Designation: G185 − 06 (Reapproved 2016)

Standard Practice for
Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using the Rotating Cylinder Electrode[1]

This standard is issued under the fixed designation G185; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a generally accepted procedure to use the rotating cylinder electrode (RCE) for evaluating corrosion inhibitors for oil field and refinery applications in defined flow conditions.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1141 Practice for the Preparation of Substitute Ocean Water
D4410 Terminology for Fluvial Sediment
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements
G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]
G16 Guide for Applying Statistics to Analysis of Corrosion Data
G31 Guide for Laboratory Immersion Corrosion Testing of Metals
G46 Guide for Examination and Evaluation of Pitting Corrosion
G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements
G96 Guide for Online Monitoring of Corrosion in Plant Equipment (Electrical and Electrochemical Methods)
G102 Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements
G106 Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements
G111 Guide for Corrosion Tests in High Temperature or High Pressure Environment, or Both
G170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory

3. Terminology

3.1 The terminology used throughout shall be in accordance with Terminologies G15 and D4410 and Guide G170.

4. Summary of Practice

4.1 This practice provides a method of evaluating corrosion inhibitor efficiency in a RCE apparatus. The method uses a well-defined rotating specimen set up and mass loss or electrochemical measurements to determine corrosion rates in a laboratory apparatus. Measurements are made at a number of rotating rates to evaluate the inhibitor performance under increasingly severe hydrodynamic conditions.

5. Significance and Use

5.1 Selection of corrosion inhibitor for oil field and refinery applications involves qualification of corrosion inhibitors in the laboratory (see Guide G170). Field conditions should be simulated in the laboratory in a fast and cost-effective manner (**1**).[4]

5.2 Oil field corrosion inhibitors should provide protection over a range of flow conditions from stagnant to that found during typical production conditions. Not all inhibitors are equally effective over this range of conditions so that is

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[1] This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

important for a proper evaluation of inhibitors to test the inhibitors using a range of flow conditions.

5.3 The RCE is a compact and relatively inexpensive approach to obtaining varying hydrodynamic conditions in a laboratory apparatus. It allows electrochemical methods of estimating corrosion rates on the specimen and produces a uniform hydrodynamic state across the metal test surface. (**2-21**)

5.4 In this practice, a general procedure is presented to obtain reproducible results using RCE to simulate the effects of different types of coupon materials, inhibitor concentrations, oil, gas and brine compositions, temperature, pressure, and flow. Oil field fluids may often contain sand. This practice does not cover erosive effects that occur when sand is present.

6. Apparatus

6.1 Fig. 1 shows a schematic diagram of the RCE system. The RCE apparatus consists of a rotating unit driven by a motor that is attached to a sample holder. A system with a range of rotational speeds from 100 to 10 000 rpm with an accuracy of ±2 rpm is typical. It is essential to be able to rotate the electrode at both low and high speeds and to be able to measure the speed and maintain it at a constant. The accuracy of the rotation rate should be checked. At the side of the sample holder where it is outside the cell, electrical connections to the electrodes are made by a brush contact. It is important for the connection to be as noise free as possible.

6.2 The cylinder geometry is usually defined in terms of the length-to-diameter ratio. Both low and high ratios are used, with ratios varying between 0.3 and 3.0. The rotating cylinder can also be used as a mass loss coupon when the mass loss is sufficiently large to be accurately measured using a conventional balance (with accuracy of 0.1 mg).

6.3 The RCE geometry may have an inner cylinder and an outer cylinder. The geometry is usually defined in terms of the radius of the inner cylinder and the radius of the outer cylinder. When the outer diameter is several times the diameter of the inner electrode the hydrodynamics are essentially controlled by the diameter of the inner rotating cylinder (**2**). The outer cylinder may act as counter electrode. An RCE with only an inner cylinder may also be used.

6.4 A saturated calomel electrode (SCE) with a controlled rate of leakage or a saturated calomel electrode utilizing a semipermeable membrane or porous plug tip or silver/silver chloride or any other suitable electrode should be used as reference electrode. The potential of the reference electrode should be checked at periodic intervals to ensure the accuracy of the electrode. For experiments at higher-temperature, a higher-pressure, reference electrode arrangement that can withstand higher temperature and pressure should be used (**22**). This may require special care.

