



Designation: G 170 – 01a

## Standard Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory<sup>1</sup>

This standard is issued under the fixed designation G 170; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This guide covers some generally accepted laboratory methodologies that are used for evaluating corrosion inhibitors for oilfield and refinery applications in well defined flow conditions.

1.2 This guide does not cover detailed calculations and methods, but rather covers a range of approaches which have found application in inhibitor evaluation.

1.3 Only those methodologies that have found wide acceptance in inhibitor evaluation are considered in this guide.

1.4 This guide is intended to assist in the selection of methodologies that can be used for evaluating corrosion inhibitors.

1.5 *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 requirements prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1141 Practice for Preparation of Substitute Ocean Water<sup>2</sup>

D 4410 Terminology for Fluvial Sediment<sup>3</sup>

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens<sup>4</sup>

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing<sup>4</sup>

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements<sup>4</sup>

G 15 Terminology Relating to Corrosion and Corrosion Testing<sup>4</sup>

G 16 Guide for Applying Statistics to Analysis of Corrosion Data<sup>4</sup>

G 31 Practice for Laboratory Immersion Corrosion Testing of Metals<sup>4</sup>

G 46 Guide for Examination and Evaluation of Pitting Corrosion<sup>4</sup>

G 59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements<sup>4</sup>

G 96 Guide for On-Line Monitoring of Corrosion in Plant Equipment (Electrical and Electrochemical Methods)<sup>4</sup>

G 102 Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements<sup>4</sup>

G 106 Practice for Verification of Algorithm and Equipment for Electrochemical Impedance Measurements<sup>4</sup>

G 111 Guide for Corrosion Tests in High Temperature or High Pressure Environment, or Both<sup>4</sup>

#### 2.2 NACE Standards:<sup>5</sup>

NACE-5A195 State-of-the-Art Report on Controlled-Flow Laboratory Corrosion Test, Houston, TX, NACE International Publication, Item No. 24187, December 1995

NACE-ID196 Laboratory Test Methods for Evaluating Oil-Field Corrosion Inhibitors, Houston, TX, NACE International Publication, Item No. 24192, December 1996

NACE-TM0196 Standard Test Method "Chemical Resistance of Polymeric Materials by Periodic Evaluation," Houston, TX, NACE International Publication, Item No. 21226, 1996

#### 2.3 ISO Standards:<sup>6</sup>

ISO 696 Surface Active Agents — Measurements of Foaming Power Modified Ross-Miles Method

ISO 6614 Petroleum Products — Determination of Water Separability of Petroleum Oils and Synthetic Fluids

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *atmospheric pressure experiment*—an experiment conducted at the ambient atmospheric pressure (typically less than 0.07 MPa (10 psig)), using normal laboratory glassware.

3.1.2 *batch inhibitor*—an inhibitor that forms a film on the metal surface that persists to effect inhibition.

3.1.3 *batch treatment*—a method of applying a batch inhibitor. Batch inhibitors are applied as a plug between pigs or as

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.02.

<sup>5</sup> Available from National Assoc. of Corrosion Engineers, P.O. Box 218340, Houston, TX 77218.

<sup>6</sup> Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

slugs of chemical poured down the well bore. The batch inhibitor is dissolved or dispersed in a medium, usually hydrocarbon and the inhibited solution is allowed to be in contact with the surface that is to be protected for a fixed amount of time. During this period, the inhibitor film is formed on the surface and protects the surface during the passage of multiphase flow, for example, oil/water/gas.

3.1.4 *continuous inhibitor*—an inhibitor that is continuously injected into the system in order to effect inhibition. Since the surface receives full exposure to the inhibitor, the film repair is continuous.

3.1.5 *emulsification-tendency*—a property of an inhibitor that causes the water and hydrocarbon mixture to form an emulsion. The emulsion formed can be quite difficult to remove and this will lead to separation difficulties in the production facilities.

