ASTM INTERNATIONAL

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Standard Test Method for
Corrosion Inhibiting Admixtures for Steel in Concrete by Polarization Resistance in Cementitious Slurries[1]

This standard is issued under the fixed designation G180; 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 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
C150/C150M Specification for Portland Cement
C595/C595M Specification for Blended Hydraulic Cements
C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
D632 Specification for Sodium Chloride
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements
G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements
G193 Terminology and Acronyms Relating to Corrosion

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice see Terminology G193.

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 test method shall not be used to predict performance in the field.

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 Reference Test Method G5.

5.2 Potentiostat, as described in Reference Test Method G5, 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 Reference Test Method G5.

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*—Two graphite rods or platinized-niobium or platinum mesh.

5.7 *Reference Electrodes*—A saturated calomel electrode with a controlled rate of leakage (about 3 µL/h) is recommended.

6. Reagents and Materials

6.1 Type I/II cement (C3A content between 6 % and 10 %), according to Specification C150/C150M, or a Type 1L cement (portland-limestone cement) according to Specification C595/C595M.

6.2 Filter paper with 1.1 µm retention.

6.3 PTFE stir bars.

6.4 Carbon steel C1215 samples, cylindrical in shape, with 5.1 cm^2 exposed area.[3]

6.5 Sodium chloride, reagent grade, according to Specification D632.

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. Water measured at 35 mL to 965 mL is equivalent to 5 L/m^3 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 Set up a standard electrochemical cell according to Reference Test Method G5 and fill it with 900 mL of filtered slurry solution. Purge the cell with carbon-dioxide-free air. Airflow rate should be at least 300 cc/min.

7.4 *Specimen Preparation before Testing:*

7.4.1 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.

7.4.2 Wet abrade the specimen with 600 grit paper and rinse with deionized water.

7.4.3 Repeat 7.4.1. Make sure the sample is thoroughly dried before mounting it on the electrode holder.

Note 3—This process removes machine oils and inhibitors that might be on the surface due to packaging.

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 CO_2-free air), to obtain a 0.5M or a 1M solution, and continue to stir and purge for a further 4 h. After 4 h, stop stirring and continue purging for another 20 h.

Note 4—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 G3.

7.5.5 Determine the polarization resistance R_p, as the tangent of the curve at $i = 0$, as described in Test Method G59. The corrosion rate is expressed as $1/R_p$ in µS/cm^2.

Note 5—An example of a polarization resistance curve is given in 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 6—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 µS/cm^2.

10. Precision and Bias[5]

10.1 Based on the pooled estimates of precision, the following statement of precision and bias 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

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[3] The sole source of supply of the apparatus known to the committee at this time is Metal Samples, AL, sample type P/N410. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] A CO_2 free air gas generator (typically used for FT-IR equipment) can be used.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G01-1021.

containing 30 % calcium nitrite). Practice E691 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 7—These numbers represent, respectively, the (1s) and (2ds) limits as described in Practice C670.

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

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 E691 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(μS/cm^2] (1s),
r = 95 % repeatability limit within a laboratory [log(μS/cm^2] (2ds),
S_R = reproducibility standard deviation [log(μS/cm^2] (1s), and
R = 95 % reproducibility limit between laboratories [log(μS/cm^2] (2ds).

X1.2 This study meets the minimum requirements for determining precision prescribed in Practice E691 in terms of the number of laboratories, materials and determinations (see 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, respec-

TABLE X1.1 Statistical Analysis of Corrosion Potential Data Practice E691 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 E691:

	This Study	Practice E691 Minimum
Laboratories	6	6
Materials	4	3
Determinations	2	2

