Cramer, and Margaret Ziomek-Moroz, "Laboratory Evaluation of an Electrochemical Noise System for Detection of Localized and General Corrosion of Natural Gas Transmission Pipelines" to be published and presented at CORROSION/2003 (San Diego CA), NACE International, Houston TX, April, 2003

7.0 REFERENCE

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8.0 Appendix A.

State-of-the-Art Review of Electrochemical Noise Sensors

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State-of-the-Art Review of Electrochemical Noise Sensors

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Introduction

There are a number of different techniques capable of being used to measure corrosion within equipment. The most simple, the use of metal coupons, usually causes the process to be shut down, is manpower intensive, and has a time delay in getting the required corrosion information. Electrical Resistance (ER) techniques are often used but their response is very sensitive to temperature and they cannot differentiate between general and localized corrosion. Electrochemical techniques, such as linear polarization resistance (LPR), electrochemical noise (EN), electrochemical impedance spectroscopy (EIS), harmonic distortion analysis (HDA), and electrochemical frequency modulation (EFM), have the capability of solving most of those drawbacks. Electrochemical probes can be mounted permanently in most equipment, give regular measurements of the intensity of corrosion, and some can detect localized corrosion.

Of all of the electrochemical techniques, EN has the most potential for being used successfully to measure general and localized corrosion rates of equipment. The EN technique was studied in the late 1970s and early 80s as a means of detecting localized (stochastic) corrosion phenomena, such as occurs with pitting, crevice and cavitation attack. EN measurements are based on fluctuations in electrochemical potential and corrosion current that occur during corrosion. Electrochemical potential is related to the driving force (thermodynamics) of the reaction, while corrosion current is related to the rate of reaction (kinetics) of the reaction. The idea is that random electrochemical events on the surface of a corroding metal will generate noise in the overall potential and current signals. Each type of corrosion (for example general corrosion, pitting corrosion, crevice corrosion, and stress corrosion cracking) will have a characteristic "fingerprint" or "signature" in the signal noise. This "fingerprint" can be used to predict the type and severity of corrosion that is occurring.

By comparison, conventional electrochemical techniques such as LPR, EIS, HDA and EFM rely on a steady-state analogy for the determination of *general* corrosion rates. Early studies were carried out using potential EN measurements, using time domain, statistical and frequency domain analyses to characterise the electrochemical response of systems undergoing localised

corrosion. Current EN measurements followed quickly using zero resistance ammetry to study the current noise between two identical electrodes. For general corrosion processes, EN has been demonstrated independently by several workers to provide information similar to LPR. Noise technology has been used to study systems undergoing very low to very high rates of corrosion, for example, coatings performance, passive systems undergoing pit initiation/propagation, condensing systems, systems undergoing stress corrosion cracking, and general corrosion through to the very high corrosion rates experienced during chemical cleaning processes.

This review will describe: state of the art methods and probes used to measure EN, data acquisition requirements, theory to analyze the signal and to relate the signal to corrosion rates and types, the results of EN field trials, and laboratory results in environments similar to gas-pipelines.

EN Measurements, Methods, and Probes

Electrochemical noise sensors typically consist of three electrodes, in one of three arrangements: WE-RE-CE, WE-WE-RE, and WE-WE-WE, where WE is a working electrode (consisting of metal of the same type that is being monitored), RE is a reference electrode (an electrode that maintains a constant potential in the environment), and CE is a counter electrode (typically platinum). The WE-RE-CE arrangement is extensively used in laboratory settings for electrochemical studies under polarized conditions. In contrast, the WE-WE-RE arrangement could be used for EN measurements without potential control (i.e., freely corroding). The two WEs allow for the simultaneous measurements of current and potential noise. It is widely used in plant monitoring and surveillance situations. Special WE arrangements can be used to monitor specific types of corrosion. For example, when susceptibility to stress corrosion cracking (SCC) needs to be monitored, the two WEs could consist of one unstressed electrode and one stressed electrode. The third type uses three working electrodes. This type can be useful when reference electrodes are impractical or otherwise unsatisfactory. [Eden]

The sensor array design depends upon the application and the type of corrosion being monitored. Consideration must be given to maintain electrical isolation between the electrodes, to have materials able to withstand the environment, to have a surface appropriate to the type of corrosion being monitored, and to the conductivity of the environment. Some example sensor arrangements and considerations are:

> **General Corrosion:** In general corrosion studies, typically in low-pressure, low-temperature aqueous systems with high conductivities, a three-finger electrode assembly is often used. The electrodes protrude into the solution through o-ring seals and are combined into one fitting through the container wall. [Eden]

> **Flowing Conditions;** In flowing conditions such as in oil and gas flow-lines, high-pressure spool-piece fittings can be used that replace a section of pipe. The fitting consists of three circumferential rings of WEs that are separated from each other and the pipe flanges with insulators. [Eden]

> **Pitting:** Sensors for detecting pitting must have sufficient surface areas so that the probability of attack during monitoring is high. Eden (1998) suggests a surface area above 10 cm^2. Care must also be taken to eliminate crevices in the design since systems that are prone to pitting are also prone to crevice corrosion, and crevice corrosion may initiate prior to pitting. [Eden]

Coatings and Linings: In situations with coatings and linings, the high impedance of the coatings combined with the necessity of obtaining a representative sample area typically require electrode areas in excess of 30 cm^2. The thicker the coating, the larger the electrode surface area needs to be. One of two arrangements is typically used. In the first, an isolated WE-WE-RE array is used with the two nominally identical specimens coated as the WEs. In the second, the sensor array is beneath the coating so that it is sensitive to permeation of the coating by the corrosive environment. [Eden]

Low Conductivity: In low conductivity environments, it is important to minimize the effects of solution conductivity. This is done by reducing the spacing between electrodes and increasing the surface areas of electrodes. Care should be taken in the insulating material, especially with respect to their wetting, since it is necessary to maintain electrical isolation between the electrodes. [Eden]

Condensing Environments: In condensing environments, where the potential for corrosive condensates exist (such as acid dewpoint in flue gas), flush mounted, laminar probes have been successfully employed to detect the onset of the dewpoint condition. [Cox]

A search for EN sensor patents obtained the following five results (each followed by a very brief summary):

S. A. Reid and D. A. Eden, 2001, US 6,264,824 B1, "Assessment of Corrosion," A method and apparatus for using EN to asses corrosion, preferably with skewness and kurtosis analysis using neural nets.

G. A. Martincheck and M. R. Yaffe, 2000, US 6,015,484, "Detection of Pitting Corrosion," A modification of the WE-WE-RE sensor arrangement where one WE is anodically biased with respect to the other WE. It claims to allow the prediction of the on-set of pitting prior to it occurring.

D. H. Pope, Y. J. Lin, E. J. St. Martin and J. R. Frank, 1999, US 5,888,374, "In-Situ Process for the Monitoring of Localized Pitting Corrosion," It uses a WE-WE-RE sensor arrangement for monitoring pitting corrosion in metal pipes and storage vessels using EN. Analysis transforms the EN into power spectral density (PSD) data using fast Fourier transform.

Y. Tanaka, S. Iwahashi, M. Miyazawa and K. Morita, 1996, 10019826 JP, "Apparatus for Measuring Corrosion of Metallic Material," It describes a general process for EN measurement and data analysis.

D. H. Roarty and D. A. Eden, 1994, EP 0 607 057 A1, "Electrochemical Monitoring of Vessel Penetrations," It is a through-vessel wall sensor array for electrochemical measurements, including EN, for monitoring SCC.

Data Acquisition System Requirements

The data acquisition system (DAS) should resolve signals in the μV range and be able to accommodate voltage offsets that result from the use of reference electrodes. At least a 16 bit analogue-to-digital converter is required. The DAS should allow simultaneous measurement of current and voltage. Very high data acquisition rates are not needed; indeed, at high rates the amplitude of the instrumental noise can approach EN levels. Therefore a minimum logging period of one second is satisfactory. [Eden]

The importance of DAS equipment has been shown by laboratory round robin testing of EN standards. In these tests, reproducible information was obtained only when there was sufficient

standardization in techniques. The round robin testing resulted in recommending: 1) the use of equipment able to test in the frequency range 0.01 Hz to 10 Hz, 2) the sampling rate must be twice the highest frequency of the phenomenon being studied (however, for modern equipment this is no longer an issue), 3) the complete measuring apparatus must exhibit small amounts of background noise of less than ±100 µV or ±10 nA, 4) continuous measurements of at least 10 minutes in duration, 5) better standardization in methods used in data evaluation, and 6) greater attention to be paid to the interactions between the ways that EN measurements are made (such as sampling rate, filtering, data processing, and background noise). [Goellner]

