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*This Technical Committee Report has been prepared
by NACE International Task Group T-1D-34[*] on
Laboratory Corrosion Inhibitor Test Parameters*

# Laboratory Test Methods for Evaluating Oilfield Corrosion Inhibitors

### FOREWORD

This NACE technical committee report summarizes the approaches and experiences of Task Group T-1D-34 on oilfield corrosion inhibitor testing and evaluation. The objectives of T-1D-34 for this report were defined and agreed on as follows:

(1) Establish the pertinent parameters to be observed and/or controlled during laboratory and field evaluation of

corrosion inhibitors for the purpose of establishing a common baseline for the comparison of the results from such evaluations; and

(2) Conduct a round-robin test in order to establish the reproducibility of corrosion inhibitor ranking as indicated by two different tests conducted in different laboratories.

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This report is based on

(1) feedback obtained from a survey conducted by the task group and extensive discussion among the task group membership, and

(2) results of a round-robin test among participating laboratories specifying the use of the linear polarization resistance (LPR)—also called *kettle*—and wheel tests for the performance ranking of three corrosion inhibitors under identical test conditions.

The first part of this report presents a discussion of the pertinent parameters that control corrosion inhibitor performance in order to provide a basic understanding of corrosion inhibitor performance under different conditions, and to provide a common basis for corrosion inhibitor evaluations. Specific test parameters are generally dictated by the field-specific conditions toward which the selection of corrosion inhibitors is oriented. Another focus of the report is on the LPR and wheel tests, which are the most commonly used test procedures for the selection of oilfield corrosion inhibitors.

The second part of the report presents the results of the round-robin test program based on the two test methods. Details of the test results are summarized in Appendix A.

Definitions of terms are given in Appendix B.

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## SYSTEMATIC ANALYSIS OF FIELD CONDITIONS (SYSTEMS ANALYSIS)

Failure records, corrosion history, nondestructive inspection of equipment, failure analysis, iron counts, corrosion coupons, corrosion test nipples, and corrosion rates obtained by instrumentation are some of the tools that have been at the disposal of the engineer to determine the type and severity of corrosion experienced in the oil field.

The corrosion history of the oilfield system in general has provided valuable data in conjunction with the pertinent production parameters when the latter were available. For instance, when the failures have occurred in high-velocity areas, corrosion inhibitor screening tests were designed in order to simulate the flow regime. The prevailing shear stress (and in some instances mass transfer rates) might have been used as the pertinent modeling parameters.

An "up-to-date" flow diagram of the well and/or production system, including pipe diameters, lengths of lines, depth and completion of well, existing pig traps, coupon or probe locations, and materials of construction,

has provided essential information. For example, using test specimens consistent with the field construction material has provided comparisons between laboratory test data and effects experienced in the field.

A comprehensive analysis of the well or production system has also sometimes provided invaluable information. The following data have been used by this task group's members to perform such analyses (not ranked in order of importance):

(a)     gas, oil, and water production rates;
(b)     system or well pressure and temperature profile;
(c)     produced water and/or brine analyses;
(d)     gas composition (carbon dioxide [$CO_2$], hydrogen sulfide [$H_2S$]);
(e)     the system pH, which is a function of the acid gas partial pressures and the alkalinity of the water;
(f)     flow dynamics and phase behavior of production system or well aimed at the determination of the water and hydrocarbon dewpoints;
(g)     velocities/wall shear stresses of production system or well;
(h)     type of corrosion attack on walls and/or test specimens;
(i)     measured field corrosion rates;
(j)     dispersed or suspended solid concentrations and types;
(k)     lift method or injection/disposal method used;

(l)     reservoir information (bottomhole temperature, bottomhole pressure, formation type, porosity, etc.);
(m)     secondary or tertiary recovery method (polymer, waterflood, fireflood, $CO_2$ flood, etc.);
(n)     fluid levels of wells and vessels; and
(o)     completion and simulation work performed or scheduled.

Field conditions have often limited the use of corrosion inhibitor treatment techniques. The treatment method to be used for specific systems has, therefore, been another consideration in laboratory test design. For instance, continuous injection has not always been feasible in a certain location; therefore, continuous laboratory test procedures would have provided dubious corrosion inhibitor screening results for such locations.

