Standard Test Method for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments[1]

This standard is issued under the fixed designation G 109; 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.

ϵ^1 NOTE—Paragraph 1.2 was corrected editorially in August 2000.

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 in a chloride environment.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses are provided 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:*

A 615/A 615M Specification for Deformed and Plain Billet-Steel Bars for Concrete Reinforcement[2]
C 33 Specification for Concrete Aggregates[3]
C 143/C 143M Test Method for Slump of Hydraulic Cement Concrete[3]
C 150 Specification for Portland Cement[4]
C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method[3]
C 192/C 192M Practice for Making and Curing Concrete Test Specimens in the Laboratory[3]
C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method[3]
C 511 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes[4]
C 876 Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete[3]
C 881 Specification for Epoxy-Resin-Base Bonding Systems for Concrete[3]
C 1152/C 1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete[3]
D 448 Classification for Sizes of Aggregate for Road and Bridge Construction[5]
D 632 Specification for Sodium Chloride[5]
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[6]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]
G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing[7]
G 15 Terminology Relating to Corrosion and Corrosion Testing[7]
G 33 Practice for Recording Data from Atmospheric Corrosion Tests of Metallic-Coated Steel Specimens[7]
G 46 Guide for Examination and Evaluation of Pitting Corrosion[7]

2.2 *NACE Standards:*

SSPC SP 5 (NACE No. 1) White Metal Blast Cleaning[8]

3. Significance and Use

3.1 This test method provides a reliable means for predicting the inhibiting or corrosive properties of admixtures to be used in concrete.

3.2 This test method is useful for development studies of corrosion inhibitors to be used in concrete.

3.3 This test method has been used elsewhere with good agreement between corrosion as measured by this test method and corrosion damage on the embedded steel.[9,10 ,11,12] This test

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[1] This test method is under the jurisdiction of ASTM Committee G-1 on Corrosion, Deterioration, and Degradation of Materials and is the direct responsibility of Subcommittee G01.14 on Corrosion of Reinforcing Steel.

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[2] *Annual Book of ASTM Standards*, Vol 01.04.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 04.01.

[5] *Annual Book of ASTM Standards*, Vol 04.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] *Annual Book of ASTM Standards*, Vol 03.02.

[8] Available from Structural Steel Painting Council, Pittsburgh, PA.

[9] Berke, N. S., Shen, D. F., and Sundberg, K. M., "Comparison of the Polarization Resistance Technique to the Macrocell Corrosion Technique," *Corrosion Rates of Steel in Concrete, ASTM STP 1065*, N. S. Berke, V. Chaker, and D. Whitney, editors, ASTM, August 1990, pp. 38–51.

method might not properly rank the performance of different corrosion inhibitors, especially at concrete covers over the steel less than 40 mm (1.5 in.) or water-to-cement ratios above 0.45. The concrete mixture proportions and cover over the steel are chosen to accelerate chloride ingress. Some inhibitors might have an effect on this process, which could lead to results that would differ from what would be expected in actual use.[13]

4. Apparatus

4.1 The apparatus required for the evaluation of corrosion inhibitors includes a high impedance voltmeter (at least one Mohm) capable of measuring to 0.01 mV, a 100-ohm (± 5 %) resistor.

5. Reagents and Materials

5.1 *Cement*, that conforms to Type I or Type II of Specification C 150. Coarse aggregate shall conform to Specification C 33 and Classification D 448, with nominal maximum size between 9.5 and 19 mm (⅜and ¾ in.).

NOTE 1—Preferred maximum size aggregate is 12.5 mm (0.5 in.).

5.2 *Steel Reinforcement Bars*, deformed, meeting the requirement of Specification A 615/A 615M, with a diameter between 10 mm (0.4 in.) and 16 mm (0.6 in.). A length of 360 mm (14 in.), drilled and tapped at one end to be fitted with coarse-thread stainless steel and nuts, as described in 5.3 and 5.4. These bars shall be used to manufacture the test specimens, as described in Section 6.