6.5 Fig. 2 shows a typical rotating electrode unit. A rotating shaft can be modified by drilling a hole in the shaft into which a polytetrafluoroethylene (PTFE) insulator is inserted. Inside the PTFE insulator, a metal rod should be introduced (Fig. 2). One end of the metal rod is threaded so that the cylindrical electrode can be attached. The other end of the rod is attached directly to the rotating unit, through which the electrical connection is made.

6.6 After attaching the specimen to the shaft, the system should be checked for eccentricity and wobble. This can be accomplished by installing a dial micrometer so as to monitor the location of the top of the rotating cylinder and rotating the shaft slowly through one complete turn. The micrometer should then be moved to monitor the center of the specimen, and the process repeated. Finally the micrometer should be moved to the bottom of the specimen and the process repeated. The assembly should also be rotated at its maximum rotation rate and the specimen wobble checked again using, for example, a laser indicator or vibration monitor.

6.7 Appropriate cylinder specimen (such as, carbon steel) is machined and snugly fitted into the PTFE or any other suitable specimen holder (Fig. 2). The presence of gap between specimen and holder will create crevice corrosion as well as change the flow pattern. If necessary, apply a very small amount of epoxy to fit the specimen into the holder. Tightly attach or screw an end-cap so that only the outer cylindrical area of known length is exposed to the solution. The specimen holder is then attached to the rotating unit. Specimen, holder, and end-cap should all have the same diameter.

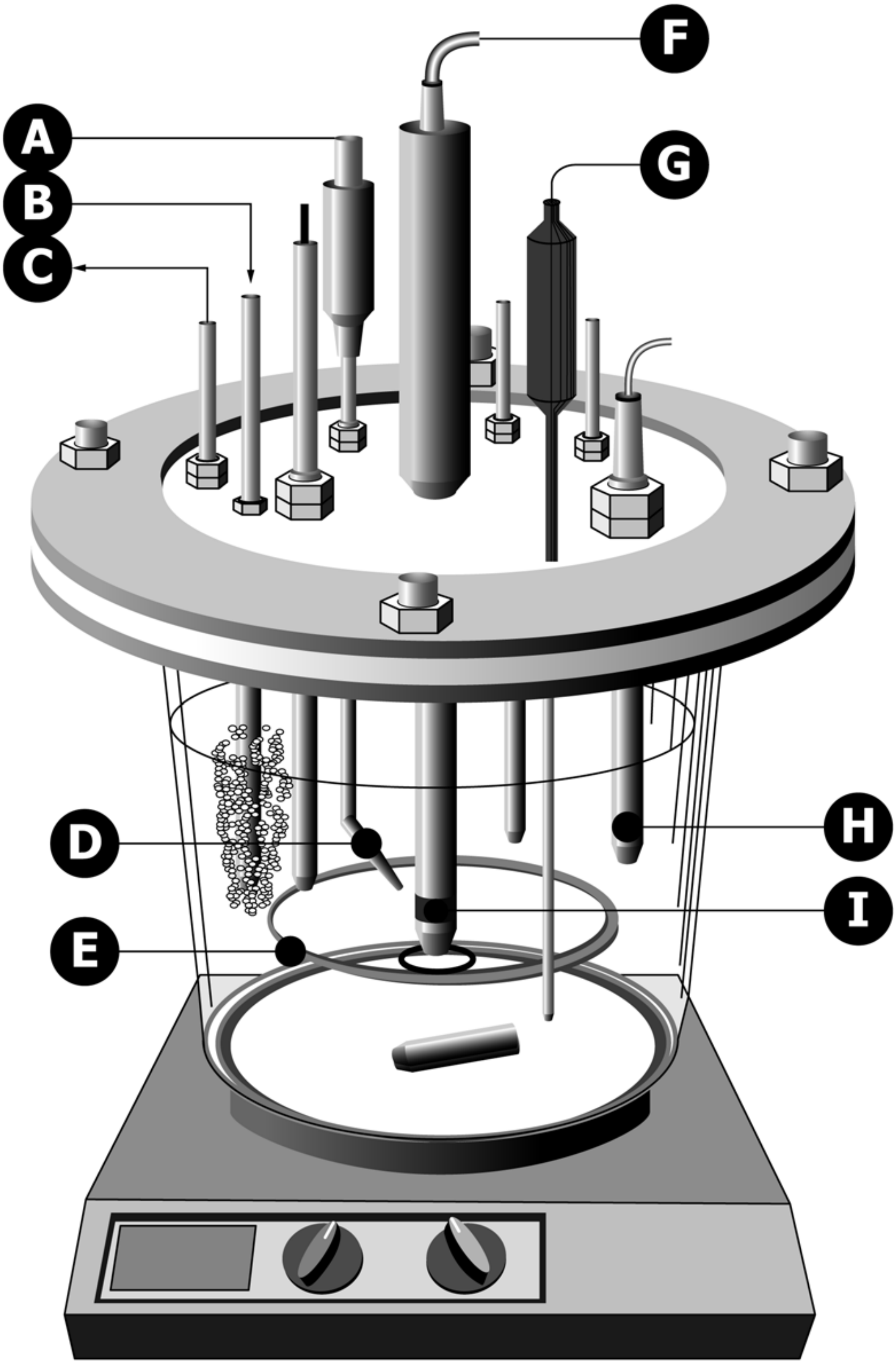
6.8 The rotating unit is attached into the experimental vessel, ensuring that there is no leakage through the rotating shaft and the holder and that the rotating shaft is vertically positioned. Even a very slight inclination could drastically change the flow pattern.