Current Theory in EN Analysis

Analysis of EN signals can be classified into a variety of methods: visual, sequence-independent, and sequence-independent. Visual examination of the time record trace can give indications as to the type of corrosion processes that are occurring. Statistical analysis of the noise signals indicates that the noise data generated during general corrosion has a relatively normal Gaussian distribution and will exhibit few rapid transients. In contrast, localized corrosion processes such as pitting and SCC have transients in the time record trace, and have characteristics that help distinguish between them. Localized corrosion leads to deviation from a normal distribution (Poisson distribution) as may be determined from the skewness and kurtosis of the signals. Other means of identifying localized corrosion involve the use of the Localization Index (L.I.) derived from the ratio of the standard deviation of the current to the root mean square (rms) of the current. If techniques such as LPR and HDA are also used, then independent derivation of the general corrosion current (I_{corr}) is possible. The ratio of the current noise (standard deviation of the current) to the general corrosion current may then be used as an improved indicator of localized corrosion activity.

Statistical analyses that treat the data as a set of values, without regard as to the order that the data was collected are termed "sequence-independent" and are shown below. [Eden, Cottis]

Statistic	Formula	Comment
Mean, $\hat{y}$	$$\hat{y} = \frac{1}{N} \sum_{i=1}^{N} y_i$$	Mean current indicates galvanic coupling current. Mean potential verses a reference electrode indicates reaction type. Estimated error of $\sigma(1/N)^{1/2}$.
Variance, m_2	$$m_2 = \frac{1}{N} \sum_{i=1}^{N} (y_i - \hat{y})^2$$	Indicates the power in the noise signal.
Third moment, m_3	$$m_3 = \frac{1}{N} \sum_{i=1}^{N} (y_i - \hat{y})^3$$	A measure of the asymmetry of the data around the mean.
Skewness, g_1	$$g_1 = \frac{m_3}{m_2^{3/2}}$$	A normal distribution will have a g_1 of zero. A distribution with more of a tail in the positive direction will have a positive g_1, and one with more of a tail in the negative direction will have a negative g_1. Estimated error of $(6/N)^{1/2}$.

Fourth moment, m_4	$$m_4 = \frac{1}{N}\sum_{i=1}^{N}\left(y_i - \hat{y}\right)$$	
Normalized Kurtosis, g_2	$$g_2 = \frac{m_4}{m_2^2} - 3$$	A normal distribution will have a g_2 of zero. A positive g_2 reflects a more peaked distribution and a negative g_2 a less peaked distribution. Estimated error of $(24/N)^{1/2}$. A $g_2 \geq 2$ is typical for localized corrosion
Standard deviation, σ	$$\sigma = \sqrt{m_2}$$	A measure of the spread of the data that relates to the broadband ac component of the signal. Estimated error of $\sigma(2/N)^{1/2}$.
Coefficient of variance, C of V	$$C\ of\ V = \frac{\sigma}{\hat{y}}$$	Measures the distribution of the data around the mean. Similar meaning as LI.
Root mean square, rms	$$rms = \sqrt{\frac{1}{N}\sum_{i=1}^{N}y_i^2}$$	
Localization Index, LI (of current, i)	$$LI = \frac{\sigma_i}{i_{rms}}$$	Measures the distribution of the current around the rms. LI ranges in value from 0 to 1. When LI is close to 1, localized corrosion is likely. A LI on the order of 0.001 indicates general corrosion.