Flow conditions have sometimes affected the corrosion inhibitor performance.[1] In order to simulate flow conditions, shear stress has commonly been used as the modeling parameter to correlate laboratory with field conditions. Shear stress can vary considerably with the flow characteristics. The three types of flow characteristics in single-phase flow identified by this task group are laminar, transition, and turbulent flow. The actual flow characteristic is determined by the Reynolds number (Re). In addition to flow characteristics, flow regimes have been considered and replicated, especially for multiphase flow systems. Typical flow regimes are bubble flow, slug flow, stratified flow, annular flow, and mist flow.[2,3]

## LABORATORY TEST DESIGN

Typically, laboratory evaluations of corrosion inhibitors have been designed to provide test results that were reliable, reproducible, and comparable with field conditions.[4] Tests that have given the closest correlation with the field results have integrated the system flow dynamics, fluid compositions, conditions, and corrosion inhibitor treatment limitations.

Specific laboratory tests to determine the solubility, dispersibility, and partitioning characteristics of the corrosion inhibitor(s) under consideration have generally resulted in more effective product selection. Environmental factors and corrosion inhibitor toxicity have become more important in the corrosion inhibitor evaluation process.

### General Considerations Regarding the Round-Robin Test Methods and Equipment

The following test methods are described because they were used in the round-robin tests performed by this task group. Detailed test procedures and the results are given in Appendix A. Other test methods, including, but not limited to, flow loops, jet impingement, and rotating specimens, have found application.

Nine companies participated in the round-robin testing. Two companies were end users and seven were suppliers. All the companies generated similar rankings

of inhibitor effectiveness with only minor differences for both tests.

*Linear Polarization Resistance (LPR) Test Method*[5]

The LPR test method measured the corrosion rate in real time electrochemically. Metal electrodes, typically well-characterized carbon steel, were used to determine the corrosion rate of the corrosive fluid. The reference and auxiliary electrodes were sometimes made of the same material and sometimes from different materials. The test vessel contained the electrodes, sparge and gas outlet tubes, thermometer or thermocouple, and stirrer. The test vessel was heated by a heating mantle, constant-temperature bath, hot plate, or lamp. The fluids were stirred by a stir bar or overhead stirrer.

The test fluid was a conductive liquid such as brine or a brine/oil mixture. To simulate field conditions and maintain the corrosiveness of the fluid, the test vessel was sparged with $CO_2$, $H_2S$, or gas mixtures.

The fluids were added to the corrosion test vessels. These vessels were usually glass flasks equipped so that the fluids could be agitated and heated. The fluids were heated to the test temperature while being sparged with the corrosive gas(es). The fluids were under continuous agitation.

Field conditions were considered in the deter-

mination of the degree of agitation. Mild agitation meant the oil and brine phases remained as two distinct layers, while a high agitation meant that the hydrocarbon phase was dispersed into the aqueous phase. Probe fouling in high-agitation tests has caused erroneous readings.

The electrodes were placed in the aqueous phase and allowed to equilibrate. A blank corrosion rate was established. A suitable amount of corrosion inhibitor was added and the corrosion rate determined. This step was repeated for increasing inhibitor dosages.

Linear polarization was performed by (1) controlling the potential and measuring the current or (2) controlling the current and measuring the potential. The maximum potential excursion was typically 25 mV. LPR data were analyzed by fitting multiple data points or a simple analysis using a single data point.

When the change in potential was plotted against the current change, a straight line was obtained. The slope of this line is the polarization resistance and is inversely proportional to the corrosion rate.

The relationship between the corrosion rate (or the corrosion current, $I_{corr}$) and the polarization resistance, $R_p$, is given in Equation (1).

$$I_{corr} = \frac{I}{R_p} \times \left( \frac{\beta_c \times \beta_a}{2.3 \times (\beta_c + \beta_a)} \right) = \frac{\Delta I_{pol}}{\Delta V_{pol}} \times \beta_{factor} \qquad (1)$$

The proportionality factor between the corrosion current and the inverse polarization resistance is the *beta factor*. It is determined on the basis of the anodic and cathodic Tafel slopes ($\vartheta_a$ and $\vartheta_c$). Observations that indicate that the beta factor is different for uninhibited and inhibited systems have been made. Similarly, the beta factor can change during the test. In low-conductivity solutions (e.g., highly dilute brines or hydrocarbon liquids), an additional correction is often made to account for the IR drop which becomes part of the polarization potential, even at very small values of 5 and 10 mV.

