



Designation: G 180 – 04

Standard Test Method for Initial Screening of Corrosion Inhibiting Admixtures for Steel in Concrete¹

This standard is issued under the fixed designation G 180; 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 describes a procedure for determining the effects of chemical admixtures on the corrosion of metals in concrete. This test method can be used to evaluate materials intended to inhibit chloride-induced corrosion of steel in concrete. It can also be used to evaluate the corrosivity of admixtures by themselves or in a chloride environment. This test is not applicable for emulsions.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

- C 150 Specification for Portland Cement
- C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials
- D 632 Specification for Sodium Chloride
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G 5 Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements
- G 15 Terminology Relating to Corrosion and Corrosion Testing
- G 59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

¹ This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.14 on Corrosion of Metals in Concrete, Mortar, or Cement.

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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. Terminology

3.1 *Definitions*—For definitions of terms used in this practice see Terminology G 15.

4. Significance and Use

4.1 This test method provides a means for assessing corrosion-inhibiting concrete admixtures.

4.2 This test method is useful for development of admixtures intended to reduce corrosion of reinforcing steel in concrete.

4.3 This test method is useful in determining the corrosivity of admixtures toward steel reinforcing if the admixture sample is compared to a control without admixtures.

4.4 Good performance, a reduction in corrosion rate versus chloride alone by at least one order of magnitude in this test, is a strong indication that an admixture is a corrosion inhibitor. However, poor performance requires additional testing to determine if the admixture improves corrosion resistance.

4.5 This method is a screening test and shall not be used to predict performance in the field. However, it is useful to determine which admixtures shall be included into long-term evaluation programs.

4.6 The filtering process makes this test not suitable for the evaluation of emulsions.

5. Apparatus

5.1 The test cell as described in Test Method G 5.

5.2 Potentiostat, as described in Test Method G 5, capable of varying potential at a constant scan rate and measuring the resulting current.

5.3 A method of recording the varying potential and resulting current is needed.

5.4 Electrode holder such as described in Fig. 3 of Test Method G 5.

5.5 Electrodes:

5.5.1 Working electrode, prepared from a 12.7 mm length of 9.5 mm diameter rod stock. Carbon steel C1215 should be used.

NOTE 1—If specimen forms are used other than those called for by this test method, for example flat sheet specimens, care should be taken not to introduce crevices which can lead to erroneous results.

5.6 Auxiliary Electrodes:

5.6.1 Two graphite rods or platinized-niobium or platinum mesh.

5.7 Reference Electrodes:

5.7.1 A saturated calomel electrode with a controlled rate of leakage (about 3 $\mu\text{L/h}$) is recommended.

6. Reagents and Materials

6.1 Type I/II cement (C3A content between 6 and 10 %), according to Specification C 150.

6.2 Filter paper with 1.1 μm retention.

6.3 Teflon stir bars.

6.4 Carbon steel C1215 samples.³

6.5 Sodium chloride, reagent grade, according to Specification D 632.

6.6 Calcium hydroxide, reagent grade.

6.7 Admixtures to be tested.

6.8 Carbon dioxide free compressed air.⁴

7. Experimental Procedure

7.1 Prepare a cement slurry consisting of 1000 g of water and 200 g cement. Mix thoroughly, stir for 60 min and filter.

NOTE 2—An admixture should be added at a quantity consistent with its addition rate in concrete. 35 to 965 mL of water is equivalent to 5 L/m³ in concrete. If other dosages are desired, proportion them based on this ratio.

7.2 Filter, and add 4 g/L calcium hydroxide and stir a further 30 min.

7.3 Setup a standard electrochemical cell according to Test Method G 5 and fill it with 900 mL of filtered slurry solution. Purge the cell with carbon dioxide free air. Air flow rate should be at least 300 cc/min.

7.4 Degrease the metal sample by cleaning ultrasonically in hexane for 2 min. If an ultrasonic bath is not available, soak the samples in hexane and wipe dry. Make sure the sample is thoroughly dried before mounting it on the electrode holder.