3.1.6 *film persistency*—ability of inhibitor film (usually batch inhibitor) to withstand the forces (for example, flow) that tend to destroy the film over time.

3.1.7 *flow loop*—an experimental pipe that contains various corrosion probes to monitor corrosion rates. A flow loop can be constructed in the laboratory or attached to an operating system.

3.1.8 *foaming tendency*—tendency of inhibitor in solution (water or hydrocarbon) to create and stabilize foam when gas is purged through the solution.

3.1.9 *gas to oil ratio (GOR)*—ratio of the amount of gas and oil transported through a pipe over a given time.

3.1.10 *high-pressure*—a pressure above ambient atmospheric pressure that cannot be contained in normal laboratory glassware. Typically, this is greater than 0.07 MPa (10 psig).

3.1.11 *high-temperature*—temperatures above ambient laboratory temperature where sustained heating of the environment is required.

3.1.12 *laboratory methodology*—a small laboratory experimental set up, that is used to generate the corrosion. Examples of laboratory methodologies include rotating cylinder electrode (RCE), rotating cage (RC), and jet impingement (JI) under flowing conditions.

3.1.13 *live water*—aqueous solution obtained from a pipeline or well. Usually live water is protected from atmospheric oxygen.

3.1.14 *mass transfer coefficient (k, m/s)*—the rate at which the reactants (or products) are transferred to the surface (or removed from the surface).

3.1.15 *measuring technique*—technique for determining the rate of corrosion and the inhibitor efficiency. Examples of measuring techniques are mass loss, linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS), electrical resistance (ER), and potentiodynamic polarization (PP) methods.

3.1.16 *multiphase flow*—simultaneous passage or transport of more than one phase, where the phases have different states (gas, liquid, and solid) or the same state (liquid), but different fluid characteristics (viscosity, density, and specific gravity).

3.1.17 *synthetic water*—a synthetic solution prepared in the laboratory using various chemicals. The composition is based on the composition of fluid found in an oil production system.

3.1.18 *Schmidt Number (Sc)*—a measure of the ratio of the hydrodynamic boundary layer to the diffusion boundary layer. This dimensionless parameter is equal to kinematic viscosity divided by diffusion coefficient.

3.1.19 *wall shear stress ( $\tau$ , N/m<sup>2</sup>)*—a force per unit area on the pipe due to fluid friction.

3.2 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology D 4410 or G 15. Definitions provided herein and not given in Terminology D 4410 or G 15 are limited only to this guide.

## 4. Summary of Guide

4.1 Inhibitor evaluation in the laboratory consists of two steps (1) evaluation of inhibitor efficiency and (2) evaluation of secondary inhibitor properties.

4.2 Four laboratory methodologies, flow loop, rotating cylinder electrode (RCE), rotating cage (RC), and jet impingement (JI) are available to evaluate the inhibitor efficiency in the laboratory. All four methodologies can be operated at atmospheric and high pressure conditions. The corrosion rates can be measured using mass loss or electrochemical methods. Using the methodologies, several variables, compositions of material, composition of environment (gas and liquid), temperature, pressure, and flow, that influence the corrosion rate in the field can be simulated in the laboratory. Rotating cylinder electrode (RCE), rotating cage (RC), and jet impingement (JI) methodologies are compact, inexpensive, hydrodynamically characterized, and scalable; that is, can be carried out at various flow conditions.