*Wheel Test Method*[6]

The wheel test method was based on the mass (weight) loss of a metal test specimen during a period of exposure to corrosive liquids. The corrosive liquids were usually produced fluids, although synthetic brines and refined oils were sometimes used. These fluids and the test vessels were sparged to exclude oxygen contamination and to saturate the liquids with corrosive gases to simulate field conditions. Test duration ranged from 18 to 72 hours.

The carbon steel test specimens were typically UNS[(1)] G10180. Shim stock, drill rod, or machined test specimens were used. A wheel box (a closed box containing a rotating wheel) was used for these tests. The

test vessels (usually beverage bottles) were attached to the wheel and were in constant circular motion during the test at approximately 30 rpm. The wheel box was capable of maintaining a constant temperature over a wide temperature range.

The test specimens were prepared, cleaned, and weighed. The desired amount of corrosion inhibitor was added to the test vessels. The corrosive liquids were then measured into the test vessel. The test specimen was added and the vessel sealed and attached to the wheel. The test vessels were rotated at temperature for the desired test period. Unless the field temperature was outside the operating temperature of the wheel box and test vessel, the temperature used was usually the field temperature.

At the end of the test period, the test vessels were cooled and the test specimens removed, cleaned, and weighed. Inhibitor effectiveness values were obtained by comparing the inhibited weight loss to the blank weight loss. Visual inspection of test specimens provided important information on the type of corrosion attack. Some laboratories corrected the total weight loss by the weight loss attributable to the cleaning procedure.

**Generic Information About Testing Equipment and Procedures**

*Metal Test Specimens*

The size of the test specimens has generally been small in relation to the volume of the test fluid. This has minimized significant changes in the chemistry or maintained an adequate amount of corrosive material in the test fluid during the test. A number of guidelines exist regarding the size and shape of test specimens.[7,8]

A consistent surface finish on test specimens typically has minimized variation in results. A number of suitable techniques have been reported.[7,8,9,10]

The test specimens have sometimes been equipped to reproduce or cause crevice corrosion.

Several cleaning techniques, such as mechanical, chemical, electrolytic, or a combination of these techniques, have been used and reported.[7,8,9,10]

*Collection of Fluids*

Field fluids have normally been collected in a manner that excluded oxygen contamination.

Fluid samples taken from a contaminated or treated source have introduced numerous errors into the testing process.

Corrosion-resistant containers have typically been used for storage and shipment of fluids.

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[(1)] Metals and Alloys in the Unified Numbering System (latest revision), a joint publication of the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers (SAE), 400 Commonwealth Drive, Warrendale, PA 15096.

*Test Fluid Reconstitution*

The test fluid chemistry has generally been pre-defined and reproducible.

The reconstitution of the test fluids has been based on the detailed brine analysis, which included alkalinity and concentration of all or most anions and cations. It was generally recognized that the pH of the test fluid depended on the alkalinity and the partial pressure of the acid gases.

When the corrosive gas(es) pressure has been higher than atmospheric pressure, purging at atmospheric pressure has sometimes been found inadequate to generate the field environment. Pressure vessels have often been used to perform the testing. Simple techniques for purging gas(es) through the test fluids are illustrated in ASTM[(2)] Standard G 31[7] and NACE Standard TM0169.[8] Glass tubing or fritted glass diffusers were immersed in the water to purge the gas(es) through the fluids.

*Flow Conditions*

Selection of laboratory tests has depended on analysis of the system's flow dynamics.

Wheel test and LPR test methods have been used to reproduce static, laminar, or other flow conditions.

A system in which all phases have contacted essentially all metal surfaces has generally been used. The wheel test and LPR test methods have been used for moderately turbulent conditions.

Wheel tests and LPR tests generally have not achieved high-shear-stress conditions. Flow loops, impingement tests, and rotating specimen tests have commonly been used to simulate high-shear conditions. Solids have sometimes played a major role in erosion-corrosion.

*Gas-to-Liquid Ratio*

The ratio of gas to liquid in the test vessel has generally been high so that the corrosion process has not significantly decreased the amount of corrosive gas(es) except perhaps in the wheel test, where the gas space has traditionally been small.

*Corrosion Product Accumulation*

Corrosion product accumulation during the test has been observed in the test fluids and on the test specimens. Field observations have indicated that iron carbonate, sulfate, sulfide, and oxide layers may prove difficult to inhibit. Corrosion inhibitor evaluations have, therefore, often been carried out on precorroded test specimens.