Statistical analyses that take into account the order in which the data were collected are termed "sequence-dependent". These methods retain more of the information in the data than the sequence-independent methods, but are much more difficult analyses to perform in terms of computational requirements and in error propagation. A description of each sequence-dependent method follows:

Autocorrelation Function: The autocorrelation function (ACF) is the expected value of the product of the time series at one time and at certain later times. For example, with Gaussian white noise (typical of general corrosion), where each reading is independent from each other and from a normal distribution, the ACF is zero. [Cottis]

Power Spectra: The estimate of the power present at various frequencies results in plots of the power spectra density (PSD) as a function of frequency. The units of PSD for potential and current are V^2/Hz and A^2/Hz, respectively. There are two primary methods used in the corrosion field: Fourier transform and maximum entropy. [Cottis]

The Fourier transform method computes the combination of sine waves needed to obtain the observed signal. The fast Fourier transform (FFT) algorithm is usually used, where the PSD is determined as the amplitude squared of the sine waves, divided by the frequency separation. To minimize artificial noise in the spectrum, trend removal and windowing are applied to the time record prior to computing the spectrum and the results from several spectra are then averaged. [Cottis]

The maximum entropy method (MEM) fits functions to the ACF and then uses a Fourier transform to obtain the PSD. This procedure acts to smooth the power spectrum. The MEM computes a number of coefficients that describe the ACF; the number of coefficients is known as the order of the MEM and

is an adjustable parameter. A low-order MEM results in a very smooth PSD, while a high-order MEM results in a very noisy PSD (much like the unsmoothed FFT). [Cottis]

Higher-Order Spectra: Similar to the higher-order statistics in the sequence-independent methods, higher-order sequence-dependent methods are available. However, the results are even more difficult to interpret, and it is not yet known how to extract the relevant information contained within the spectra. [Cottis]

Other Methods: There are several other methods being examined for use in EN analysis. *Wavelet* methods use a variant of the Fourier analysis where transients with a finite duration ("wavelets") are used instead of sine waves to obtain the observed signal. The use of *chaos theory* has been attempted by several researchers, but the results are still theoretical. [Cottis]

Thus far, only parameters obtained from either the potential or current time record have been considered. However, when potential and current are measured simultaneously, combinations between the two can be used. Examples include the noise resistance, R_n, which is the ratio of the potential to current standard deviations, and the noise impedance, which is the square-root of the ratio of the potential to current PSDs (calculated at specific frequencies). [Cottis]

The above methods are used to analyze the EN from a system. Attention must also be paid to noise resulting from non-corrosion processes. These include *thermal noise* from the thermally-activated charge carrier motion (which generates a potential PSD equal to 4kTR, where k is the Boltzmann constant, T is the absolute temperature, and R is resistance), *shot noise* from the quantum nature of charge (which generates a current PSD equal to 2qI, where q is the charge on the charge carrier and I is the mean current), and *flicker noise* where the PSD is proportional to the reciprocal of the frequency. Of more interest are EN sources from corrosion processes: [Cottis]

Pitting: Metastable pit nucleation, growth, and termination all produce current transients with a duration of a few seconds. Metastable pits frequently exhibit a slow current rise followed by a sharp current fall for stainless steel and a moderate current rise followed by a slower fall for carbon steel and aluminum alloys. At high frequencies, the log (potential PSD) verses log (frequency) plot will have a characteristic "roll-off" slope, which depends on the double layer capacitance. For aluminum at low frequencies, it has been found that shot noise analysis can yield an estimate of pit dimensions. [Cottis]

Particle Impact: Particle or bubble impacts on a surface may increase or decrease the corrosion rate by removal of passive films or by shielding of the surface, respectively. The EN produced can be analyzed with essentially the same methods as in pitting. [Cottis]

Activation-Controlled Dissolution: Activation-controlled dissolution of the metal typically occurs at atomic ledges and kinks on its surface. This gives rise to a shot noise process with a charge of between 100 to 300,000 electrons (depending upon the dislocation density). This results in noise in the current PSD at about $2neI_{corr}A^2/Hz$, where n is the number of electrons involved in each burst of charge and e is the charge of an electron. [Cottis]

Crevice Corrosion: Crevice corrosion is similar to pitting and will give similar characteristics. However the onset of crevice corrosion often has a large drop in potential as the active crevice polarizes the passive non-crevice surface. If this drop is permanent, then visual inspection of the potential-time record indicates crevice corrosion. If the passive surface is unable to sustain the current

needed in the active crevice, then oscillations in the potential-time record can occur as the crevice activates and passivates. [Cottis]

Stress Corrosion Cracking (SCC): Stress corrosion cracks can grow either in a continuous or discontinuous manner, and have quite different EN signatures. Discontinuous crack growth produces transient currents as new surface is exposed. It can be analyzed in a similar manner as pitting. Continuous crack growth should give lower EN levels. In either case, as the crack propagates deeper into the surface, greater shielding of EN may limit detection. [Cottis]

Corrosion Fatigue: Corrosion fatigue is expected to generate EN that is dominated by the applied load frequency. [Cottis]

The Reid and Eden patent gives the following table to identify the corrosion mechanism with skewness and Kurtosis analyses of the potential and current signals.