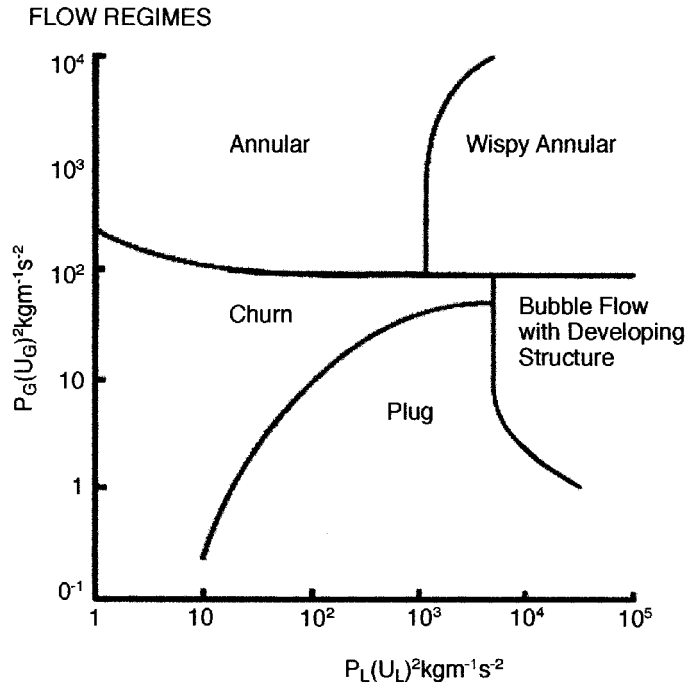
4.3 Several secondary properties of the inhibitor are evaluated before the inhibitor is applied in the field. They are water/oil partitioning, solubility, emulsification tendency, foam tendency, thermal stability, toxicity, and compatibility with other additives/materials. Laboratory methods to evaluate the secondary properties are described.

## 5. Significance and Use

5.1 Corrosion inhibitors continue to play a key role in controlling internal corrosion associated with oil and gas production and transportation. This results primarily from the industry's extensive use of carbon and low alloy steels, which, for many applications, are economic materials of construction that generally exhibit poor corrosion resistance. As a consequence, there is a strong reliance on inhibitor deployment for achieving cost-effective corrosion control, especially for treating long flowlines and main export pipelines (1).<sup>7</sup>

5.2 For multiphase flow, the aqueous-oil-gas interphases can take any of an infinite number of possible forms. These forms are delineated into certain classes of interfacial distribution called flow regimes. The flow regimes depend on the inclination of the pipe (that is, vertical or horizontal), flow rate (based on production rate), and flow direction (that is, upward or downward). The common flow regimes in vertical upward flow, vertical downward flow, and horizontal flow are presented in Figs. 1-3 respectively (2, 3).

<sup>7</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.



NOTE— $\rho_G$  and  $\rho_L$  are gas and liquid densities and  $U_L$  and  $U_G$  are superficial velocities or the volume of flow rates of the liquid and gas per unit cross-sectional area of the channel.(2)