Alternatively, it has also been observed that corrosion inhibitors require a small corrosion product layer to become active. This is particularly true for sulfide and oxide scales. The build-up of soluble corrosion products (bicarbonate) has often resulted in a continuous change of the pH during the test. Because pH can affect both the

instantaneous corrosion rate as well as the effectiveness of the corrosion inhibitor, the practice of maintaining a constant pH during the test (LPR test) has been adopted by some laboratories.

**Secondary Properties of Corrosion Inhibitors**

Some of the secondary properties of corrosion inhibitors that have been tested separately from the performance tests in order to avoid operational problems are:

(a) foaming tendency;
(b) emulsion tendency;
(c) thermal stability;
(d) potential toxicity;
(e) susceptibility to solvent stripping;
(f) viscosity at use temperatures; and
(g) compatibility with other products in the system.

Foaming tendency testing has ranged from the simple to the elaborate. A simple test that has been used successfully involved the bubbling of gas through a column of fluid (such as produced fluids, glycol units, monoethanolamine [MEA] units, etc.) treated with the selected corrosion inhibitor dosage. A more elaborate test has involved the use of a pressurized bottle (e.g., a seltzer bottle) or flow loop with an inline mixer where the gas(es) are introduced. Foam quality, foam height, and foam stability (half-life) have generally been measured.

Emulsion characteristics have commonly been tested by hand-agitation bottle tests, seltzer bottle tests, and high-shear blender tests comparing treated fluids with untreated fluids. The corrosion inhibitor concentrations have ranged from actual treating rates to high arbitrary rates. The fluids have normally been observed for the degree of water and oil separation at timed intervals and the resolution of pads or interfaces with time and heat. The quality of the separated (breakout) water has sometimes been of additional concern and testing has been performed to address this issue.

The thermal stability of a corrosion inhibitor has commonly been tested at use concentrations and/or near the extremes of temperature, pressure, and duration expected in the system. Visual inspection of the corrosion inhibitor, infrared spectra, and performance data have been compared before and after exposure. The corrosion inhibitor sample has typically been inspected for changes in chemical composition, performance changes, decomposition, polymerization, etc.

The ease of stripping of the solvent from a corrosion inhibitor when applied in gas lines has been evaluated by determining the mobility of the corrosion inhibitor after solvent removal. Methods commonly used have involved flashing solvent off in high-temperature ovens and simply bubbling gas(es) through the neat or diluted product to determine fluid loss. Loss of solvent has sometimes led to solubility changes and limited the corrosion inhibitor

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effectiveness due to changes in its partitioning ability.

Viscosity profiles of the corrosion inhibitor at different temperatures have helped determine packaging and have been used to predict whether the corrosion inhibitor was deliverable in a specific environment.

Compatibility of corrosion inhibitors with system fluids and other chemicals used in a system has commonly been measured by visual observation and performance testing of the corrosion inhibitor in the presence of all other chemicals being used or contemplated for use.

Compatibility of corrosion inhibitor(s) with the materials of construction in the system has often been overlooked and has caused operational problems. Materials typically compatible with the corrosion inhibitor in a successful operation have included various metals and nonmetallic materials that make up the system.

## DATA PRESENTATION

### Qualitative

The test vessels and test specimens have typically been examined during and following the test for any characteristics that might provide information concerning the suitability of the corrosion inhibitor in those fluids used for the test.

Discoloration of the test fluids has usually been noted. For example, a red coloration typically indicated that oxygen had been introduced into the test. The presence of solids or deposits on the test specimen or in the test vessel has usually indicated that the corrosion inhibitor was incompatible with the test fluids. Blistering observed on the test specimen has been noted.

If the testing was designed and performed correctly, a reasonable correlation with field data has been seen. It has often been difficult to gauge the actual corrosion inhibitor concentration needed in the field based on laboratory tests.

### Quantitative

The corrosiveness of field conditions has generally been used as a guide to the order of magnitude of corrosion rates expected in laboratory testing. Agreement of laboratory test corrosion rates within 0.25 mm/y (10 mpy) has been considered adequate. A variation in the corrosion rates of 0.025 mm/y (1 mpy) or approximately 1 mg weight loss (based on test specimen size) in laboratory testing has at times been used to assess the statistical significance of the results. This latter criterion might have been larger for blank corrosion rates.

