Designation: C 403/C 403M – 99

Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance[1]

This standard is issued under the fixed designation C 403/C 403M; 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 the determination of the time of setting of concrete, with slump greater than zero, by means of penetration resistance measurements on mortar sieved from the concrete mixture.

1.2 This test method is suitable for use only when tests of the mortar fraction will provide the information required.

1.3 This test method may also be applied to prepared mortars and grouts.

1.4 This test method is applicable under controlled laboratory conditions, as well as under field conditions.

1.5 The values stated in either inch-pound units or SI units shall be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining values in any way.

1.6 *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:*

C 125 Terminology Relating to Concrete and Concrete Aggregates[2]

C 143/C 143M Test Method for Slump of Hydraulic Cement Concrete[2]

C 172 Practice for Sampling Freshly Mixed Concrete[2]

C 173 Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method[2]

C 192/C 192M Practice for Making and Curing Concrete Test Specimens in the Laboratory[2]

C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

D 1558 Test Method for Moisture Content Penetration Resistance Relationships of Fine Grained Soils[3]

E 1 Specification for ASTM Thermometers[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

3. Terminology

3.1 *Definitions*—Definitions are given in Terminology C 125.

4. Summary of Test Method

4.1 A mortar sample is obtained by sieving a representative sample of fresh concrete. The mortar is placed in a container and stored at a specified ambient temperature. At regular time intervals, the resistance of the mortar to penetration by standard needles is measured. From a plot of penetration resistance versus elapsed time, the times of initial and final setting are determined.

5. Significance and Use

5.1 Since the setting of concrete is a gradual process, any definition of time of setting must necessarily be arbitrary. In this test method, the times required for the mortar to reach specified values of resistance to penetration are used to define times of setting.

5.2 This test method can be used to determine the effects of variables, such as water content; brand, type and amount of cementitious material; or admixtures, upon the time of setting of concrete. This test method may also be used to determine compliance with specified time-of-setting requirements.

5.3 This test method may also be applied to prepared mortars and grouts. However, when the setting time of concrete is desired, the test shall be performed on mortar sieved from the concrete mixture and not on a prepared mortar intended to simulate the mortar fraction of the concrete; it has been shown that the initial and final setting times may be increased when using the prepared mortar.

6. Apparatus

6.1 *Containers for Mortar Specimens*—The containers shall be rigid, watertight, nonabsorptive, free of oil or grease, and

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

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

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

either cylindrical or rectangular in cross section. Mortar surface area shall be provided for ten undisturbed readings of penetration resistance in accordance with clear distance requirements specified in Procedure. The lateral dimension shall be at least 6 in. [150 mm] and the height at least 6 in. [150 mm].

6.2 *Penetration Needles*—Needles shall be provided which can be attached to the loading apparatus and which have the following bearing areas: 1, ½, ¼, 1⁄10, 1⁄20, and 1⁄40 in.^{2} [645, 323, 161, 65, 32, and 16 mm^2]. Each needle shank shall be scribed circumferentially at a distance 1 in. [25 mm] from the bearing area. The length of the 1⁄40 in.^{2}[16-mm^2] needle shall be not more than 3½ in. [90 mm].

6.3 *Loading Apparatus*—A device shall be provided to measure the force required to cause penetration of the needles. The device shall be capable of measuring the penetration force with an accuracy of ±2 lbf [10 N] and shall have a capacity of at least 130 lbf [600 N].

NOTE 1—Suitable loading apparatus can be of the spring-reaction type as described in Test Method D 1558, or of other types with a calibrated force measuring device, such as an electronic load cell or a hydraulic pressure gage.

6.4 *Tamping Rod*—The tamping rod shall be a round, straight, steel rod 5⁄8in. [16 mm] in diameter and approximately 24 in. [600 mm] in length, having the tamping end or both ends rounded to a hemispherical tip, the diameter of which is 5⁄8 in. [16 mm].

6.5 *Pipet*—A pipet or other suitable instrument shall be used for drawing off bleed water from the surface of the test specimen.