6.9 A versatile and convenient apparatus, consisting of a kettle or flask (Fig. 1) of suitable size (usually 500 to 5000 mL), inlet and outlet ports for deaeration, thermowell and temperature-regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system, should be used. The volume (of the solution) to surface area (of the specimen) ratio has some effect on the corrosion rate and hence inhibitor efficiencies. A larger volume/surface area (minimum 40 mL/cm^2) should be preferred.

6.10 In some cases a wide-mouth jar with a suitable closure can be used, but open-beaker tests should not be used because of evaporation and contamination. Do not conduct the open-beaker test when H_2S (hydrogen sulfide) is used. In more complex tests, provisions might be needed for continuous flow or replenishment of the corrosive liquid, while simultaneously maintaining a controlled atmosphere.

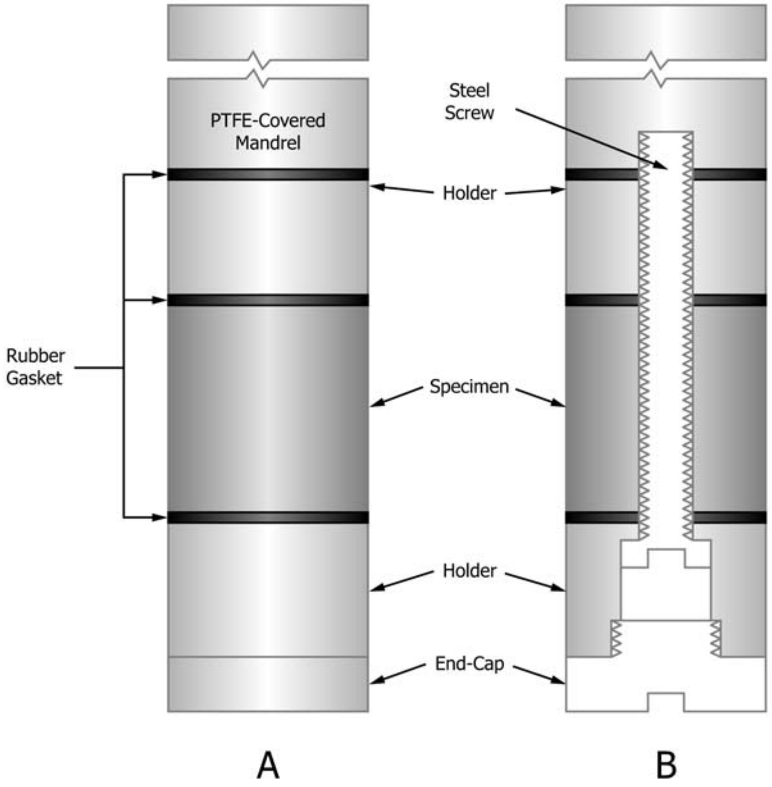
6.11 For experiments above atmospheric pressure, a high-temperature, high-pressure rotating cylinder electrode (HTHPRCE) system with an electrically isolated electrode system, an electrically isolated motor for rotating the electrode, and a vessel that can withstand high pressure without leakage should be used.