The fluid composition at the end of the test has provided information about the performance of the corrosion inhibitor during the test. For instance, an increase in the pH of the test fluid during the test has indicated that the corrosive gases have been consumed or bicarbonate has been accumulated, indicating that the corrosion process might not have been reduced by the addition of the corrosion inhibitor.

The size, depth, shape, and amount of pitting on the test specimen has typically been noted and recorded. A microscopic evaluation has often been performed to make this assessment.

The LPR electrodes have normally been weighed before and after the LPR test. A weight-loss corrosion rate was then calculated for comparison with the LPR results.

Several well-known methods for evaluating the reproducibility of test methods have been used to monitor the tests. These have included standard deviation measurements and a number of computer programs designed to measure the reproducibility of test methods.

## TECHNIQUES TO ELIMINATE ERRORS IN TEST METHOD SELECTION

To avoid misleading test results, the test method selected has generally mimicked the targeted field application method as closely as possible. For example, film persistency methods have been used to measure the ability of a corrosion inhibitor to form a long-lasting film on the metal surface. These methods have ideally modeled the forces (such as shear stress) exerted on the pipe wall and corrosion inhibitor film while measuring the film life of the inhibitor.

Understanding the reasons for performing the testing has sometimes indicated which test methods to avoid. Some techniques that have been used to avoid errors in test method selection have included the following:

(a) Asking whether the test is a screening test or whether it is being done to choose corrosion inhibitor treating concentrations and treatment intervals.

(b) Determining which parameters the tester believes are the most important in selecting a corrosion inhibitor and discussing them with the laboratory personnel overseeing the testing.

(c) Communicating as much information to laboratory personnel as possible along with exactly what is expected from the data. One test has not always covered all the parameters and secondary characteristics needed.

(d) Comparing additional test results with the primary results. For example, the most effective corrosion inhibitor has sometimes caused an emulsion problem or oil carryover into the water.

**Test Fluid Collection and Transportation**

The test fluids have generally been collected and transported in a way to minimize chemical changes. In some instances, the test fluids have been collected under pressure or in plastic, glass, or lined metal containers. **Field safety regulations have been observed.** Containers have been capable of withstanding pressure build-up while excluding oxygen contamination.

Transportation of test fluids has been in accordance with all applicable local, state, federal, or international regulations. Some test fluids have been best preserved by packing and shipping them under cool conditions. The laboratory personnel have typically been able to advise about shipping conditions for the test fluids and what conditions they were set up to handle.

**Preparation of Field or Synthetic Fluids for Testing**

There have been questions regarding what to do when field fluids were contaminated by corrosion inhibitors. These questions have included whether to remove the polar compounds contaminating the fluid, and whether to prepare and use synthetic fluids.

Corrosion inhibitors have been removed by filtering the fluids through diatomaceous earth and/or activated charcoal.

Synthetic test fluids have been prepared and adjusted to provide a predefined chemistry based on the field fluids. The chemistry has been adjusted to proper pH, alkalinity content, and organic acid content.

When synthetic hydrocarbons were used, the hydrocarbon's viscosity, wetting ability, asphaltene content, wax content, and the inhibitor's solubility and dispersibility in the hydrocarbons have been closely matched. Kerosene, diesel, and mineral oils have commonly been used as synthetic hydrocarbon fluids with mixed success.

The gas sources have sometimes reintroduced corrosive gases. Oxygen contamination in the gas has caused reproducibility problems for many researchers.

**Selection of Test Method**

Not all conditions can be simulated in the laboratory; therefore, testers have attempted to select the most important parameters or run multiple tests to ensure all test criteria have been met.

**Preparation and Conditioning of Test Specimens**

Preparation of the test specimen has ideally produced a surface finish similar to that found in the field.

Specifying a specific test specimen surface finish (i.e., sand blasted, sanded or polished, acid activated, glass bead blasted, etc.) has prevented or caused damage to the metal test specimens. Stamping versus cutting the metal stock has induced edge attack.

**Corrosion Inhibitor Application—Batch Treatment or Continuous Injection**

Questions that have commonly been asked when considering whether to use batch or continuous corrosion inhibitor application have included:

(a)     What field application will be used?
(b)     Will the corrosion inhibitor be applied neat or diluted?
(c)     Is the corrosion inhibitor soluble in diluent over all concentration ranges expected?
(d)     Is the corrosion inhibitor/diluent package soluble in fluids?
(e)     Will the corrosion inhibitor and/or diluent cause adverse effects in the fluid? For example, if the corrosion inhibitor is alcohol-based, will it cause asphaltene precipitation from oil and cause secondary problems in production operations?