NOTE 2—Interlaboratory test program and statistical data in Section 11 are based upon 13-mm (0.5-in.) steel bars, 12.5-mm maximum size aggregate, and 19-mm (0.75-in.) and 25-mm (1 in.) cover

5.3 *316 Stainless Steel Screws*, with diameter smaller than bar diameter (coarse thread < 5 mm (0.2 in.)), 25 to 35-mm (1 to 1.5-in.) long (one per bar).

5.4 *316 Stainless Steel Nuts*, two per bar to fit stainless steel screws, as described in 5.3.

5.5 *Two-part Waterproof Epoxy*[14]— This epoxy shall meet the chemical resistance requirements of a Type IV, Grade 3, Class E of Specification C 881.

5.6 *Sulfuric Acid*, 10 % by mass, for pickling (optional).

5.7 *Electroplater's Tape*[15]

5.8 *Neoprene Tubing*, with 3-mm (⅛-in.) wall thickness and the same ID as the diameter of the bar used.

5.9 *Sodium Chloride*, complying with Specification D 632.

5.10 *Salt Solution*, prepared by dissolving 3 parts of sodium chloride (as described in 5.9) in 97 parts of water mass.

5.11 *Epoxy Sealer*, for application to the concrete specimens after manufacture. This sealer shall be of Type III, Grade 1, Class C in accordance with Specification C 881.[16]

5.12 *Plastic Dams*, 75-mm (3-in.) wide and 150-mm (6-in.) long with a minimum height of 75 mm (3 in.) for placement on the test specimens. The wall thickness shall be ± 1 mm (⅛ ± 1⁄32 in.

5.13 *Silicone Caulk*, for sealing the outside of the plastic dam to the top of the concrete specimen.[17]

5.14 *Reference Electrode*, such as a saturated calomel or silver/silver chloride electrode for measuring the corrosion potential of the bars, as defined in Terminology G 15.

5.15 *Hexane*

6. Preparation of Test Specimens

6.1 Power wire brush or sand blast the bars to near white metal (see Specification SSPC SP-50), clean by soaking in hexane, and allow to air dry.

NOTE 3—Pickling the bars with 10 % sulfuric acid for 10 to 15 min and rinsing with potable water prior to wire brushing is recommended when the bars have an excessive amount of rust.

6.2 Use the same method to clean all bars in the test program.

6.3 Drill and tap one end of each bar, attach a stainless steel screw and two nuts, as described in 5.3 and 5.4, and tape each end of the bar with electroplater's tape so that a 200-mm (8-in.) portion in the middle of the bar is bare. Place a 90-mm (3.5 in.) length of neoprene tubing, as described in 5.8, over the electroplater's tape at each end of the bar, and fill the length of tubing protruding from the bar ends with the two-part epoxy, as described in 5.5.

NOTE 4—For example, for a 12.5-mm (0.5 in.) aggregate, place the top bar 25 mm (1 in.) from the surface. For a 9.5-mm (0.375-in.) aggregate, place the bar 19 mm (.75 in.) from the top surface.

6.4 Specimen size is 280 × 150 × 115 mm (11 × 6 × 4.5 in.).

6.5 Place the bars in the molds so that 40 mm (approximately 1.5 in.) of the bars are protected within each exit end from the concrete (minimizes edge effects). This will expose 200 mm (8 in.) of steel. Place the bars with the longitudinal ribs so that they are nearer the side of the beam, that is, both ridges are equidistant from the top or bottom of the specimen.

6.6 Make the concrete specimens (controls and those with admixtures to be tested) in accordance with Practice C 192/C 192M, using the same source of materials. Determine the air content, using either Test Method C 231 or C 173. The water-to-cement ratio (*w/c*) shall not exceed 0.5. The minimum slump is 50 mm (2 in.) (see Test Method C 143/C 143M). Place and consolidate the concrete in the molds containing the bars in accordance with Practice C 192/C 192M.

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[10] Berke, N. S. and Hicks, M. C., "Electrochemical Methods of Determining the Corrosivity of Steel in Concrete," *Corrosion Testing and Evaluation: Silver Anniversary Volume*, Babraiam/Dean editors, ASTM STP 1000, ASTM, November 1990, pp. 425–440.

