Designation: G202 – 12 (Reapproved 2016)

Standard Test Method for Using Atmospheric Pressure Rotating Cage[1]

This standard is issued under the fixed designation G202; 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 test method covers a generally accepted procedure to conduct the rotating cage (RC) experiment under atmospheric pressure.

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]

D1141 Practice for the Preparation of Substitute Ocean Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
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
G170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory
G184 Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using Rotating Cage

3. Significance and Use

3.1 The rotating cage (RC) test system is relatively inexpensive and uses simple flat specimens that allow replicates to be run with each setup. (**1-11**).[3]

3.2 The RC method can be used to evaluate either corrosion inhibitors, or materials, or both. Guide G184 describes the procedure to use rotating cage to evaluate corrosion inhibitors.

3.3 In this test method, 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, and flow. Oil field fluids may often contain sand; however, this test method does not cover erosive effects that occur when sand is present.

4. Apparatus

4.1 Fig. 1 shows the schematic diagram of the RC system. The vessel is manufactured from acrylic. At the bottom of the container, a PTFE base is snugly fitted. At the center of the base, a hole is drilled, into which the lower end of the rotating shaft is placed. This arrangement stabilizes the rotating shaft and the coupons. The length of the rotating shaft between the top and bottom covers is 40 cm (15.7 in.). The rotating cage is attached to the shaft in such a way that the top of the cage is 30 cm (11.8 in.) from the bottom cover.

4.2 Eight coupons (each of length 75 mm, width 19 mm, thickness 3 mm, and surface area 34.14 cm^2)) are supported between two PTFE disks (of 80-mm diameter) mounted 75 mm apart on the stirring rod (Fig. 2). Holes (diameter 10 mm) 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 rotation speeds up to 1000 rpm.

4.3 Flow patterns inside the RC depend on the rotation speed, the volume of the container, and the nature of the fluids used. The flow patterns are described in Guide G170.

4.4 Volume of solution to the surface area of the specimen has some effect on the corrosion rate. The minimum solution volume (cm^3) to metal surface area (cm^2) is not less than 14 cm (cm^3/cm^2) (**10**).

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[1] This test method 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 a list of references at the end of this standard.

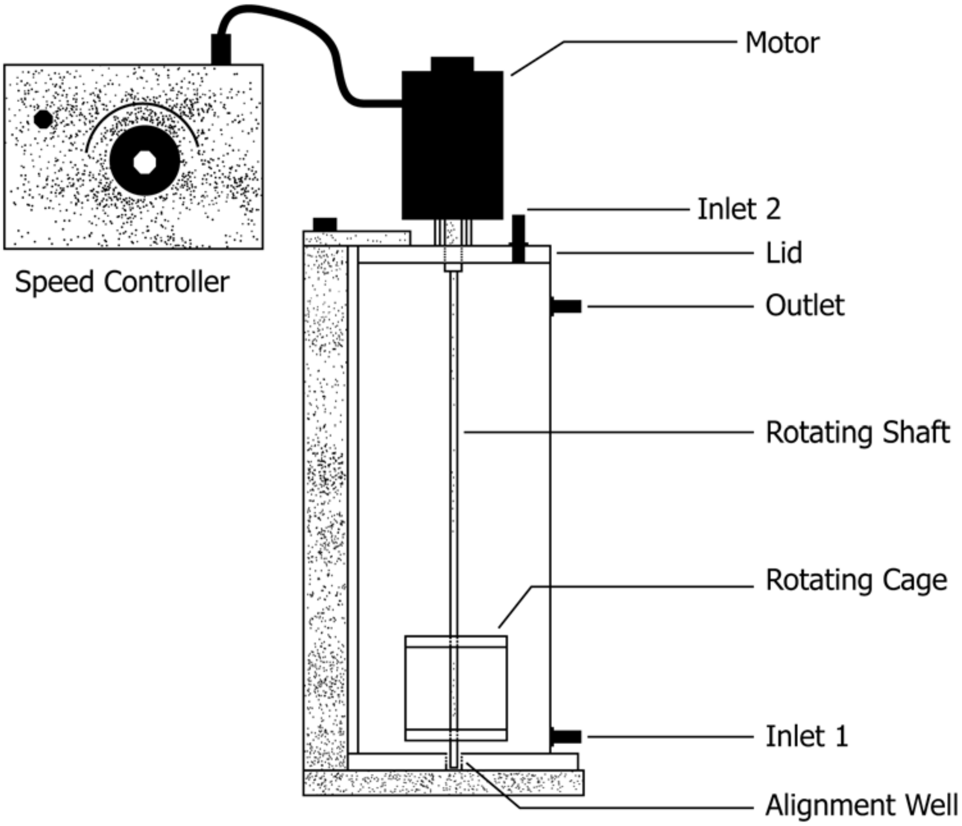

FIG. 1 Schematic Diagram of Rotating Cage

NOTE 1—Gaps (typically 0.85 ± 0.01 cm) between the coupons introduce localized turbulence.