7.5 While purging the cell with carbon dioxide free air, precondition the electrode in the solution for 24 h.

7.5.1 Add NaCl to the solution obtained in 7.3 (having been purged for 24 h with CO₂-free air), to obtain a 0.5 or a 1M solution, and continue purging for a further 4 h.

NOTE 3—The multi-laboratory test was run at the two different chloride levels to develop the precision statement. The higher chloride level would be representative of a more severe exposure.

7.5.2 Measure the open circuit potential.

7.5.3 Measure the polarization resistance (R_p) by recording the potentiodynamic polarization curve at a scan rate of 0.167 mV/s, from -20 mV to +20 mV versus open circuit potential.

7.5.4 Plot the polarization resistance curve as a linear potential-current density plot as shown in Practice G 3.

7.5.5 Determine the polarization resistance R_p , as the tangent of the curve at $i = 0$, as described in Test Method G 59. The corrosion rate is expressed as $1/R_p$ in $\mu\text{S/cm}^2$.

³ The samples are cylindrical in shape, with 5.1 cm² exposed area, and can be purchased from Metal Samples, AL, sample type P/N410.

⁴ A CO₂ free air gas generator (typically used for FT-IR equipment) can be used.

NOTE 4—An example of a polarization resistance curve is given in the Appendix as Fig. X1.4.

8. Interpretation of Results

8.1 An admixture is behaving as a corrosion inhibitor in this test method if the average $\log_{10}(1/R_p)$ value is 1.0 or less than that of the chloride only average.

8.2 If the admixture does not reduce average $1/R_p$ by an order of magnitude another test method is needed to determine if it is an inhibitor.

8.3 An admixture that increases average $1/R_p$ by an order of magnitude over a slurry without chloride or inhibitor is corrosive.

NOTE 5—The change in $\log_{10}(1/R_p)$ by 1.0 is an order of magnitude change in $1/R_p$. Log values are useful in comparing corrosion rates since rates from different specimens or conditions can differ by orders of magnitude making a linear scale less useful.

9. Report

9.1 Report the following information:

9.1.1 Value of the open circuit potential (OCP) versus SCE, and

9.1.2 Corrosion rate given by $1/R_p$ in $\mu\text{S/cm}^2$.

10. Precision and Bias

10.1 Based on the pooled estimates of precision, the following statement of precision and bias⁵ can be made:

10.1.1 *Interlaboratory Test Program*—An interlaboratory study of a pore solution test for corrosion inhibiting admixtures for steel reinforcement in concrete was conducted in 2001. Each of six laboratories tested two randomly drawn samples of each of four materials (two sodium chloride solutions, 0.5M and 1.0M, each with and without 35 mL/L of a solution containing 30 % calcium nitrite). Practice E 691 was followed for the design and analysis of the study.

10.1.2 *Single-Operator Precision*—The single operator standard deviation of the logarithm to the base 10 of a single test result is 0.36. Therefore, $\log_{10}(1/R_p)$ values of two properly conducted tests by the same operator should not differ by more than 1.0.

10.1.3 *Interlaboratory Precision*—The interlaboratory standard deviation of the logarithm to the base 10 of a single test result is 0.44. Therefore, $\log_{10}(1/R_p)$ values of two properly conducted tests in different laboratories should not differ by more than 1.24.

NOTE 6—These numbers represent, respectively, the (1s) and (2ds) limits as described in Practice C 670.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

11. Keywords

11.1 admixtures; cement; corrosion inhibitors; slurry; solution

⁵ Supporting data have been filed at ASTM Headquarters. Request RR:G01-1021.