[11] Virmani, Y. P., Clear, K. C., and Pasko, T. J., "Time-to Corrosion of Reinforcing Steel in Concrete Slabs, Volume 5: Calcium Nitrite Admixture or Epoxy-Coated Reinforcing Bars as Corrosion Protection Systems," Report No. FHWA/RD-83/-12, Federal Highway Administration, Washington DC, 1983, pp. 71.

[12] Berke, N. S., Pfeifer, D. W., and Weil, T. G., "Protection Against Chloride-Induced Corrosion," *Concrete International*, December 1988, pp. 45–55.

[13] Berke, N. S., Hicks, M. C., Hoopes, R. J., and Tourney, P. J., "Use of Laboratory Techniques to Evaluate Long-Term Durability of Steel Reinforced Concrete Exposed to Chloride Ingress," ACI SP 145-16, 1994, pp. 299-328.

[14] PC-Epoxy made by Protective Coating Co., Allentown, PA, has been found to be suitable for this purpose.

[15] Minnesota Mining and Manufacturing Company (3M), 1999 Mt. Read Boulevard, Rochester, NY 14615, has been found suitable for this purpose.

[16] Epoxy Concrete Scaler # 12560 made by Devcon has been found to be suitable for this purpose.

[17] 3M Marine Adhesive 5200 has been found to be suitable for this purpose.

NOTE 5—The concrete parameters used in the inter-laboratory test were as follows: cement content of 355 ± 3 kg/m^3 (600 ± 5 lb/yd ^{3}), 0.50 ± 0.01 W/C (ssd aggregates), and 6 ± 1 % air.

6.7 Add the admixture to be tested at the manufacturer's recommended dosages. A water reducer is allowed, if needed, to achieve the desired slump. Record the admixtures used. Except for the test admixtures, use the same admixtures in all mixtures.

6.8 A minimum of three replicates shall be made. Make the same number of replicates per admixture tested and control (see Note 6). An addition cylinder 100 × 200 mm in diameter (4 × 8 in.) shall be produced for background chloride analysis.

NOTE 6—A larger number of replicates is preferred.

6.9 Apply a wood float finish after consolidation. After removal from the forms, cure the specimens for 28 days in a moist room in accordance with Test Method C 192 and Specification C 511.

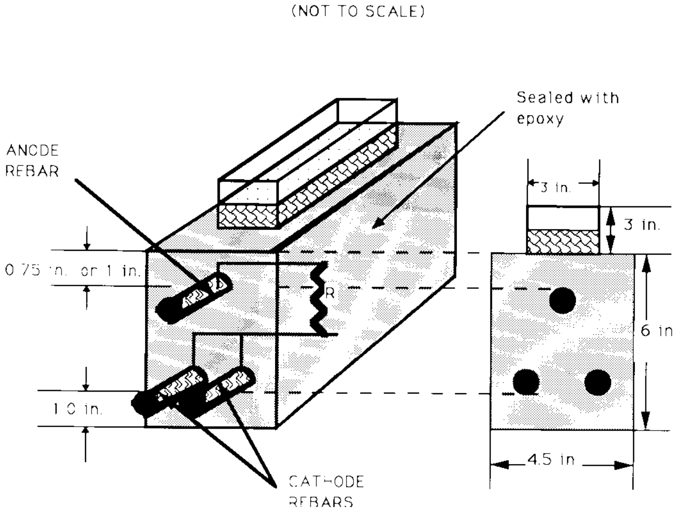
6.10 Upon removal from the moist room, hand wire brush the specimens on the concrete top surface (wood floated surface). Allow the specimens to dry for two weeks in a 50 % relative humidity (RH) environment before sealing the four vertical sides with an epoxy sealer, as described in 5.11, in accordance with the manufacturer's recommendation. Place a plastic dam with dimensions, as described in 5.12, on the specimen, as shown in Fig. 1, and about 13 mm (0.5 in.) from each side so that it does not extend over the taped sections of the bars (see Fig. 2). Use a silicone caulk to seal the dam from the outside, and apply epoxy sealer to the top surface outside of the dam.