6.6 *Thermometer*—The thermometer shall be capable of measuring the temperature of the fresh mortar to ±1°F [±0.5°C]. ASTM liquid-in-glass thermometers having a temperature range from 0 to 120°F [−20 to 50°C], and conforming to the requirements of Thermometer 97F (or 97C) as prescribed in Specification E 1 are satisfactory. Other thermometers of the required accuracy, including the metal immersion type, are acceptable.

7. Sampling, Test Specimens, and Test Units

7.1 For tests under field conditions, prepare three specimens from each sample of concrete.

7.2 For tests under laboratory conditions, the requirements depend upon the purpose of the tests.

7.2.1 For testing to prove compliance of a material with performance requirements, make at least three separate concrete batches for each variable under investigation. Perform one time of setting test on each batch. Make an equal number of batches for each variable on any one day. When it is impossible to perform at least one test for each variable on any one day, mix the entire series of batches in as few days as possible, and repeat one of the mixtures each day as a standard for comparison.

7.2.2 For other tests, prepare three test specimens from one batch of concrete for each test variable.

7.3 Record the time at which initial contact was made between cement and mixing water.

7.4 For tests under field conditions, obtain a representative sample of the fresh concrete in accordance with Practice C 172. For tests under laboratory conditions, make the concrete in accordance with Practice C 192. Determine and record the slump (Test Method C 143) and air content (Test Method C 173 or C 231) of the fresh concrete.

7.5 From the concrete not used in the slump and air content tests, select a representative portion of sufficient volume to provide enough mortar to fill the test container, or containers, to a depth of at least 5½ in. [140 mm].

7.6 Using the procedure in Practice C 172, obtain a mortar sample by wet-sieving the selected portion of concrete through a 4.75-mm sieve[6] and onto a nonabsorptive surface.

7.7 Thoroughly remix the mortar by hand methods on the nonabsorptive surface. Measure and record the temperature of the mortar. Place the mortar in the container, or containers, using a single layer. Consolidate the mortar to eliminate air pockets in the specimen and level the top surface. This may be accomplished by rocking the container back and forth on a solid surface, by tapping the sides of the container with the tamping rod, by rodding the mortar, or by placing the container on a vibrating table (see Note 2). If rodding is used, rod the mortar with the hemispherical end of the tamping rod. Rod the mortar once for each 1 in.^{2} [645 mm^2] of top surface area of the specimen and distribute the strokes uniformly over the cross section of the specimen. After completion of the rodding, tap the sides of the containers lightly with the tamping rod to close voids left by the tamping rod and to further level the surface of the specimen. Upon completion of specimen preparation, the mortar surface shall be at least ½ in. [10 mm] below the top edge of the container to provide space for the collection and removal of bleed water and to avoid contact between the mortar surface and the protective covering specified in Section 8.

NOTE 2—Sieved mortar is generally of fluid consistency and air pockets are readily removed by the listed consolidation methods. The user should exercise judgment in the selection of the consolidation method. Rocking the container or tapping of the sides should be sufficient for fluid mortars. Rodding or using a vibrating table may be desirable for stiffer mortars. When using a vibrating table, use low-amplitude vibration so that portions of the sample are not ejected from the container.

8. Conditioning

8.1 For tests under laboratory conditions, the storage temperature for specimens shall be within the range 68 to 77°F [20 to 25°C], or as specified by the user.

8.2 For tests under field conditions, store the specimens under ambient conditions, or as specified by the user. Shield the specimens from direct sunlight.

8.3 Measure and record the ambient air temperature at the start and finish of the test. To prevent excessive evaporation of moisture, keep the specimens covered with a suitable material such as damp burlap or a tight-fitting, water-impermeable cover for the duration of the test, except when bleed water is being removed or penetration tests are being made.