FIG. 2 Photo of Rotating Cage Containing Coupons

5. Reagents

5.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 The composition of the solution shall be determined and reported. Alternatively, standard brine (such as in Practice D1141) shall be used. The solutions shall be prepared using analytical grade reagents and deionized water (in accordance with Specification D1193).

5.3 The solutions shall be deoxygenated by passing nitrogen or any other inert gas for sufficient time to reduce the oxygen content below 5 ppb. The solution shall 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.

5.4 **Warning—**Hydrogen sulfide (H_2S) and carbon dioxide (CO_2) are corrosive gases. H_2S is poisonous and shall not be released to the atmosphere. The appropriate composition of gas can be obtained by mixing H_2S and CO_2 streams from the

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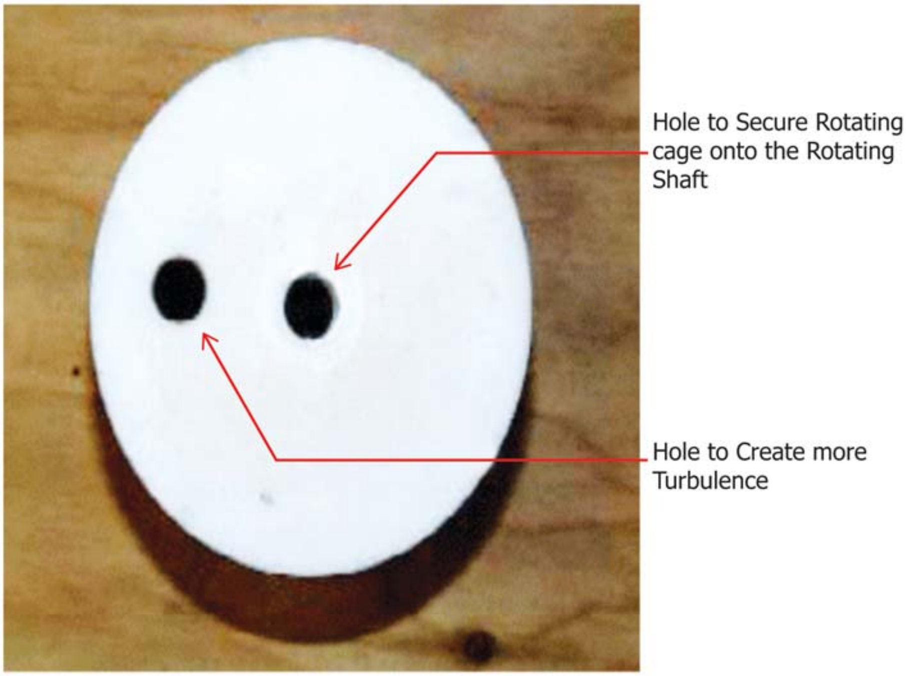
[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



NOTE 1—Holes (typically 1.0 cm diameter, about 1.5 cm from the center) introduce localized turbulence

FIG. 3 Photo of Rotating Cage (Top View)

standard laboratory gas supply. Nitrogen 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 below 5 ppb.

5.5 The solution pH before and after testing shall be measured, recorded, and reported (in accordance with Test Methods D1293).

6. Test Specimens

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

6.2 The coupon shall have the same metallographic structure as that used in the service components. The coupons shall be ground to a surface finish of 150 grit. The grinding shall produce a reproducible surface finish with no rust deposits, pits, or deep scratches. All sharp edges on the coupon shall be ground. All loose dirt particles shall be removed.

6.3 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 shall 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.

6.4 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.

7. Procedure

7.1 A detailed procedure to determine corrosion rates from mass loss is described in Practice G31.

7.2 Solutions are prepared and presaturated with the experimental gas mixture. If the solution is prepared in a separate container, it shall be transferred from the preparation vessel to the experimental vessel under positive nitrogen pressure to minimize air contamination during the transfer operation.