Precision Statement for Test Method
Precision and Reproducibility of $\log_{10}(1/R_p)$ for Each Material
Precision, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the materials to be:
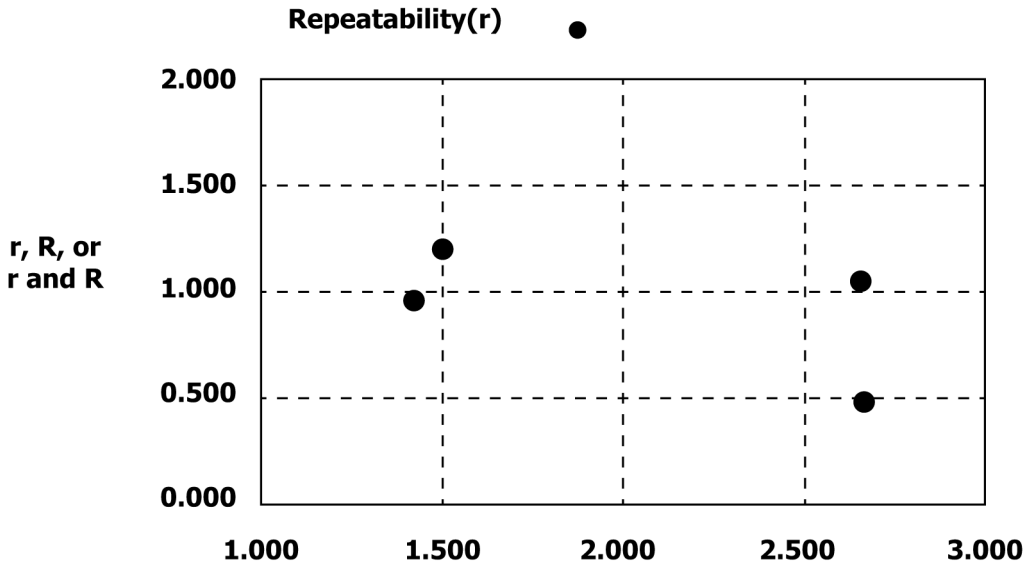
Material	Average	S_r	S_R	r	R
	(All dimensions are Log$_{10}$(μS/cm^2))				
0.5M NaCl	1.65	0.38	0.59	1.05	1.64
0.5M NaCl + Ca(NO$_2$)$_2$	0.42	0.34	0.40	0.95	1.11
1.0M NaCl	1.66	0.17	0.23	0.49	0.66
1.0M NaCl + Ca(NO$_2$)$_2$	0.49	0.43	0.43	1.20	1.20

Precision and Reproducibility of $\log_{10}(1/R_p)$ Pooled Over All Materials

|	Average	S_r	S_R	r	R
	(All dimensions are Log$_{10}$(μS/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)

tively. 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 Fig. X1.4 is an example of a polarization resistance curve.

3

Repeatability(r)

r, R, or
r and R

Analysis: Log$_{10}$(1/R$_p$) + 1

Reproducibility (R)

r, R, or
r and R
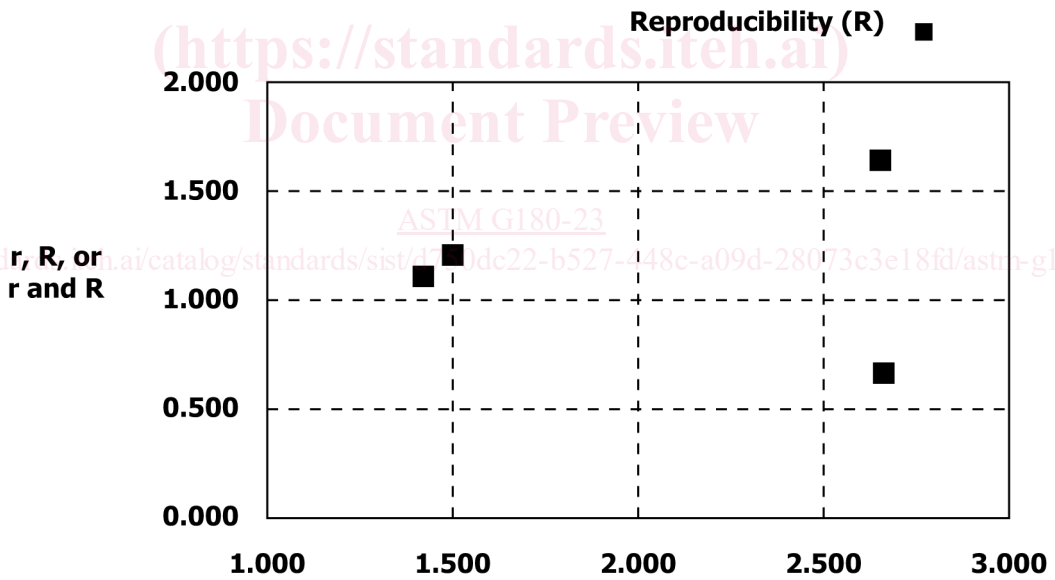
Average Log$_{10}$(1/R$_p$) + 1

FIG. X1.1 *r* and *R* versus Material Averages
Analysis: Log$_{10}$(1/R$_p$) + 1