NOTE 7—Allowing the specimens to dry before applying the concrete epoxy will make the initial exposure to chloride more severe, and more closely follow the inter-laboratory test program conditions.

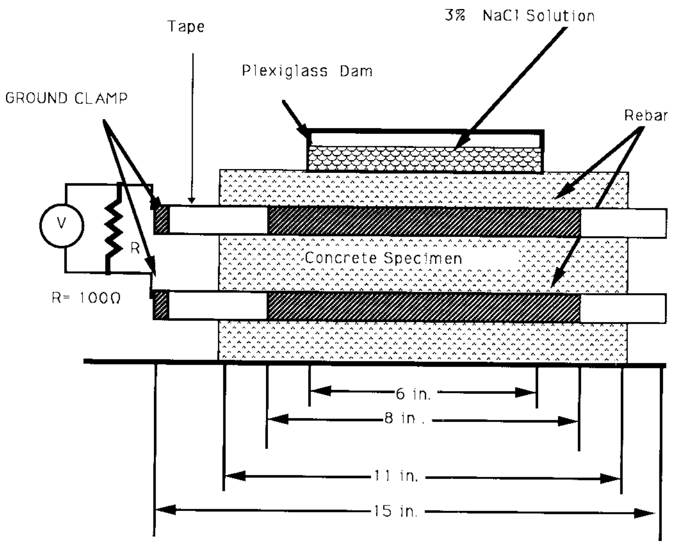
6.11 Attach wires and resistors.

7. Procedure

7.1 Support each test specimen on two nonelectrically conducting supports at least 13-mm (0.5-in.) thick, thus allowing air flow under most of the specimen. Start the test one month after the samples are removed from the 100 % RH atmosphere (moist room). Pond the specimens for two weeks at 23 ± 3°C (73 ± 5°F) with the salt solution, as described in 5.10. The volume of this solution is approximately 400 mL at a depth of 40 mm (1.5 in.). Use a plastic loose fitting cover to minimize evaporation. Maintain a relative humidity around the specimens of 50 ± 5 %. After two weeks, vacuum off the solution and allow the samples to dry for two weeks. Repeat this cycle.



NOTE 1—All measurements in inches (25.4 mm = 1 in.).
FIG. 1 Concrete Beam



NOTE 1—All measurements in inches (not to scale) (25.4 mm = 1 in.).
FIG. 2 Concrete Beam (Side View)

7.2 Measure the voltage across the resistor at the beginning of the second week of ponding using the voltmeter defined in 4.1. Calculate the current, I_j, from the measured voltage across the 100-ohm resistor, V_j, measured in volts (see Note 8) as:

$$I_j = V_j/100$$

NOTE 8—With the common terminal on the bottom bar, negative voltages correspond to positive galvanic current (that is, the top bar is the anode).

7.3 At the same time, measure the corrosion potential of the bars against a reference electrode that is placed in the dam containing the salt solution (see Practice G 3 and Test Method C 876). Connect the voltmeter between the reference electrode (ground or common terminal) and the bars.

8. Period of Testing

8.1 Monitor the current as a function of time once every four weeks, as described in 7.2, until the average integrated macrocell current of the control specimens is 150 C or greater, as determined in 10.1.8, and at least half the samples show integrated macrocell currents equal to or greater than 150 C (see Note 9).

NOTE 9—The value of 150 C is consistent with a macrocell current of 10 μA over six months. The value of 10 μA was measured by all laboratories on all specimens showing corrosion (controls and samples with calcium chloride at 19-mm (¾-in.) cover). This degree of integrated macrocell current is sufficient to ensure the presence of sufficient corrosion for visual evaluation.

8.2 In those cases where the admixtures being tested are corrosive, end the test three full cycles after an average integrated macrocell current of 75 C is observed and the integrated macrocell current of at least half the specimens being tested is equal or greater than 75 C.