9. Procedure

9.1 Just prior to making a penetration test, remove bleed water from the surface of the mortar specimens by means of a

[6] Detailed requirements for this sieve are given in Specification E 11.

pipet or suitable instrument. To facilitate collection of bleed water, tilt the specimen carefully to an angle of about 10° from the horizontal by placing a block under one side 2 min prior to removal of the water.

9.2 Insert a needle of appropriate size, depending upon the degree of setting of the mortar, in the penetration resistance apparatus and bring the bearing surface of the needle into contact with the mortar surface. Gradually and uniformly apply a vertical force downward on the apparatus until the needle penetrates the mortar to a depth of 1 ± 1⁄16 in. [25 ± 2 mm], as indicated by the scribe mark (Note 3). The time required to penetrate to the 1-in. [25-mm] depth shall be 10 ± 2 s. Record the force required to produce the 1-in. [25-mm] penetration and the time of application, measured as elapsed time after initial contact of cement and water. Calculate the penetration resistance by dividing the recorded force by the bearing area of the needle, and record the penetration resistance. In subsequent penetration tests take care to avoid areas where the mortar has been disturbed by previous tests. The clear distance between needle impressions shall be at least two diameters of the needle being used, but not less than ½ in. [15 mm]. The clear distance between any needle impression and the side of the container shall be at least 1 in. [25 mm].

NOTE 3—To facilitate determination of when the required penetration has been attained, a sliding marker may be attached to the needle shaft. For example, a paper clip or masking tape may be placed on the shaft so that it coincides with the scribe mark. The marker should not interfere with the penetration of the needle into the mortar. The position of the marker should be checked prior to making a penetration.

9.3 For conventional concrete mixtures at laboratory temperatures of 68 to 77°F [20 to 25°C], make the initial test after an elapsed time of 3 to 4 h after initial contact between cement and water. Subsequent tests should be made at ½- to 1-h intervals. For concrete mixtures containing accelerators, or at temperatures higher than laboratory, it is advisable to make the initial test after an elapsed time of 1 to 2 h and subsequent tests at ½-h intervals. For concrete mixtures containing retarders, or at temperatures lower than laboratory, the initial test may be deferred until an elapsed time of 4 to 6 h. In all cases, time intervals between subsequent tests may be adjusted as necessary, depending upon the rate of setting, to obtain the required number of penetrations.

9.4 Make at least six penetrations for each time-of-setting test, with time intervals of such duration as to provide a satisfactory curve of penetration resistance versus elapsed time (Note 4). Continue testing until one at least penetration resistance reading equals or exceeds 4000 psi [27.6 MPa].

NOTE 4—A satisfactory curve is one which represents the overall development of penetration resistance and includes points before and after the times of initial and final setting to improve the accuracy of the required interpolation. For normal setting mixtures, test points are usually at equally spaced time intervals. Premature penetration testing will result in too many data points earlier than the initial setting time. This may decrease the accuracy of the estimated setting time by biasing the best fit line when regression analysis is used to analyze the penetration resistance data.

9.5 *Plotting Test Results*—One of the following alternative procedures may be used to plot the test results and obtain times of setting (Note 5). Appendix X1 illustrates the application of these procedures.

NOTE 5—The plot of penetration resistance versus elapsed time provides information on the rate of setting. The plot may be used to select the time for subsequent penetration tests and it can assist in identifying spurious test results. Therefore, it is recommended that the data be plotted as they are being accumulated.

9.5.1 Use the following plotting procedure to determine the times of setting by hand-fitting a smooth curve through the data. Prepare a graph of penetration resistance, as the ordinate, versus elapsed time, as the abscissa, using a scale such that 500 psi [3.5 MPa] and 1 h are each represented by a distance of at least ½ in. [15 mm]. Plot the values of penetration resistance as a function of elapsed time.