**Reproducibility**

When testing has not provided reproducible numbers and/or rankings of corrosion inhibitors, the test method or techniques have been considered suspect. Statistical analysis has often been used to set limits.

## EFFECTS OF CONTAMINANTS

Corrosion inhibition has generally been easier with clean systems. Contaminants can affect the corrosion inhibitor's performance. Laboratory tests normally have not considered the other system conditions, such as the presence of microbiological contamination, scale, oxygen, $CO_2$, $H_2S$, and dissolved solids. Microbiological contamination has been known to:

(a)     produce acids;
(b)     destroy protective coatings;
(c)     create corrosion cells;
(d)     produce $H_2S$;
(e)     oxidize metal;
(f)     depolarize cathodic sites; and
(g)     foul equipment.

Scale has sometimes created a porous or nonporous layer that adhered to the surface of the equipment or was removed by the produced fluids. When a porous layer has adhered to the surface, serious localized attack has sometimes resulted. This scale in most cases has interfered with the formation of a protective surface film.

Oxygen in very low concentrations has sometimes caused serious corrosion problems, such as severe pitting. Oxygen can oxidize iron to the ferrous ion or it can oxidize the ferrous ion to ferric ion, which is a species more corrosive to the environment. Oxygen can depolarize the cathodic reaction, thereby accelerating the corrosion rate.

$CO_2$ corrosion rates are dependent on temperature, pressure, flow rate (velocity), pH of the produced water, and partial pressure of the $CO_2$.

$H_2S$ corrosion has resulted in the formation of cor-

rosion products, mainly $Fe_xS_y$. In some cases the partial pressure of $H_2S$ and the pH of the produced water have determined the type of scale formed on the surface. These scales were said to be cathodic in nature compared with the underlying steel. This condition has

created a galvanic couple in the presence of water, causing either cracks or pitting attack on the exposed areas of steel. These corrosion processes have started slowly but accelerated rapidly.

## ANALYSIS AND INTERPRETATION OF RESULTS

Written results have normally included:

(a)    method summary;
(b)    date;
(c)    exposure periods;
(d)    pressure;
(e)    temperature;
(f)    test media composition—both liquid and gaseous;
(g)    surface-to-liquid ratio;
(h)    liquid volume to gas phase ratio;
(i)    type of test specimen used, including surface finish of test specimen;
(j)    dimensions of test specimen;
(k)    corrosion inhibitor concentrations;
(l)    cleaning methods used; and

(m)    pertinent observations such as type of attack, estimated or measured area of attack, deposits observed on the test specimen, emulsion of fluids, etc.

### Analysis
Standard calculations performed on the test results have included both corrosion rates and percent protection afforded by the corrosion inhibitor (see NACE Publication 1D182[6]).

The corrosion inhibitor's performance has been ranked using all known data. All test results have been included, with any exclusion having an explanation.

### Interpretation
Secondary effects including emulsion tendency of fluids and localized corrosion attack have been considered in the final analysis and ranking of corrosion inhibitors.

## QUALITY ASSURANCE

The following examples depict areas in which quality assurance measurements have played a vital role in the evaluation process:

(a)    field data collection;
(b)    sampling techniques;
(c)    proper transportation of test fluids;
(d)    safety training of personnel;
(e)    calibration of the equipment used to measure parameters; and
(f)    calibration of laboratory equipment (balances, potentiostats, etc.).

### Training of Laboratory Personnel
Training laboratory personnel in laboratory safety, standard laboratory techniques, and specifically in the testing procedures to be performed has contributed to the quality of the final results. Capable laboratory personnel have reproduced results within reasonable experimental error.

## ENVIRONMENTAL FACTORS

Environmental effects have been and continue to be considerations in the corrosion inhibitor evaluation process. The specific local, state, and national government

guidelines applicable for the system under study and for the laboratory doing the testing have typically been reviewed and strictly adhered to.

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## APPENDIX A
## Round-Robin Testing Program

Nine companies participated in the round-robin testing program that formed the basis for this report. These included seven suppliers to the industry and two user companies. Two different test methods were selected for corrosion inhibitor evaluation. To ensure that variability in the results from the laboratories due to varying test procedures would be minimized, the task group, by consensus, established the following procedures for the tests. In addition, a common test environment was established. This was intended to eliminate all factors except corrosion inhibitor performance. Laboratories were encouraged to provide any comments concerning difficulties with the tests.