FIG. 1 Flow Regimes for Vertical Upward Multiphase Flow

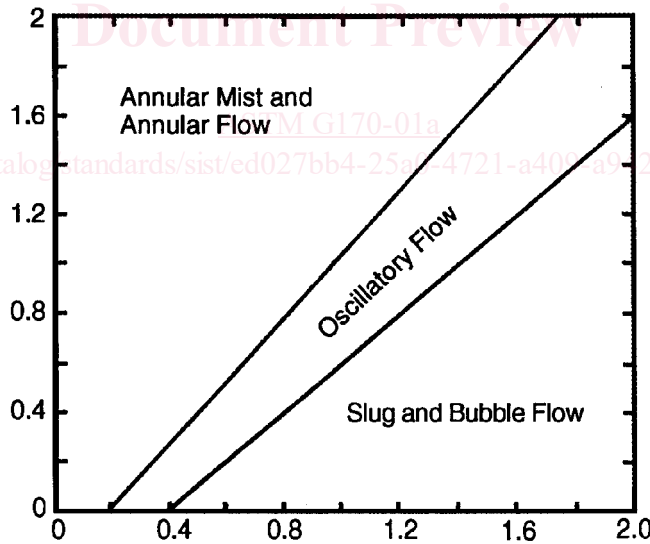
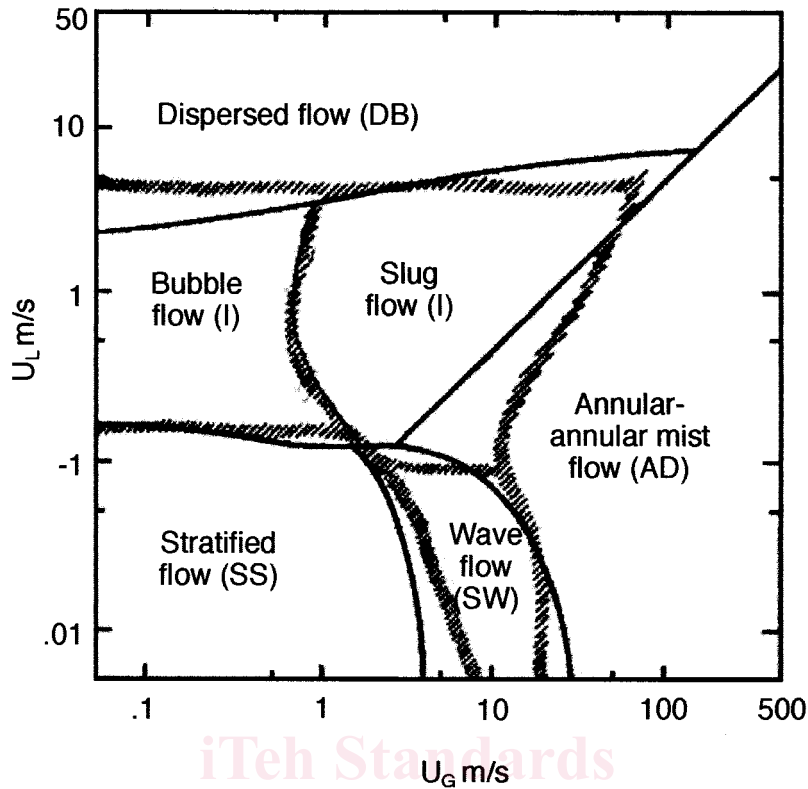


FIG. 2 Flow Regimes for Vertical Downward Flow (2)

5.3 Depending on the flow regime, the pipe may undergo various forms of corrosion, including general, localized, flow-induced, and erosion-corrosion. One of the predominant failure mechanisms of multiphase systems is pitting corrosion.

5.4 The performance of a corrosion inhibitor is influenced primarily by the nature of inhibitor, operating conditions of a system, and the method by which it is added. Two types of inhibitors are used in the oil field, continuous and batch. Water-soluble and oil-soluble, water-dispersible inhibitors are

added continuously. Oil-soluble inhibitors are, in general, batch treated. The test methods to evaluate the inhibitors for a particular field should be carried so that the operating conditions of the system are simulated. Thus during the evaluation of a corrosion inhibitor, an important first step is to identify the field conditions under which the inhibitor is intended to be used. The environmental conditions in the field locations will dictate the laboratory conditions under which testing is carried out.



NOTE—Boundary conditions given by two studies are presented.(2)

FIG. 3 Flow Regimes for Horizontal Flow

5.5 Various parameters that influence corrosion rates, and hence, inhibitor performance in a given system are (1) composition of material (2) composition of gas and liquid (3) temperature (4) flow and (5) pressure.

5.5.1 In order for a test method to be relevant to a particular system, it should be possible to control the combined effects of various parameters that influence corrosion in that system. A test method is considered to be predictive if it can generate information regarding type of corrosion, general and localized corrosion rates, nature of inhibition, and life of inhibitor film (or adsorbed layer). Rather than try to perfectly reproduce all the field conditions, a more practical approach is to identify the critical factors that determine/impact inhibitor performance and then design experiments in a way which best evaluates these factors.