7.3 The experiment shall be conducted at room temperature (21 to 24°C).

7.4 The pre-weighed coupons and holder (described in 4.2) are inserted into the apparatus.

7.5 The lid of the apparatus is sealed such that oxygen cannot leak into the system through the lid.

7.6 Initially all ports of the experimental vessel (Inlet 1, Inlet 2, and outlet) are closed. A nitrogen gas (or any other inert gas) cylinder is hooked up to Inlet 2. The outlet is hooked to a gas bubbler or gas trap which allows only one way flow of gas (flowing out of the apparatus). Both Inlet 2 and the outlet are opened allowing the nitrogen gas to pass through the apparatus. The apparatus shall be deoxygenated by passing nitrogen for a minimum of 1 h/L of internal volume to reduce the oxygen content below 5 ppb.

7.7 Inlet 1 is hooked up to the container of the prepared deoxygenated solution. Inlet 2 is closed and Inlet 1 is opened. The deoxygenated solution is pumped into the apparatus without allowing the entry of oxygen. Inlet 1 is closed.

7.8 The experimental gas mixture is hooked up to Inlet 2. Inlet 2 is opened allowing the experimental gas mixture to enter the apparatus. A continuous flow of gas shall be maintained through the apparatus (entering Inlet 2 and exiting the outlet) throughout the experiment in order to avoid oxygen contamination. Precautions shall be taken so that the gas does not entrain with the solution.

7.9 The speed controller is used to set the rotation speed and start the motor.

7.10 The experiment is terminated (after 24 h), and the corrosion rate is determined from the amount of mass loss in accordance with Practices G1 and G31. The samples are examined and evaluated for pitting corrosion in accordance with Guide G46. The average, standard deviation, and coefficient of variation of the coupons' corrosion rate for each run shall be calculated using the method presented in Guide G16. If pitting corrosion is observed, then the general corrosion rate determined from mass loss could be invalid.

8. Report

8.1 All information and data shall be recorded as completely as possible. Practice G31 provides a checklist for reporting corrosion data.

8.2 Average corrosion rates and the standard deviation at each rotation rate shall be reported.

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

8.3.1 Solution chemistry and concentration (any changes during test);

8.3.2 Volume of test solution;

8.3.3 Volume of the experimental vessel;

8.3.4 Duration of the test;

8.3.5 Chemical composition or tradename of metal;

8.3.6 Number, form, and metallurgical conditions of specimen;

8.3.7 Exact size, shape, and area of each specimen;

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

8.3.9 Initial and final masses and actual mass losses; and

8.3.10 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.

TABLE 1 Repeatability Statistics[A]

Item	Unit	Ave	s_r	r
C_{ave}	mpy	23.1	4.7	13.3
SD	mpy	1.76	1.12	3.11
C_{ave}	mm/yr	0.587	0.119	0.334
SD	mm/yr	0.045	0.028	0.079
CV_r	%	7.7	4.9	13.7

[A] It should be noted that the SD and CV values cannot be negative so that the limits on these values range from zero to the sum of the average value plus the repeatability or reproducibility value.

9. Precision and Bias[5]

9.1 *Precision*—The precision of this test method was determined by an interlaboratory test study, ILS, with seven laboratories participating. The results of this program were analyzed using Practice E691. Three other laboratories submitted data that was found to be unsuitable for a variety of reasons.

9.1.1 *Repeatability*—The repeatability, r, (within laboratory variation) and the repeatability standard deviation, s_r, were determined from the results of the seven laboratories participating in the ILS. The results included in this analysis were the average corrosion rate based on the eight specimens in the cage, C_{ave}, the standard deviation of these eight results, SD, and the coefficient of variation of the data set, CV_r. These results are summarized in Table 1.

9.1.2 *Reproducibility*—The reproducibility, R, (between laboratory variation), the reproducibility standard deviation, s_R, and the reproducibility coefficient of variation, CV_R, were also determined from the ILS. These results are summarized in Table 2.

9.1.3 *Bias*—This test method has no bias because the (property measured) is defined only in terms of this test method.

10. Keywords

10.1 laboratory evaluation; mass loss; rotating cage (RC)

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G01-1025. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Reproducibility Statistics[A]

Item	Unit	Ave	s_R	R
C_{ave}	mpy	23.1	5.4	15.2
SD	mpy	1.76	1.59	4.44
C_{ave}	mm/yr	0.587	0.138	0.387
SD	mm/yr	0.045	0.040	0.113
CV_R	%	7.7	6.5	18.3

[A] It should be noted that the SD and CV values cannot be negative so that the limits on these values range from zero to the sum of the average value plus the repeatability or reproducibility value.

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