9.5.2 Use the following plotting procedure to determine the times of setting by linear regression analysis of the logarithms of the data by using a suitable calculator. Using log-log graph paper, prepare a graph of penetration resistance, as the ordinate, versus elapsed time in minutes, as the abscissa. The limits of penetration resistance on the ordinate should extend from 10 psi [0.1 MPa] to 10 000 psi [100 MPa], and the limits of elapsed time on the abscissa should extend from 10 to 1000 min. If slow setting mixtures are used, the time limits may have to be 100 to 10 000 min. Plot the values of penetration resistance as a function of elapsed time (Note 4).

9.5.3 Use the following procedure if a computer is used to plot test results and obtain the times of setting by regression analysis of the data. As the test results are obtained, enter the time and penetration resistance into the computer and plot the penetration resistance as the ordinate and the elapsed time as the abscissa. For software that permits only linear regression analysis, convert the data by taking their logarithms. The converted data will be fitted by a straight line[7] (see Eq 1):

$$\text{Log}\,(PR) = a + b\,\text{Log}\,(t) \tag{1}$$

where:
PR = penetration resistance
t = elapsed time
a and b = regression constants

The data do not have to be converted if the software permits direct fitting of a power function:

$$PR = ct^{d} \tag{2}$$

where:
c and d = regression constants

9.5.4 The procedures in 9.5.2 and 9.5.3 assume that the data obey (Eq 1) or (Eq 2). Verify that the data obey one of these relationships. If the correlation coefficient for the regression analysis, after removal of outliers (see Note 6), is less than 0.98, use the procedure in 9.5.1.

10. Calculation

10.1 For each variable under investigation, separately plot the results of three or more time-of-setting tests. For each plot prepared according to 9.5.1, hand fit a smooth curve to the data

[7] Popovics, S., 1971, "Physical Aspects of the Setting of Portland Cement Concrete," *Journal of Materials*, JMLSA, Vol. 6, No. 1, March, pp. 150–162.

points. For each plot prepared according to 9.5.2 or 9.5.3, use the method of least squares to obtain the constants of the best-fit relationship given by (Eq 1) or (Eq 2), whichever is applicable. Disregard data points that are obvious outliers from the trend defined by the rest of the points (Note 6).

NOTE 6—Outliers may occur because of factors such as: interferences due to the larger particles in the mortar; presence of large voids within the penetration zone; interferences from the impressions created by adjacent penetrations; failure to maintain the instrument perpendicular to the test surface during penetration; errors in reading the load; variations in the penetration depths; or variations in rate of loading. Judgement of the operator is required to identify those points that should not be included in the data analysis.

10.2 For each plot, determine the times of initial and final setting as the times when the penetration resistance equals 500 psi [3.5 MPa] and 4000 psi [27.6 MPa], respectively. For plots made according to 9.5.1, determine times of setting by visual inspection of the drawn curves. For plots made according to 9.5.2 or 9.5.3, determine the times of setting by interpolation using the best-fit regression equation. Record the times of setting in hours and minutes to the nearest 5 min.

10.3 For each variable under investigation, calculate the times of initial and final setting as the average values of the individual test results. Record the average times in hours and minutes to the nearest 5 min.

11. Report

11.1 *Data on Concrete Mixture*—Report the following information on the concrete mixture:

11.1.1 Brand and type of cementitious materials, amounts (mass) of cementitious materials, fine aggregate and coarse aggregate per cubic yard [per cubic metre] of concrete, nominal maximum aggregate size, and water-cement or water-cementitious material ratio,

11.1.2 The name, type, and amount of admixture(s) used,

11.1.3 Air content of fresh concrete and method of determination,

11.1.4 Slump of concrete,

11.1.5 Temperature of mortar after sieving,

11.1.6 Record of ambient temperature during the test period, and

11.1.7 Date of test.

11.2 *Time of Setting Results*—Report the following information on the time of setting tests:

11.2.1 A plot of penetration resistance versus elapsed time for each time of setting test,

11.2.2 The times of initial and final setting for each test, reported in hours and minutes to the nearest minute, and

11.2.3 The average times of initial and final setting for each test condition, reported in hours and minutes to the nearest 5 min.