5.6 Composition of material, composition of gas and liquid (oil and water), temperature, and pressure are direct variables. Simulation of them in the laboratory is direct. Laboratory experiments are carried out at the temperature of the field using coupons or electrodes made out of the field material (for example, carbon steel). The effect of pressure is simulated by using a gas mixture with a composition similar to the field for atmospheric experiments and by using partial pressures similar to those in the field for high pressure experiments.

5.7 In multiphase systems there are three phases, oil, aqueous (brine water), and gas. Corrosion occurs at places where the aqueous phase contacts the material (for example, steel). The corrosivity of the aqueous phase is influenced by the

composition and the concentration of dissolved gases (for example,  $H_2S$  and  $CO_2$ ). In evaluating corrosion inhibitors in the laboratory, aqueous phase is usually used with a positive pressure of gas mixture to simulate the gaseous phase. The oil may have a major effect on the corrosion rate and inhibitor efficiency. The presence of oil phase in the test environment can have significantly different effects (4). The primary effect of the oil phase is apparently on the protectiveness of the corrosion inhibitor. The oil phase may have the following effects: (1) partitioning of inhibitor between phases (2) changing the contact time of the aqueous phase on the pipe (3) changing the wetting behaviour of the pipe surface (4) introducing protective compounds that are naturally occurring in the oil.

5.7.1 Inhibitor evaluation in the absence of the oil phase cannot give an accurate picture of the behaviour of steel in multiphase environments. Ideally, the oil phase should be present when testing the inhibitor in the laboratory.

5.8 Flow is an indirect variable, and simulation of flow in the laboratory is not direct. For this reason, the hydrodynamic flow parameters are determined, and then the laboratory corrosion tests are conducted under the calculated hydrodynamic parameters. The fundamental assumption in this approach is that, when the hydrodynamic parameters of different geometries are the same, then the corrosion mechanism will be the same. Under these conditions, the corrosion rate and the efficiency of corrosion inhibition in the laboratory and in the

field are similar. The commonly used hydrodynamic parameters are wall shear stress, Reynolds number, and mass transfer coefficient (3, 5).

5.9 Neither the flow rate (m/s) nor dimensionless parameters can be directly related to the local hydrodynamic forces at the material surface that may be responsible for accelerated localized attack. Local hydrodynamic forces are influenced by several factors including pipe inclination, position (that is, 3, 6, 9 o'clock), presence of bends, deposits, edges, welds, expansion, and contraction. The flow rate and dimensionless parameters describe only bulk, or average, properties of the dynamic system. Thus the wall shear stress and mass transfer coefficient can be calculated only as averages at the surface with an average surface roughness.

5.10 Inhibitors are first screened in the laboratory, then evaluated in the field, and finally used in engineering operations. The laboratory methodologies, therefore, should be carried out in a compact system with the capacity to evaluate various products quickly with the flow pattern and regime characterized. The results obtained should be relevant to field operation, should be predictive of field performance in terms of inhibitor efficiency, and should be scalable, that is, the experiments can be carried out at various hydrodynamic conditions.

5.11 Flow loops are used to evaluate corrosion inhibitors either in the laboratory or by attaching to a live pipe. The loop simulates the flow regime, but the apparatus is relatively sophisticated, and experiments are expensive and time consuming. The loop is considered sophisticated to be an ideal laboratory methodology under the scope of this guide.

5.12 This guide discusses test facilities and considers the necessary elements which need to be built into a laboratory strategy for testing corrosion inhibitors for multiphase systems. The emphasis is on those methodologies that are compact and scalable, hydrodynamically well characterized, and relatively inexpensive to use. The laboratory methodologies are (1) rotating cylinder electrode (RCE) (2) rotating cage (RC) and (3) jet impingement (JI). These methodologies can be used under both atmospheric and high pressure conditions. Detailed description of RCE and JI are presented in NACE-5A195.

5.13 Laboratory tests for inhibitor evaluation consist of two main components—laboratory methodology and measurement technique. The combinations of laboratory methodology and measurement technique for inhibitor evaluation for multiphase systems are presented in Table 1.