Mechanism	Potential		Current	
	Skewness	Kurtosis	Skewness	Kurtosis
General	$< \pm 1$	< 3	$< \pm 1$	< 3
Pitting	< -2	$\gg 3$	$> \pm 2$	$\gg 3$
Transgranular SCC	+4	20	-4	20
Intergranular SCC #1	-6.6	18 to 114	1.5 o 3.2	6.4 to 15.6
Intergranular SCC #2	-2 to -6	5 to 45	3 to 6	10 to 60

EN Field Experience

There is experience in using EN sensors in several field applications. A selection of these efforts are given below:

Hydrocarbon Pipelines (offshore pipeline): EN sensors with data analyses of noise resistance, R_n, and the product of the corrosion current and the localization index, $I_{corr} \cdot LI$, were used along with linear polarization to monitor corrosion in an inhibited pipeline. The low corrosion rates from the EN and linear polarization methods were in close agreement. [Ryder]

Sour Gas Processing Plant (Alberta, Canada): EN sensors were installed in several process units for two years. EN sensors identified the onset of pitting in a sulfinol contactor tower where the solvent was contaminated with air.   [Teevens]

Sour Gas Field (Alberta, Canada): EN monitoring was used in field evaluation of inhibitor effectiveness and minimum effective levels in a pitting prone environment with an acid gas content of 17% H_2S and 3.5% CO_2. Corrosion inhibitors account for up to 25% of operating costs. [Barr]

Nuclear Waste Storage Tanks (Hanford, WA): EN sensors were installed in double shell underground waste tanks in both immersion and vapor-phase placements. The sensors were employed in an attempt at early detection of localized corrosion of the tanks. Early results were plagued with extraneous noise from electrostatic interference using long analog data cable and cross-talk problems. After solving these issues all channels have shown approximately 1 mpy corrosion in liquid immersion and 0 mpy in vapor-phase placement. [Edgemon and Edgemon]

Water Treatment Plant (Lost Hills, CA): Five-element probes that simultaneously performed EN and linear polarization measurements were installed at locations with historical corrosion rates of 0.5 to 450 mpy. Problems included probe fouling, data collection volume, and awkward data manipulation routines. However, the monitoring did provide useful plant operation data. [Bell]

Freshwater Cooled Heat Exchangers (Ontario, Canada): EN sensors were designed for field testing of microbiologically influenced corrosion (MIC). A reproducible electrochemical response was observed that may be a MIC EN signature. [Brennenstuhl]

EN Laboratory Studies in Gas Pipeline-Type Environments

Three laboratory studies were identified as being in environments similar to gas pipeline conditions:

A 7.5 cm diameter, 10 m long multiphase oil/water/gas flow system was examined with EN measurements with a variety of 2-phase and 3 phase slug flows. EN analysis using FFT identified uniform corrosion with full pipe flow (roll-off slopes greater than –40 dB/decade). In slug flow has characteristic frequency transients indicating pitting (roll-off slopes less than –20 dB/decade). Slugging frequencies were also identified. [Deva]

Corrosion measurements in flowing oil-electrolyte (water with 3% NaCl) mixtures was examined with EN sensors. Three distinct noise patterns were observed. The first was a low noise baseline for oil without electrolyte, for oil with small additions of electrolyte, and for oil with electrolytes but with under low-flow conditions. The second was a low noise signal but with periodic bursts of current for moderate electrolyte additions under high-flow conditions. The third pattern was a high amplitude signal for high electrolyte additions under high-flow conditions. The use of neural networks was explored in an effort to automatically analyze for the three conditions. [Malo and Malo]

The CO_2 corrosion of a carbon steel pipe was examined using EN measurements. The EN parameters of noise resistance, R_n, and noise impedance were related to corrosion activity and flow turbulence. [Krebs]

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