9. Examination of Embedded Bars

9.1 At the conclusion of testing, break the specimens and examine the reinforcement bars for extent of corrosion, measure the corroded area, and record the percentage of corroded area recorded, as described in Practice G 33.

NOTE 10—Photograph the bars at the end of the test to provide a record of the corrosion damage.

9.2 Determine the acid soluble chloride content at the depth corresponding to the cover over the top-reinforcing bar, using Test Method C 1152.

9.3 Determine the acid soluble chloride content in the specimen produced for background chloride analysis, using Test Method C 1152. This value is to be subtracted from the acid soluble chloride, as determined in 9.2, to provide a corrected acid soluble chloride content reflecting ingressed chloride.

10. Report

10.1 Report the following information:

10.1.1 Full details of the concrete proportions, air content, and slump of the concrete used in the control and test specimens,

10.1.2 A plot of the corrosion current and potential for each concrete specimen versus time,

10.1.3 A plot of the average integrated current for each condition of concrete versus time,

10.1.4 Time to failure, as considered to be the time for the average macrocell current to reach 10 µA and at least half the samples showing a current greater than 10 µA,

10.1.5 Results of the visual inspection of each bar. The report shall include the percentage of original exposed steel surface corroded and optionally the number and depths of corrosion pits where present, as described in Practice G 46,

10.1.6 Photographs of the bars at the end of the test (optional), and

10.1.7 Chloride content at the top reinforcing bar depth from the surface. This value is the corrected total chloride content, as corrected 9.3.

10.1.8 The ratio of total integrated current of the test specimen to that of the control and time the test ended. The total integrated current is:

$$TC_j = TC_{j-1} + [(t_j - t_{j-1}) \times (i_j + i_{j-1})/2]$$

where:

TC = total corrosion (coulombs),

t_j = time (seconds) at which measurement of the macrocell current is carried out, and

i_j = macrocell current (amps) at time, t_j.

A sample calculation is given in Appendix X1.

11. Precision and Bias [18]

11.1 Information on the precision of the results obtained by this test method was derived from an inter-laboratory test with two to three specimens per laboratory. Eleven laboratories participated in the study. The repeatability and reproducibility of the test results were dependent on the magnitude of the mean macrocell current.

11.2 Precision is as follows:

11.2.1 *95 % Repeatability Limit (Within Laboratory)*—The within-laboratory precision of the average macrocell current (for each laboratory), as expressed by the repeatability limit, *r*, is given by the following relation:

$$\log r = 0.931 \log I_{avg} + 0.441 \tag{1}$$

11.2.2 *95 % Reproducibility Limit (Between Laboratories)*—The between-laboratory precision of the average macrocell current (for all laboratories), as expressed by the reproducibility, *R*, is given by the following relation:

$$\log R = 0.833 \log I_{avg} + 0.624 \tag{2}$$

11.2.3 The repeatability and reproducibility limits of the average macrocell current were calculated in accordance with Practice E 177. The respective standard deviations of the variation among test results can be obtained by dividing by 2.8 the values of *r* and *R* calculated using (Eq 1) and (Eq 2). The following equations were then obtained:

$$\log S_r = 0.931 \log I_{avg} - 0.006 \tag{3}$$

$$\log S_R = 0.833 \log \ I_{avg} + 0.177 \tag{4}$$

11.2.4 The data used for compiling the test method precision, together with the statistical parameters as defined in Practice E 691, are given in the research report.[18] The graphical representations of the repeatability and reproducibility limits are given in Figs. 3 and 4.