6.12 A design of the vessel that can be used in elevated pressure conditions (**23, 24**) include a standard autoclave (Fig. 3) modified by lining on the inside with PTFE. The stirring rod can be modified by drilling a hole into that a PTFE insulator is inserted. Inside the PTFE insulator, a metal rod is introduced.



A. Reference Electrode
B. Inlet
C. Outlet
D. Luggin Capillary
E. Counter Electrode
F. Rotating Cylinder
G. Temperature Probe
H. pH Electrode
I. Rotating Cylinder Electrode or Coupon

FIG. 1 Schematic of a RCE System (18)



A. Outside View
B. Cross-Sectional View

FIG. 2 Schematic Representation of a RCE with its Components (adapted from Ref 18)

Three O-rings are used to secure and to prevent leakage. One end of the metal rod is threaded so that cylindrical (Fig. 3) electrode can be attached. The other end of the rod, projecting slightly above the motor unit, is attached directly the rotating unit, through which the electrical connection is made. The rod is rotated by a motor connected to the rod using a belt. The counter and reference electrodes are inserted inside the autoclave.

6.13 The suggested components can be modified, simplified, or made more sophisticated to fit the needs of a particular investigation.

7. Materials

7.1 Methods for preparing specimens for tests and for removing specimens after the test are described in Practice G1. Standard laboratory glassware should be used for weighing and measuring reagent volumes.

7.2 The specimen shall be made of the material (such as, carbon steel) for which the inhibitor is being evaluated. The specimen should have same metallographic structure as that used in the service components. The specimens should be ground to a specified surface finish (such as, 150-grit). The grinding should produce a reproducible surface finish, with no rust deposits, pits, or deep scratches. All sharp edges on the specimen should be ground. All loose dirt particles should be removed.

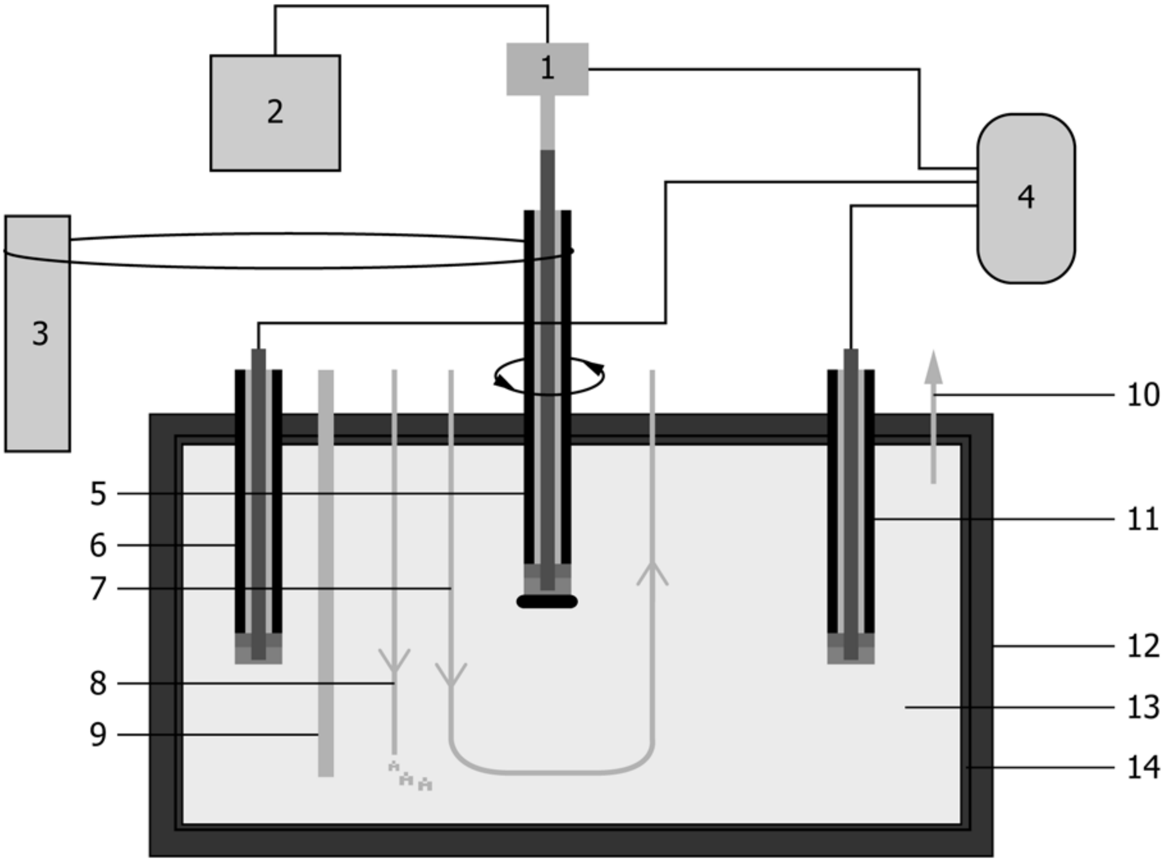
7.3 The specimens are rinsed with distilled water, degreased by immersing in acetone (or any suitable alcohol), ultrasonically cleaned for 1 minute, and dried. The surface of the specimens should not be touched with bare hands. The specimens are weighed to the nearest 0.1 mg (for mass loss measurements), the dimensions are measured to the nearest 0.1 mm, and the surface area is calculated.

7.4 Freshly prepared specimens are installed in the RCE holder. If the test is not commenced within 4 h, the prepared coupons shall be stored in a desiccator to avoid pre-rusting.

8. Test Solutions

8.1 All solutions (oil and aqueous) should be obtained from the field for which the inhibitor is being evaluated. These are known as live solutions. It is important that live solutions do not already contain corrosion inhibitor. In the absence of live solutions, synthetic solutions should be used, the composition of which should be based on field water analysis. The composition of the solution should be determined and reported. Alternatively, standard brine (such as per Practice D1141) should be employed. The solutions should be prepared using analytical grade reagents and deionized water.

8.2 The solutions should be deoxygenated by passing nitrogen or any other inert gas for sufficient time to reduce the oxygen content below 5 ppb and preferably below 1 ppb in solution. The solution must be kept under deoxygenated conditions. The oxygen concentration in solution depends on the quality of gases used to purge the solution. Any leaks through vessel, tubing, and joints shall be avoided.



1. Electrical Contact Unit
2. Techometer (Rotation Speed Display)
3. Rotation Controller
4. Electrochemical Instruments
5. Working Electrode
6. Reference Electrode
7. Water Cooler Coil
8. Gas Inlet
9. Thermocouple
10. Gas Outlet
11. Counter Electrode
12. Autoclave Body
13. Solution
14. PTFE Liner

FIG. 3 Schematic Diagram of HTHPRCE System (20,21)

8.3 The appropriate composition of gases is determined by the composition of gases in the field for which the inhibitor is evaluated. Hydrogen sulfide (H_2S) and carbon dioxide (CO_2) are corrosive gases. H_2S is poisonous and should not be released to the atmosphere. The appropriate composition of gas can be obtained by mixing H_2S and CO_2 streams from the standard laboratory gas supply. Nitrogen or other inert gases can be used as a diluent to obtain the required ratios of the corrosive gases. Alternatively, gas mixtures of the required compositions can be purchased from suppliers of industrial gases. The concentrations of impurities, particularly oxygen, shall be kept as low as possible with guidelines of below 5 ppb and preferably under 1 ppb oxygen in solution.

8.4 The solution pH before and after testing shall be measured, recorded, and reported. The solution pH should be monitored regularly (at least once a day) during the test.

8.5 Inhibitor concentrations should be measured and reported in % mass/volume or parts per million (ppm). The method of injecting the inhibitor into the test solution should reflect the actual field application. Water-soluble inhibitors may be injected neat (as-received) into the test solution (aqueous phase). To avoid the errors associated with handling small volumes of solution, an inhibitor stock solution may be prepared by diluting the as-received chemical in an appropriate solvent. The type of solvent and the concentration of the stock solution depend on the characteristics of the inhibitor and on the specified test conditions.

8.6 Oil-soluble, water-dispersible inhibitor solutions are prepared by the partition method. The required amounts of oil and brine are placed in the partitioning vessel (usually a separation funnel). The relative volumes of oil and aqueous phases should reflect the ratios of water and oil in the field for which the inhibitor is evaluated. If samples from the field are not available, heptane, kerosine or any suitable hydrocarbon may be used. The corrosion inhibitor is added to the oil phase. The vessel is vigorously shaken for one minute to mix both phases thoroughly, and the phases are allowed to separate. Heating to the temperature of the field helps in the separation. The aqueous phase is removed and used as test solution.