12. Precision and Bias

12.1 The following precision statements are based on testing of prepared mortar using the inch-pound system and for data analysis using the hand-plotting procedure (see 9.5.1, 10.1, and 10.2).

12.2 The single-operator coefficient of variation for time of initial setting for single tests on each of three batches made on separate days has been found to be 7.1 % (Note 7). Therefore, the range of results obtained on three separate batches by the same operator with the same apparatus, using the same materials and temperature conditions, on three different days should not exceed 23 % of their average.

12.3 The multilaboratory coefficient of variation for the average time of initial setting, based on three tests from separate batches made on separate days, has been found to be 5.2 %. Therefore, the averages of three tests from two different laboratories using the same materials and temperature conditions should not differ by more than 15 % of their average (Note 8).

12.4 The single-operator coefficient of variation for time of final setting for single tests on each of three batches made on separate days has been found to be 4.7 % (Note 7). Therefore, the range of results obtained on three separate batches by the same operator with the same apparatus, using the same materials and temperature conditions, on three different days should not exceed 16 % of their average.

12.5 The multilaboratory coefficient of variation for the average time of final setting, based on three tests from separate batches made on separate days, has been found to be 4.5 %. Therefore, the averages of three tests from two different laboratories using the same materials and temperature conditions should not differ by more than 13 % of their average (Note 8).

NOTE 7—These numbers are in a strict sense not the single-operator (1s%) limits described in Practice C 670 because the results were obtained from separate batches made on different days.

NOTE 8—These numbers represent, respectively, the (1s%) and (d2s%) limits as described in Practice C 670.

12.6 The bias of this test method cannot be determined because times of setting can be defined only in terms of the test method.

13. Keywords

13.1 concrete; mortar; penetration resistance; time of final setting; time of initial setting

APPENDIX

(Nonmandatory Information)

X1. ILLUSTRATIVE EXAMPLES

X1.1 The penetration resistance (*PR*) and the elapsed time (*t*) data in Table X1.1 will be used to illustrate the procedures for determining times of setting.

X1.2 *Hand Fit*—Fig. X1.1 is a plot of the penetration resistance versus elapsed time values in Table X1.1. The smooth curve was drawn by-hand using a flexible drawing curve. The curve was drawn so as to achieve the visual best-fit to the data. Note that the penetration resistance at an elapsed time of 335 min is an obvious outlier, and this point was disregarded in drawing the best-fit curve. Horizontal lines are drawn at penetration resistance values of 500 and 4000 psi. The intersections of the horizontal lines with the curve define the times of initial and final setting, which in this case are 289 min and 389 min, respectively.

X1.3 *Regression Analysis:*

X1.3.1 Fig. X1.2 is a log-log plot of the penetration resistance versus elapsed time values. The plot shows that, with the exception of the outlier, there is approximately a straight line relationship between the logarithms of penetration resistance and elapsed time. The straight line is obtained by linear regression analysis using the logarithms shown in the third and fourth columns of Table X1.1. The equation for this line is:

$$\text{Log}\,(PR) = -14.196 + 6.871\,\text{Log}\,(t) \qquad \text{(X1.1)}$$

where:
PR = penetration resistance, and
t = elapsed time.

The correlation coefficient is 0.999, and it is, therefore, acceptable to use linear regression analysis.

X1.3.2 To obtain the times of setting, the equation is rewritten as:

$$\text{Log}\,(t) = \frac{\text{Log}(PR) + 14.196}{6.871} \qquad \text{(X1.2)}$$

X1.3.3 For time of initial setting, substitute the value 500 for *PR*:

$$\text{Log}\,(t) = \frac{\text{Log}(500) + 14.196}{6.871} = \frac{2.699 + 14.196}{6.871} = 2.458 \qquad \text{(X1.3)}$$

TABLE X1.1 Penetration Resistance

Penetration Resistance (*PR*) (psi)[A]	Elapsed Time (*t*) (min)	Log(*PR*)	Log(*t*)
44	200	1.643	2.301
110	230	2.041	2.362
216	260	2.334	2.415
540	290	2.732	2.462
1000	320	3.000	2.505
1000	335	3.000	2.525
2000	350	3.301	2.544
2560	365	3.408	2.562
3520	380	3.547	2.580
4440	395	3.647	2.597

[A] MPa = psi × 0.00689.