11.2.5 The time to failure has been analyzed using Practice E 691. This analysis is given in the research report.[18]

11.2.6 The maximum end of the 95 % confidence interval for time to failure for control specimens with 19-mm (0.75-in.)

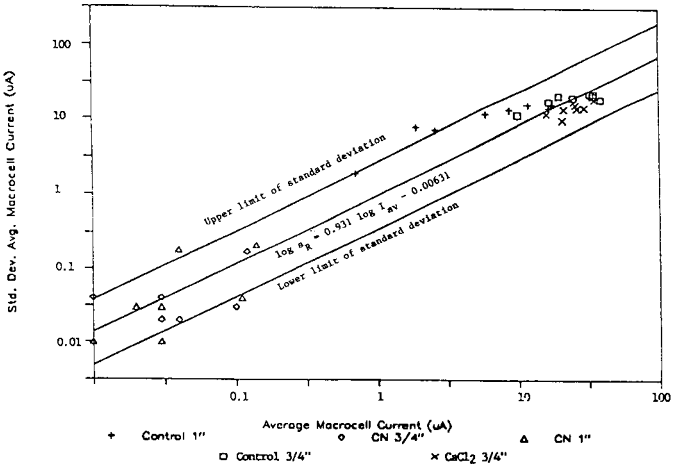


FIG. 3 Standard Deviation of Repeatability

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[18] Supporting data have been filed at ASTM Headquarters. Request RR:G01-1009.

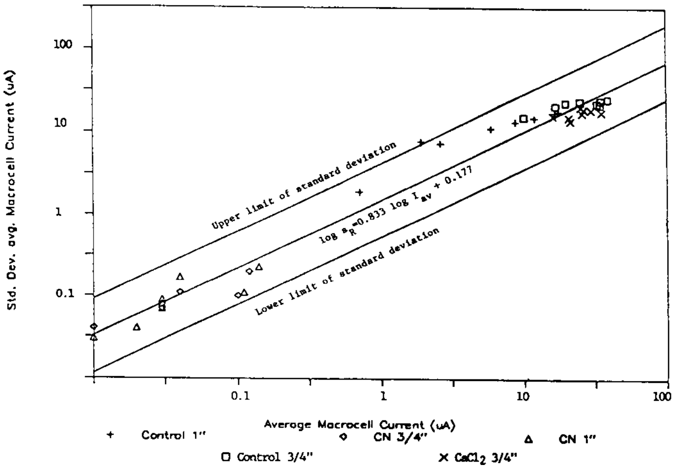

FIG. 4 Standard Deviation of Reproducibility

concrete cover is six months for both intralaboratory and interlaboratory tests. The maximum ends of the 95 % confidence intervals are two and six months for intra- and interlaboratory tests respectively for specimens containing calcium chloride.

11.2.7 The complete data for percent area corroded is given in the research report that has been filed with ASTM Headquarters.[18] In all cases where there was corrosion, the macrocell current was greater than 9 µA. However, not enough laboratories reported percent area corroded to carry out a statistical analysis following Practice E 691.

11.3 *Bias*—The procedure given in this test method has no bias because the effects of chemical admixtures on the corrosion of embedded steel of reinforcement are defined only in terms of this test method.

12. Keywords

12.1 admixtures; concrete; corrosion; corrosivity; inhibitor; reinforcing steel

APPENDIX

(Nonmandatory Information)

X1. TOTAL CORROSION CALCULATION

X1.1 *Total Corrosion Calculation:*

$$TC_j = TC_{j-1} + [(t_j - t_{j-1}) * (i_j + i_{j-1})/2] \qquad (X1.1)$$

X1.1.1 Assume the following readings were obtained over a 90 day period of time:

Days	0	30	60	90
i_{mac} (µA)	0	20	27	35

X1.1.2 At the end of the first 30 day period the total corrosion is:

$$TC_1 = 0 + [(30 - 0) * 86400 * (20 + 0)/2 * 10^{-6}] = 25.92\ \text{C} \qquad (X1.2)$$

X1.1.3 At the end of the 60 day period:

$$TC_2 = 25.92 + [60 - 30) * 86400 * (20 + 27)/2*10^{-6}] = 86.83\ \text{C} \qquad (X1.3)$$

X1.1.4 At the end of the 90 day period:

$$TC_3 = 86.83 + [(90 - 60) * 86400 * (27 + 35)/2*10^{-6}] = 167.18\ \text{C} \qquad (X1.4)$$

NOTE X1.1—Conversion factor from days to seconds = 24 × 60 × 60 = 86 400.