8.7 Oil-soluble inhibitors (usually as batch inhibitors) are dissolved in the oil phase to form an inhibited oil-phase. The coupons are exposed to this solution for a certain amount of time (usually 30 min). The coupons are then removed and introduced into the experimental vessel.

9. Experimental Procedure for Atmospheric Pressure Experiments

9.1 Detailed procedures to determine corrosion rates using electrochemical instruments are described in Practice G3, Test Method G5, Test Method G59, Guide G96, Practice G102, and Practice G106, and a procedure to determine corrosion rates from mass loss is described in Practice G31.

9.2 Solutions are usually prepared in a separate container called the preparation vessel, pre-saturated with the required gas mixture, and preheated to the required temperature. Pretreatment described in 8.5 – 8.7 is usually carried out in the preparation vessel. Transfer solutions from the preparation vessel to the experimental vessel (described in Section 6) under positive nitrogen or other inert gas pressure to minimize air contamination during the transfer operation.

9.3 Depending on the size of experimental vessel, heating unit (mantle, bath, or wrapper around the vessel), difference between room and experimental temperatures, a range of temperature may prevail within the vessel. Take care to avoid or minimize the temperature differentials. Heat the test vessels slowly (usually at a rate of 0.1°C/s) to avoid overheating. The exact protocol depends on the controller, the size and output of the heater, and parameters such as vessel size, amount of liquid, thermal conductivity of liquid, and agitation. Maintain the test temperature within 2°C of the specified temperature.

9.4 Insert pre-weighed coupons (pretreated as necessary, such as with batch inhibitors), thermometer, and pH probes (as appropriate). Position the liquid inlet and outlet such that none of them are protruding into the solution.

9.5 Initially all other ports of the experimental vessel, except inlet and outlet ports are closed. The inlet tube should have a Y-joint. Attach one end to the experimental vessel. Attach the other two ends to the preparation vessel and to an inert gas, for example, argon or nitrogen cylinder.

9.6 First, pass the inert gas to expel oxygen from the experimental vessel.

9.7 After 15 min, stop the gas flow, and close the passage between the experimental vessel and the gas cylinder.

9.8 Close the passage between the experimental and preparation vessels.

9.9 Open the passage between the experimental and preparation vessels, and pump the gas-saturated brine, which may or may not contain inhibitor prepared as per 8.6 or 8.7, into the experimental vessel. Maintain the experimental vessel with the heater or the water bath at the required temperature.

9.10 The additional gas-inlet on top of the vessel should allow for keeping the gas mixture blanket on top of the solution, which is required when the experiment is planned for longer duration, for example, more than 24 h. Keep the gas flow rate to a minimum. Take care that the gas does not entrain with the solution.

9.11 The speed controller is used to preset the rotation speed and to start the motor. The rotation speed usually stabilizes, as displayed by the tachometer, within 30 s. Alternatively, the rotation speed can be set prior to pumping the solution into the vessel.

9.12 The electrodes (working, counter, and reference) are connected to the electrochemical instruments. After the potential has reasonably stabilized (usually within 1 h), perform the electrochemical measurements (LPR or EIS) at regular intervals. Procedures to determine corrosion rate from electrochemical measurements are described in Guide G96.

9.13 After the predetermined time or after the corrosion rate is stabilized, the continuous inhibitor may be injected and the corrosion rate can be monitored.

9.14 For mass loss experiments, terminate the experiment (typically after 24 h), and determine the corrosion rate from the amount of metal loss (after proper cleaning as described in Practice G1) as described in Practice G31. The samples are examined and evaluated for pitting corrosion as in Guide G46. If the experiments are repeated two or more times, then calculate the average, standard deviation and coefficient of variation using the methods provided in Guide G16.

9.15 Determine the Inhibitor efficiency at each rotation speed and at each inhibitor concentration using the following equation:

$$\textit{Inhibitor Efficiency, \%} = \frac{[C.R]_{No.inhibitor} - [C.R]_{Inhibitor \times 100}}{[C.R]_{No\ Inhibitor}} \quad (1)$$

where:

$[C.R]_{No.inhibitor}$ = the corrosion rate in absence of inhibitor, and

$[C.R]_{inhibitor}$ = the corrosion rate in the presence of inhibitor.