5.14 To develop an inhibitor selection strategy, in addition to inhibitor efficiency, several other key performance factors need to be evaluated: (1) water/oil partitioning (2) solubility (3) emulsification tendency (4) foaming tendency (5) thermal stability (6) toxicity (7) compatibility with other additives/materials.

## 6. Preparation of Test Solutions

6.1 Ideally, all solutions (oil and aqueous) should be obtained from the field for which the inhibitor is being evaluated. It is important that live fluids do not already contain corrosion inhibitor. In the absence of live fluids, synthetic solutions should be used, the composition of which, however, should be based on field water analysis. Alternatively, standard brine (in accordance with Practice D 1141) should be employed. The solutions should be prepared following good laboratory practice. Their composition should be specified in the work plan and recorded in the laboratory logbook. Test solutions should be prepared using analytical grade reagents and deionized water, unless otherwise specified. If other grades of chemicals are used, their purity or grade should be recorded in the laboratory logbook.

6.2 The solutions should be deaerated by passing nitrogen (or any other inert gas) or carbon dioxide and kept under deaerated conditions. The solution pH before and after testing should be measured and recorded. If possible, the solution pH should be monitored continuously during the test. Solutions should be transferred from the preconditioning vessel to the test vessel under positive nitrogen pressure to minimize air contamination during the transfer operation.

6.3 The appropriate composition of gas can be obtained by mixing H<sub>2</sub>S and CO<sub>2</sub> streams from the standard laboratory gas supply. Nitrogen can be used as a diluent to obtain the required partial pressures 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, should be kept as low as technically possible (below 5 ppb, preferably under 1 ppb oxygen in solution). The solution oxygen concentration depends on the quality of gases used to purge the electrolyte.

6.4 Measure Inhibitor concentrations and report in % weight/volume or ppm w/v (percentage or parts per million, weight in volume basis). The method of injecting the inhibitor into the test solution should reflect the actual field application that is being tested. 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 will depend on the characteristics of the inhibitor and on the specified test conditions.

6.5 Oil-soluble, water-dispersible inhibitor solutions are prepared by the partition method. Place the required amounts of crude oil, or condensate, and brine in the partitioning vessel (usually a separation funnel). The relative volumes of hydrocarbon and aqueous phases reflect the water cut to be tested. If actual field condensate is not available, heptane, kerosene, or any suitable hydrocarbon can be used as a substitute for the oil

**TABLE 1 Laboratory Methodologies and Measurement Techniques for Corrosion Inhibitor Evaluation**

Laboratory Methodology	Measurement Technique	Aqueous/Oil/ Gas Phase	Remarks
RCE	mass loss, electrochemical	aqueous phase	specimen is a cylinder
RCE	mass loss	aqueous/oil phase	specimen is a cylinder
JI	mass loss, electrochemical	aqueous phase	specimen is a disc
JI	mass loss	aqueous/oil phase	specimen is a disc
JI	electrochemical measurements	aqueous phase	specimen is a ring
RC	mass loss	aqueous phase or aqueous/oil phase	electrochemical measurements cannot be carried out

phase. Add the corrosion inhibitor to the oil phase. Vigorously shake the vessel to mix both phases thoroughly and allow the phases to separate. Heating to the minimum expected field temperature may help in the separation and will also provide more meaningful results; remove the aqueous phase and use as the test solution.

6.6 Oil-soluble inhibitors (usually as batch inhibitors) are applied in a separate procedure and the corrosion test is carried out after this. The inhibitor is dissolved in the oil phase to form an inhibited oil-phase. The corrosion coupon or electrode is exposed to this solution for a certain amount of time (usually 30 min). The coupon or electrode is then removed and introduced into the experimental vessel for the corrosion test.