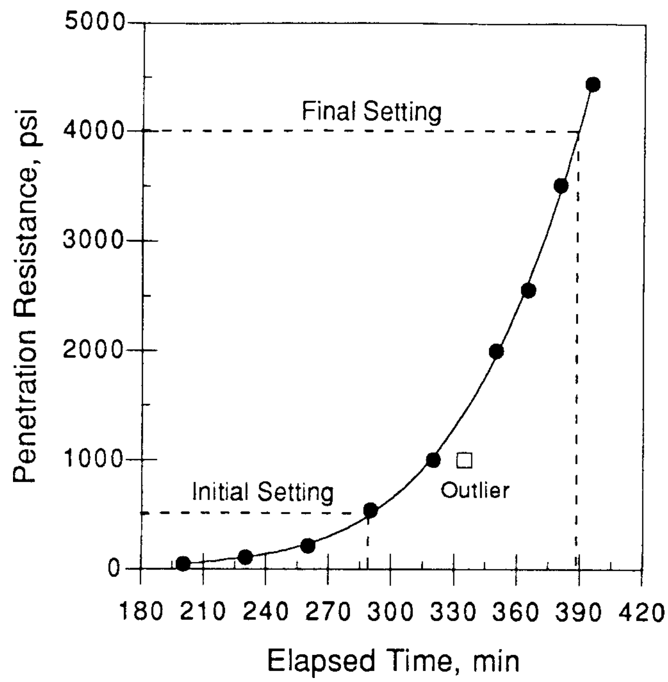

FIG. X1.1 Plot of Penetration Resistance Values Versus Elapsed Time and Hand Fit Curve Used to Determine Time of Setting (Note: Not drawn to actual scale)

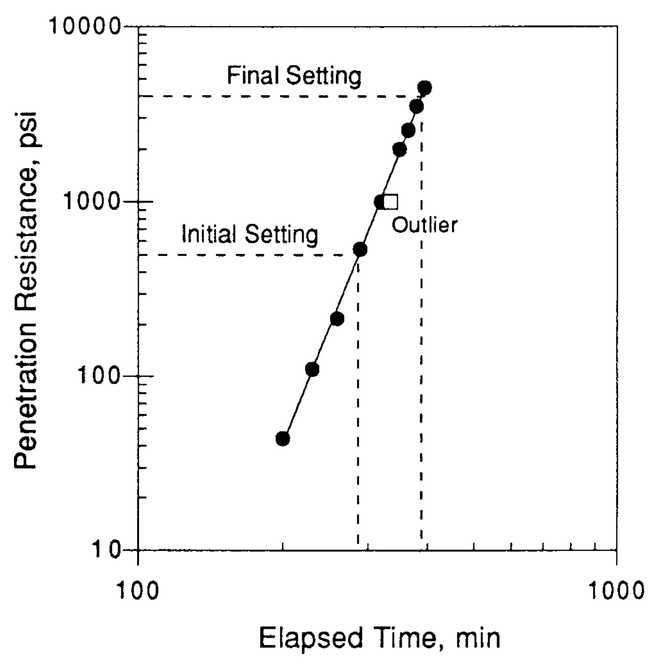

FIG. X1.2 Log-Log Plot Showing Straight Line to Determine Times of Setting by Using Regression Analysis

therefore:
$t = (10)^{2.458} = 287$ min.

X1.3.4 For time of final setting, substitute the value 4000 for *PR*:

$$\text{Log}\,(t) = \frac{\text{Log}(4000) + 14.196}{6.871} = \frac{3.602 + 14.196}{6.871} = 2.590 \quad \text{(X1.4)}$$

therefore:

$t = (10)^{2.590} = 389$ min.