10. Experimental Procedure for High-Temperature, High-Pressure Experiments

10.1 A general procedure to carry out corrosion experiments at elevated pressure and temperature is described in Guide G111. Before the experiments, check the autoclave for safety and integrity at a pressure that is about 1.5 times the pressure at which the experiment is planned. For example, if the experiment is planned at 250 psi (1724 kPa), test the system at about 400 psi (2759 kPa). This testing is required to ensure the safety of the personnel and the equipment, and also to detect any leak. After pressurizing the autoclave to the preset value, close the inlet and outlet valves. After 30 min, test the autoclave for any leaks. If any leak is found, tighten the autoclave head screws and recheck for any further leak. If there is still any pressure loss, then the system is faulty. Stop the experiment and have the autoclave inspected and repaired. If the pressure holds constant for more than 30 min, release the pressure by opening the outlet. Once the pressure gauge shows that the pressure has been released, close the outlet valve.

10.2 As in the atmospheric pressure experiments, follow the steps in 9.2 – 9.11 to charge the autoclave.

10.3 For high-temperature, high-pressure experiments using a premixed gas composition, pressurize the autoclave using the specified gas composition, and depressurize to approximately 0.2 bars above atmospheric pressure. Repeat this cycle of pressurizing/depressurizing at least twice to ensure that the gas space has the required composition. Finally, pressurize the autoclave to the test pressure.

10.4 For high-temperature, high-pressure experiments (**25, 26**) using individual gases, pressurize the autoclave with H_2S to the required partial pressure and leave for 10 min. If there is a decrease of pressure, pressurize the autoclave again. Repeat this process until there is no further pressure loss. Then, pressurize the autoclave with CO_2 by opening the CO_2 gas cylinder at a pressure equal to the CO_2 and H_2S partial pressures, and leave it for 10 min. If there is a decrease of pressure, pressurize the autoclave again with CO_2. Repeat this process until no further pressure loss is reported. Finally, pressurize the autoclave with the inert gas by opening the inert gas cylinder at the total gas pressure at which the experiments are to be carried out.

10.5 Carry out experiments using the same procedure as that for atmospheric pressure experiments (see 9.12 – 9.14).

11. Report

11.1 Record all information and data as completely as possible. Practice G31 provides a checklist for reporting corrosion data.

11.2 Average corrosion rates and the standard deviation of each concentration of inhibitor at each rotation rate should be reported.

11.3 The following checklist is a recommended guide for reporting important information:

11.3.1 Solution chemistry and concentration (any changes during test).

11.3.2 Volume of test solution.

11.3.3 Volume of the experimental vessel.

11.3.4 Temperature (maximum, minimum, average).

11.3.5 Pressure (maximum, minimum, average).

11.3.6 Duration of each test.

11.3.7 Chemical composition or trade name of metal.

11.3.8 Form and metallurgical conditions of specimen.

11.3.9 Exact size, shape, and area of specimen.

11.3.10 Inhibitor type and concentration.

11.3.11 Treatment used (including e.g., batch inhibitor) to prepare specimens.

11.3.12 Type of corrosion measurements, such as mass loss or electrochemical (that is, linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS)). Practice G1 and Practice G102 provide methods to determine corrosion rates from mass loss and electrochemical measurements respectively.

11.3.13 For electrochemical measurements, size, shape, and distance (from working electrode) of counter and reference electrodes, Tafel constants, solution resistance, number and frequency of measurements, and EIS model. Test Method G5 and Practice G106 provides information for making polarization and impedance measurements.

11.3.14 Test method used to clean specimens after experiment and the extent of any error expected by this treatment.

11.3.15 Initial and final masses and actual mass losses for mass loss measurement

11.3.16 Inhibitor efficiency at each concentration and at each rotation speed.

11.3.17 Evaluation of attack if other than general, such as pit depth and distribution, standard deviation and coefficient of variation, crevice corrosion, and results of microscopical examination.

12. Keywords

12.1 corrosion inhibitor; electrochemical; high-pressure; high-temperature; laboratory evaluation; mass loss; oil-field inhibitors; RCE; refinery inhibitors; rotating cylinder electrode

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