6.7 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. Exercise precaution to avoid or minimize the temperature differentials. The test vessels should be heated slowly to avoid overheating and, in the case of glass autoclaves, to prevent high thermal stresses between the inner and outer walls. The exact protocol followed will depend 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. The pressure in the vessel should be monitored during heating to make sure it does not exceed the relief pressure. If necessary, some of the gas in the vessel may be bled off to reduce the pressure. The test temperature should be maintained within 2°C of the specified temperature. Once the test temperature is reached, the test pressure should be adjusted to the predetermined value. The pressure should be maintained within  $\pm 10\%$  of the specified value for the duration of the test.

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

6.9 For high-temperature, high-pressure experiments (6) using individual gases, first pressurize the autoclave with H<sub>2</sub>S to the required partial pressure. Leave it for 10 min. If there is a decrease of pressure, repressurize the autoclave again. Repeat the process until no further pressure drop occurs. Then, pressurize the autoclave with CO<sub>2</sub>, by opening the CO<sub>2</sub> gas cylinder at a pressure equal to the CO<sub>2</sub> + H<sub>2</sub>S partial pressure. Leave it for 10 min. If there is a decrease of pressure, repressurize the autoclave again with CO<sub>2</sub> gas. Repeat the process until no further pressure drop is observed. Finally, pressurize the autoclave with the inert gas, by opening the inert gas cylinder at the total gas pressure at which the experiments are intended to be carried out.

## 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 specimen should be made of the material (for example, carbon steel) for which the inhibitor is being evaluated. Corrosion rates and inhibitor performance change by several orders of magnitude as surface roughness changes from rough to fine. The surface roughness should be kept the same during inhibitor screening and, if possible, the surface roughness of specimens used in the laboratory experiments should be related to that of field pipe. The specimens should be ground to a specified surface finish. 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 wiped off using tissue paper.

7.3 Rinse the specimens with distilled water and then degrease the specimens by immersing in acetone (or methanol) and ultrasonically cleaning for 1 min; dry the specimens with a paper towel. Do not touch the surface of the specimens with bare hands; and weigh the specimens to the nearest 0.1 mg. Measure the dimensions of the specimens to the nearest 1 mm, and calculate the area of each specimen.

7.4 In general, specimens are held in an insulating specimen holder; the type of holder varies with the test. Install the freshly prepared specimens in the synthetic materials holder and tighten them. Place the specimen holder in the vessel, and close the lid. Fill the vessel with the preconditioned (deaerated) test solution and continue deaerating for at least 1 h using nitrogen.

## 8. Laboratory Methods for Evaluating Inhibitor Efficiency

### 8.1 Rotating Cylinder Electrode (RCE):

8.1.1 The RCE test system is compact, relatively inexpensive, and easily controlled (7). It operates in the turbulent regime over a wide range of Reynolds numbers. The apparatus operates under known and controlled hydrodynamic conditions. The experiments require small amounts of fluid, and mass loss and electrochemical measurements can be made. General procedures for specimen preparation, methods of cleaning, corrosion rate measurements and evaluation of results are described in detail in Guide G 16, Practices G 31, G 102, and G 106, Test Method G 59, and NACE-1D196.

8.1.2 At very low electrode rotation speeds, the flow around the RCE is laminar and occurs in concentric circles around the cylinder. At higher rotation speeds this simple flow pattern becomes unstable. Cellular motion is imposed on the flow producing toroidal Taylor vortices containing a radial component of velocity, but the bulk of the flow remains essentially laminar. As rotational speeds increase further, the flow becomes fully turbulent and eddies increasingly break up the regular flow pattern. The transition to fully turbulent flow occurs at about Re 200. In the turbulent flow region, the RCE can be applied to simulate flow behavior by hydrodynamic analysis.

8.1.3 A typical 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  $\pm 2$  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 constant. At the side of the sample holder, electrical connections to the electrodes are made by a brush or mercury contact. The cylinder geometry is