APPENDIX

(Nonmandatory Information)

X1. PRECISION CALCULATIONS

X1.1 Information on the precision of the results obtained by this test method was derived from an interlaboratory test with two samples for each molarity tested, per laboratory. Six laboratories participated in the study. Statistical analysis of the data was performed using Practice E 691 Interlaboratory Data Analysis Software. Results are shown in the attached Table A for $1/R_p$ measurements. Values of repeatability (S_r , r), and reproducibility (S_R , R) are for the logarithm (base 10) of $1/R_p$, where S_r , r , S_R , and R are defined as follows:

- S_r = repeatability standard deviation [$\log(\mu\text{S}/\text{cm}^2)$] (1s),
- r = 95 % repeatability limit within a laboratory [$\log(\mu\text{S}/\text{cm}^2)$] (2ds),
- S_R = reproducibility standard deviation [$\log(\mu\text{S}/\text{cm}^2)$] (1s), and
- R = 95 % reproducibility limit between laboratories [$\log(\mu\text{S}/\text{cm}^2)$] (2ds).

X1.2 This study meets the minimum requirements for determining precision prescribed in Practice E 691 in terms of the number of laboratories, materials and determinations (see Appendix X1, Table X1.1).

X1.3 Fig. X1.1 is a graph of r and R versus average [$\log_{10}(1/R_p) + 1$]. Because the ASTM data analysis software does not accept negative numbers, a value of 1 was added to every $\log_{10}(1/R_p)$ value. Adding a constant to all values does not change the standard deviation estimates. It does, however, change the calculated averages. In Table X1.1, the average values were corrected by subtracting 1 from the software output. In Fig. X1.1, the average values were not corrected. There is no indication that precision varies systematically with average level. Therefore, pooled estimates of precision were determined which should be valid over the range of $1/R_p$ encompassed in the interlaboratory study.

X1.4 The consistency statistics for $\log_{10}(1/R_p)$ by laboratory and by material, are shown in Figs. X1.2 and X1.3,

TABLE X1.1 Statistical Analysis of Corrosion Potential Data Practice E 691 Interlaboratory Data Analysis Software

The number of laboratories, materials, and determinations in this study DOES meet the minimum requirements for determining precision prescribed in Practice E 691:

| | This Study | Practice E 691 Minimum |
|----------------|------------|------------------------|
| Laboratories | 6 | 6 |
| Materials | 4 | 3 |
| Determinations | 2 | 2 |

Precision Statement for Test Method
Precision and Reproducibility of $\text{Log}_{10}(1/R_p)$ for Each Material
Precision, characterized by repeatability (S_r , r) and reproducibility (S_R , R) has been determined for the materials to be:

| Material | Average | S_r | S_R | r | R |
|--|---------|-------|-------|------|------|
| (All dimensions are $\text{Log}_{10}(\mu\text{S}/\text{cm}^2)$) | | | | | |
| 0.5M NaCl | 1.65 | 0.38 | 0.59 | 1.05 | 1.64 |
| 0.5M NaCl + $\text{Ca}(\text{NO}_2)_2$ | 0.42 | 0.34 | 0.40 | 0.95 | 1.11 |
| 1.0M NaCl | 1.66 | 0.17 | 0.23 | 0.49 | 0.66 |
| 1.0M NaCl + $\text{Ca}(\text{NO}_2)_2$ | 0.49 | 0.43 | 0.43 | 1.20 | 1.20 |

Precision and Reproducibility of $\text{Log}_{10}(1/R_p)$ Pooled Over All Materials
Average S_r S_R r R
(All dimensions are $\text{Log}_{10}(\mu\text{S}/\text{cm}^2)$)

| | | | | |
|------|------|------|------|------|
| 1.06 | 0.36 | 0.44 | 1.00 | 1.24 |
|------|------|------|------|------|

where:

- S_r = repeatability standard deviation
- r = 95 % repeatability limit (within laboratory)
- S_R = reproducibility standard deviation
- R = 95 % reproducibility limit (between laboratories)

respectively. The h statistic examines consistency of test results from laboratory to laboratory. The k statistic examines consistency of within-laboratory precision from laboratory to laboratory. In these plots, the horizontal lines are the critical values for h and k at the 0.5 % statistical significance level. There are a couple of excursions beyond the critical values for h or k in each plot, but there are no consistent patterns of concern in the plots. Therefore, the data appear consistent for purposes of this analysis.

X1.5 Example of a polarization resistance curve: