



Standard Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using Rotating Cage¹

This standard is issued under the fixed designation G 184; 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 cage (RC) for evaluating corrosion inhibitors for oil field and refinery applications.

1.2 The values stated in SI units are to be regarded as the 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

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G 15 Terminology Relating to Corrosion and Corrosion Testing

G 16 Guide for Applying Statistics to Analysis of Corrosion Data

G 31 Practice for Laboratory Immersion Corrosion Testing of Metals

G 46 Guide for Examination and Evaluation of Pitting Corrosion

G 111 Guide for Corrosion Tests in High Temperature or High Pressure Environment, or Both

G 170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory

D 1141 Practice for the Preparation of Substitute Ocean Water

D 4410 Terminology for Fluvial Sediment

3. Terminology

3.1 The terminology used throughout shall be in accordance with Terminologies **G 15** and **D 4410** and Guide **G 170**.

4. Summary of Practice

4.1 This practice provides a method of evaluating corrosion inhibitor efficiency in a RC apparatus. The method uses a well-defined rotating specimen setup and mass loss measurements to determine corrosion rates in a laboratory apparatus. Measurements are made at a number of rotation 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 **G 170**). Field conditions should be simulated in the laboratory in a fast and cost-effective manner (**1**).³

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

5.3 The RC test system is relatively inexpensive and uses simple flat specimens that allow replicates to be run with each setup. (**2-13**).

5.4 In this practice, a general procedure is presented to obtain reproducible results using RC 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; however, this practice does not cover erosive effects that occur when sand is present.

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 boldface numbers in parentheses refer to the list of references at the end of this standard.

6. Apparatus

6.1 Fig. 1 shows the schematic diagram of the RC system. An apparatus of suitable size (usually 7500 mL) is used, consisting of inlet and outlet ports, thermowell, temperature-regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system.

6.1.1 The vessel (typically 150-mm diameter) is manufactured from an inert material. Cast acrylic and polytetrafluoroethylene (PTFE) have been used.

6.1.2 A PTFE base is fitted at the bottom of the container. At the center of the base, a hole is drilled into which the lower end of a stirring rod is placed. This arrangement stabilizes the stirrer and the coupons.

6.1.3 Typically, eight coupons (each of 75-mm length, 19-mm width, and 3-mm thickness, and a surface area of about 34.14 cm²) are supported between two PTFE disks (of 80-mm diameter) mounted 75 mm apart on the stirring rod (Fig. 2). Holes (10-mm diameter) about 15 mm away from the center are drilled in the top and bottom PTFE plates of the cage to increase the turbulence on the inside surface of the coupon (Fig. 3). This experimental setup can be used at temperatures up to 70°C and rotation speeds up to 1000 rpm.

6.2 The flow pattern varies, depending on the rotation speed, the volume of the container, and the fluids. The flow patterns are described in Guide G 170.



NOTE—Gaps (typically 0.85 ± 0.01 cm) between the coupons introduce localized turbulence.

FIG. 2 Photo of Rotating Cage Containing Coupons

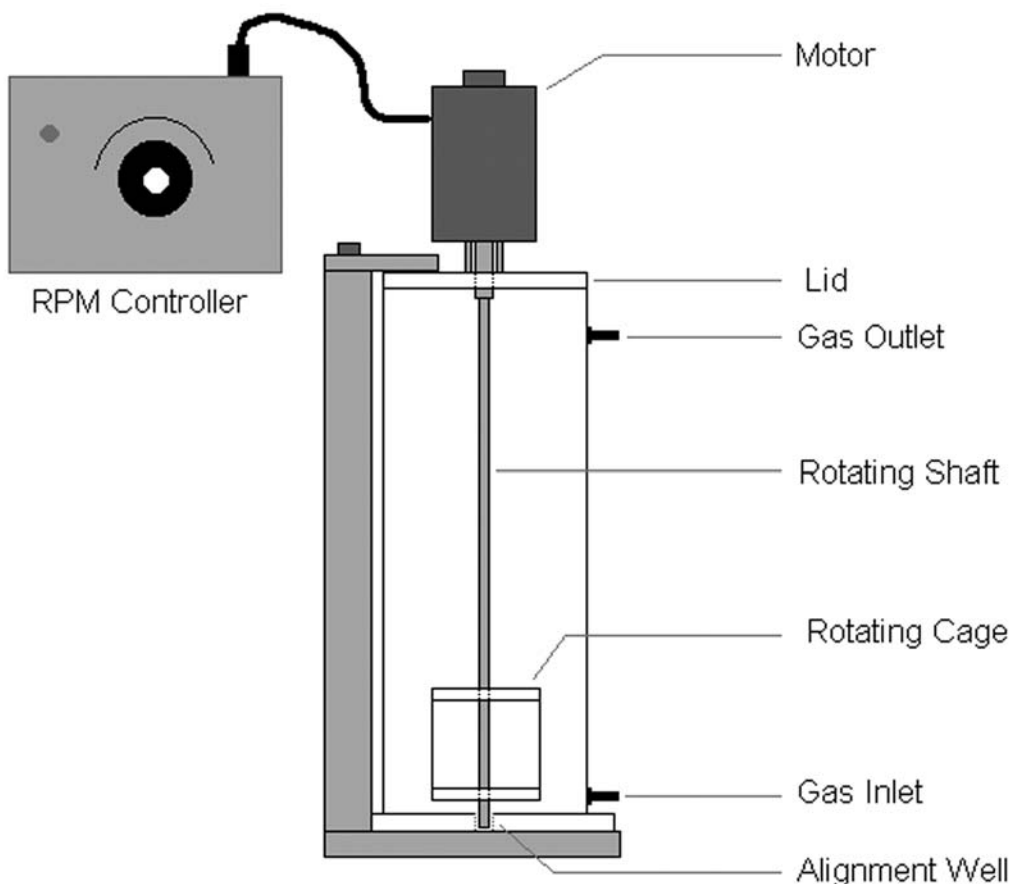
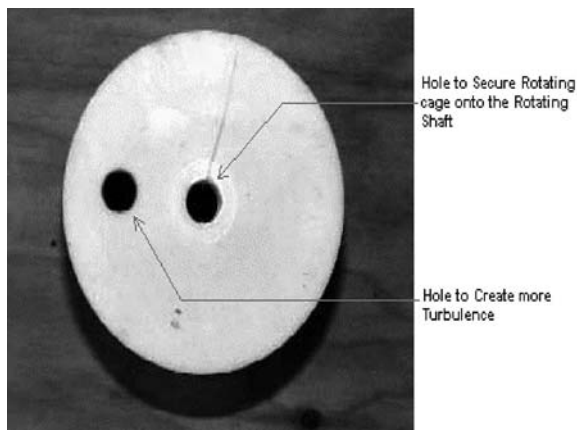


FIG. 1 Schematic Diagram of Rotating Cage



NOTE—Holes (typically 1.0 cm in diameter, and about 1.5 cm from the center) introduce localized turbulence.

FIG. 3 Photo of Rotating Cage (Top View)

6.3 Volume of solution to the surface area of the specimen has some effect on the corrosion rate and hence on the inhibitor efficiencies. The minimum solution volume to metal surface area is not less than 14 cm (11).

6.4 Open-beaker tests should not be used because of evaporation and contamination. Open-beaker test must not be conducted when H_2S (hydrogen sulfide) is used. In some tests, provisions might be needed for continuous flow or replenishment of the corrosive liquid, while simultaneously maintaining a controlled atmosphere.

6.5 For experiments above atmospheric pressure, a high-temperature, high-pressure rotating cage (HTHPRC) system and a vessel that can withstand high pressure without leakage shall be used.

6.6 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 G 1. Standard laboratory glassware should be used for weighing and measuring reagent volumes.

7.2 The coupons shall be made of the material (such as carbon steel) for which the inhibitor is being evaluated. The coupon should have the same metallographic structure as that used in the service components. The coupons 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 coupon should be ground. All loose dirt particles should be removed.

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

7.4 Freshly prepared specimens are installed in the rotating cage 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 in Practice D 1141) 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 the vessel, tubing, and joints shall be avoided.

8.3 The appropriate composition of gases is determined by the composition of gases in the field for which the inhibitor is evaluated. (Warning—Hydrogen sulfide (H_2S) and carbon dioxide (CO_2) are corrosive gases.) (Warning— H_2S is poisonous and should not be released into 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 composition of 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 following 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, kerosene, or any suitable hydrocarbon may be used. The corrosion inhibitor is added to

the oil phase. The vessel is vigorously shaken for 1 min 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 A detailed procedure to determine corrosion rates from mass loss is described in Practice G 31.

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. (Pre-treatment described in Sections 8.4, 8.5, and 8.6 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 the experimental vessel, heating unit (mantle, bath, or wrapper around the vessel), difference between room, and experimental temperatures, a range of temperature may occur 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 so that none of them is protruding into the solution.

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

9.6 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 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 8.4 or 8.5, into the experimental vessel.

9.9 Close the passage between the experimental and preparation vessels. 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 keeping the gas mixture blanket on top of the solution, which is required when the experiment is planned for a 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 Use the speed controller 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 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 G 1) as described in Practice G 31. Examine and evaluate the samples for pitting corrosion as in Guide G 46. Calculate the average, standard deviation, and coefficient of variation of the coupons corrosion rate for each run using the method presented in Guide G 16. If pitting corrosion is observed, then the general corrosion rate determined from mass loss could be invalid.

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

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

where:

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

$[C.R]_{\text{inhibitor}}$ = 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 G 111.

10.1.1 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.

10.1.2 After pressurizing the autoclave to the preset value, close the inlet and outlet valves.

10.1.2.1 After 30 min, test the autoclave for any leaks.

10.1.2.2 If any leak is found, tighten the autoclave head screws and recheck for any further leak.

10.1.2.3 If there is still any pressure loss, then the system is faulty. Stop the experiment, and have the autoclave inspected and repaired.

10.1.2.4 If the pressure holds constant for more than 30 min, release the pressure by opening the outlet. Once the pressure gage shows that the pressure has been released, close the outlet valve.

10.2 Follow the steps as in the atmospheric pressure experiments (see 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.

10.3.1 Repeat this cycle of pressurizing/depressurizing at least twice to ensure that the gas cap has the required composition.

10.3.2 Finally, pressurize the autoclave to the test pressure.

10.4 For high-temperature, high-pressure experiments (14, 15) using individual gases, pressurize the autoclave with H₂S to the required partial pressure and leave it for 10 min.

10.4.1 If there is a decrease of pressure, pressurize the autoclave again. Repeat this process until there is no further pressure loss.

10.4.2 Then, pressurize the autoclave with CO₂ by opening the CO₂ gas cylinder at a pressure equal to the CO₂ and H₂S partial pressures and leaving it for 10 min. If there is a decrease of pressure, pressurize the autoclave again with CO₂. Repeat this process until there is no further pressure loss.

10.4.3 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 the experiments using the same procedure as that for atmospheric pressure experiments (9.11 and 9.12).

11. Report

11.1 All information and data shall be recorded as completely as possible. Practice G 31 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 Number, form, and metallurgical conditions of specimens.

11.3.9 Exact size, shape, and area of each specimen.

11.3.10 Inhibitor type and concentration.

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

11.3.12 Method used to clean specimens after experiment and the extent of any error expected by this treatment.

11.3.13 Initial and final masses and actual mass losses.

11.3.14 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; high-pressure; high-temperature; laboratory evaluation; mass loss; oil-field inhibitors; RC; refinery inhibitors; rotating cage

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