Two general instructions were given to the laboratories:

1.  Make efforts to monitor tests as closely as possible.

In general, the tests may be repeated as many times as needed. Don't be restricted by any one type or brand of instrument or equipment. Any instrument or equipment that can generate reliable data is acceptable.

2.  Compare the test results and rank the three corrosion inhibitors based on their reduction of corrosion rates as "most effective" (A), "effective" (B), and "least effective" (C).

These rankings should be based strictly on percent protection. Other factors such as gunking tendency, thermal stability, chemical behavior, etc., should not be considered in this evaluation. The rankings, of course, may be different for each of the test methods.

The following test methods were used by the laboratories in testing the corrosion inhibitors:

## Linear Polarization Resistance (LPR) Test Method

A.  Materials Used:

Test vessel — 1,000-mL glass flask

Electrodes — UNS G10180 or any other carbon steel

NACE brine — 85.07 g calcium chloride dihydrate
39.16 g magnesium chloride sixhydrate
2,025 g sodium chloride
19 L distilled water

Hydrocarbon — Kerosene without corrosion inhibitor

$CO_2$ — Certified pure (CP) or reagent grade anaerobic $CO_2$ (oxygen content less than 10 ppm or free of oxygen)

Polytetrafluoroethylene (PTFE)-coated stir bar — 3.18 x 0.953 cm (1.25 x 0.375 in.)

Thermocouple or constant-temperature bath

Inhibitor — As provided

B.  Typical Conditions:

Temperature — 49°C (120°F)
Gas — $CO_2$
Inhibitor levels — 15, 25, 50, 100, 200 ppm based on total volume
Test fluid — NACE brine/kerosene, 90/10 ratio

C.  Procedure

1.  Charge 900 mL NACE brine to each flask.

2.  Add 100 mL kerosene to each flask.

3.  Add stir bar to each flask.

4.  Place a rubber stopper with holes bored in it for thermocouple, LPR probe, and gas dispersion tube in top of flask (with thermocouple and gas dispersion tube in place).

5. Place each flask on a stir plate inside thermal control box used for test.

6. Hook up temperature readout and gas lines to each flask. Turn on stir plates at 500 rpm.

7. Turn on gas cylinder (controlled to 70 kPa [10 psig] supply pressure) and adjust to steady flow, watching bubbles in outlet tube and flow meter.

8. Turn on temperature readout, which is connected to heat sources used to heat each flask to 49°C (120°F).

9. Prepare electrodes:

(a) Remove electrodes from probe.
(b) Use stiff bristle brush and clean screws on end of probe.
(c) Clean inside of electrode of any rust (if new, of any blasting material).
(d) Put electrodes back on probe and check to see whether they are parallel to each other.

(e) Prepare surface by blasting with 100 mesh (25 µm, 0.010 in.) alumina or other material, taking care to leave a uniform surface.
(f) Do not touch electrodes after blasting.

10. Place probes in flasks that are at 49°C (120°F). Be careful not to scratch the electrode surface.

11. Connect the readout meter with probes.

12. Start time, taking readings (corrosion vs. time) at one-hour intervals.

13. After one-hour reading, inject proper concentration of corrosion inhibitor into kerosene phase of each flask using pipette or syringe.

14. Adjust ranges on readout meter if needed.

15. Run for 24 hours from time of injection of corrosion inhibitor.

16. Print and plot data collected.

## Wheel Test Method

A. Materials Used:

1. Test specimen material is to be shim stock, mild steel, 7.6 x 0.64 x 0.013 cm (3.0 x 0.25 x 0.0050 in.).

(a) Degrease shim stock with xylene.
(b) Sandblast with 100 mesh (25 µm, 0.010 in.) aluminum oxide sandblast material. Use 410 kPa (60 psig) air pressure.
(c) Reclean with solvent (xylene).
(d) Measure, weigh to nearest 0.1 mg, and record.

2. Test vessels — new, 200-mL beverage bottles.

3. NACE brine — 85.07 g calcium chloride dihydrate
39.16 g magnesium chloride sixhydrate
2,025 g sodium chloride
19 L distilled water

4. Hydrocarbon —Kerosene without corrosion inhibitor

B. Typical Conditions:

Temperature — 49°C (120°F)
Gas — Certified pure (CP) or reagent grade anaerobic $CO_2$ (oxygen content less than 10 ppm)
Inhibitor levels —50, 100, 200 ppm based on total volume
Test fluid — NACE brine/kerosene, 90/10 ratio by volume

C. Procedure:

1. Purge brine 30 minutes per liter with $CO_2$ to saturate.

2. Put 18 mL kerosene into each bottle (purge may be required).

3. Put proper level of corrosion inhibitor into each bottle.

4. With appropriate gas cover on brine reservoir, purge bottle with $CO_2$ for 30 seconds, put test specimens in, fill with 162 mL brine, pull $CO_2$ purge tube from bottle, and cap immediately.

5. Include at least one blank per run (no corrosion inhibitor).

6. Put bottles into 49°C (120°F) oven.

7. Remove from oven after proper interval, typically from one to three days.

8. Clean test specimens as follows:

(a) Wash in xylene/methyl alcohol solvent.
(b) Immerse in 15% hydrochloric acid (inhibited) for 30 seconds.

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(c)     Rinse test specimens to remove acid.
(d)     Scrub with 000 extra-fine steel wool using moderate hand pressure.  Use same number of cleaning strokes for each test specimen.
(e)     Rinse with tap water, rinse with acetone, and blow dry with air.  Store in desiccator.

9.     Reweigh test specimens.

10.    Calculate mm/y (mpy) corrosion rate for each test specimen and average the three for each bottle. Record.

**Inhibitor Test Results**

**Ranking of Three Tested Inhibitors (A, B, C) for Each Test Method**
**by**
**Nine Companies**

| Company | Wheel Test | LPR Test |
|---------|-----------|----------|
| 1 | B > A > C | B > A > C |
| 2 | A = B > C | A = B > C |
| 3 | B = A > C | B > A > C |
| 4 | B > A > C | B > A > C |
| 5 | B > A > C | B > A > C |
| 6 | A = B = C | B > A > C |
| 7 | B = A > C | ---- |
| 8 | B > A > C | B > A = C |
| 9 | B > A > C | B > A > C |

Note:    Inhibitor effectiveness decreases in the order of X > Y > Z.

**APPENDIX B**
**Definitions**

**Corrosion**: The deterioration of a material, usually a metal, that results from a reaction with its environment.

**Corrosion Inhibitor**: A chemical substance or combination of substances that, when present in the environment, prevents or reduces corrosion.

**Corrosion Rate**: The rate at which corrosion proceeds.

**Crevice Corrosion**: Localized corrosion of a metal surface at, or immediately adjacent to, an area that is shielded from full exposure to the environment because of close proximity of the metal to the surface of another material.

**Electrode**: A conductor used to establish contact with an electrolyte and through which current is transferred to or from an electrolyte.

**Electrolyte**: A chemical substance containing ions that migrate in an electric field.

**Erosion-Corrosion**: A conjoint action involving corrosion and erosion in the presence of a moving corrosive fluid or a material moving through the fluid, leading to accelerated loss of material.

**Linear Polarization Resistance (LPR)**: An electrochemical test technique that measures the relationship between current and potential near the open-circuit potential.

**LPR Test**: A corrosion test using the linear polarization resistance technique performed in a kettle or suitable test vessel under controlled temperature and agitation conditions. It may be "static," "laminar," "vapor phase partitioning," or "high shear," depending on the desired results.

**Open-Circuit Potential**: The potential of an electrode measured with respect to a reference electrode or another electrode in the absence of current.

**Percent Protection**: A measure of the efficiency of a corrosion inhibitor for protecting a metallic surface during contact with corrodents.

**Pitting**: Localized corrosion of a metal surface that is confined to a small area and takes the form of cavities called pits.

**Potentiostat**: An instrument for automatically maintaining a constant electrode potential.

**Reference Electrode**: An electrode whose open-circuit potential is constant under similar conditions of measurement, which is used for measuring the relative potentials of other electrodes.

**Reynolds Number (Re)**: A dimensionless number defined as the product of velocity, density, and pipe diameter divided by viscosity.

**Wheel Test**: A corrosion test that uses a controlled temperature box with a rotating wheel to which bottles containing test specimens are attached. Test results are based on mass (weight) loss and visual examination of the test specimen in a corrosive environment.