Designation: C 289 – 94

Standard Test Method for Potential Alkali-Silica Reactivity of Aggregates (Chemical Method)[1]

This standard is issued under the fixed designation C 289; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers chemical determination of the potential reactivity of an aggregate with alkalies in portland-cement concrete as indicated by the amount of reaction during 24 h at 80°C between 1 *N* sodium hydroxide solution and aggregate that has been crushed and sieved to pass a 300-µm sieve and be retained on a 150-µm sieve.

1.2 The values stated in SI units are to be regarded as 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.* A specific precautionary statement is given in 5.7.1.

2. Referenced Documents

2.1 *ASTM Standards:*

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[2]

C 227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)[3]

C 295 Guide for Petrographic Examination of Aggregates for Concrete[3]

C 1005 Specification for Weights and Weighing Devices for Use in Physical Testing of Hydraulic Cements[2]

D 1193 Specification for Reagent Water[4]

D 1248 Specification for Polyethylene Plastics Molding and Extrusion Materials[5]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[6]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[7]

2.2 *American Chemical Society Documents:*

Reagent Chemicals, American Chemical Society Specifications

NOTE 1—For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

3. Significance and Use

3.1 This test method may be used in combination with other methods to evaluate the potential reactivity of siliceous aggregate with alkalies in portland-cement concrete. Reactions between a sodium hydroxide solution and siliceous aggregate have been shown to correlate with the performance of the aggregate in concrete structures and should be used where new aggregate sources are being evaluated or alkali-silica reactivity is anticipated.

3.2 The results from this test method can be obtained quickly, and, while not completely reliable in all cases, they provide useful data that may show the need for obtaining additional information through Test Method C 227 and Guide C 295.

4. Apparatus

4.1 *Scales*—The scales and weights used for weighing materials shall conform to the requirements prescribed in Test Method C 1005.

4.2 *Balances*—The analytical balance and weights used for determining dissolved silica by the gravimetric method shall conform to the requirements prescribed in Test Methods C 114.

4.3 *Crushing and Grinding Equipment*—A small jaw crusher and disk pulverizer or other suitable equipment capable of crushing and grinding approximately 4 kg of aggregate to pass a 300-µm sieve.

4.4 *Sieves*—300-µm and 150-µm square-hole, woven wire-cloth sieves conforming to Specification E 11.

4.5 *Containers*—Reaction containers of 50 to 75-mL capacity, made of corrosion-resistant steel or other corrosion-resistant material, and fitted with airtight covers. A container that has been found suitable is shown in Fig. 1. Other containers, made of corrosion-resistant material such as polyethylene, may be suitable. Such suitability can be demonstrated by a change in the alkalinity of the sodium hydroxide solution

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[1] This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregatesand is the direct responsibility of Subcommittee C09.26on Chemical Reactions of Materials.

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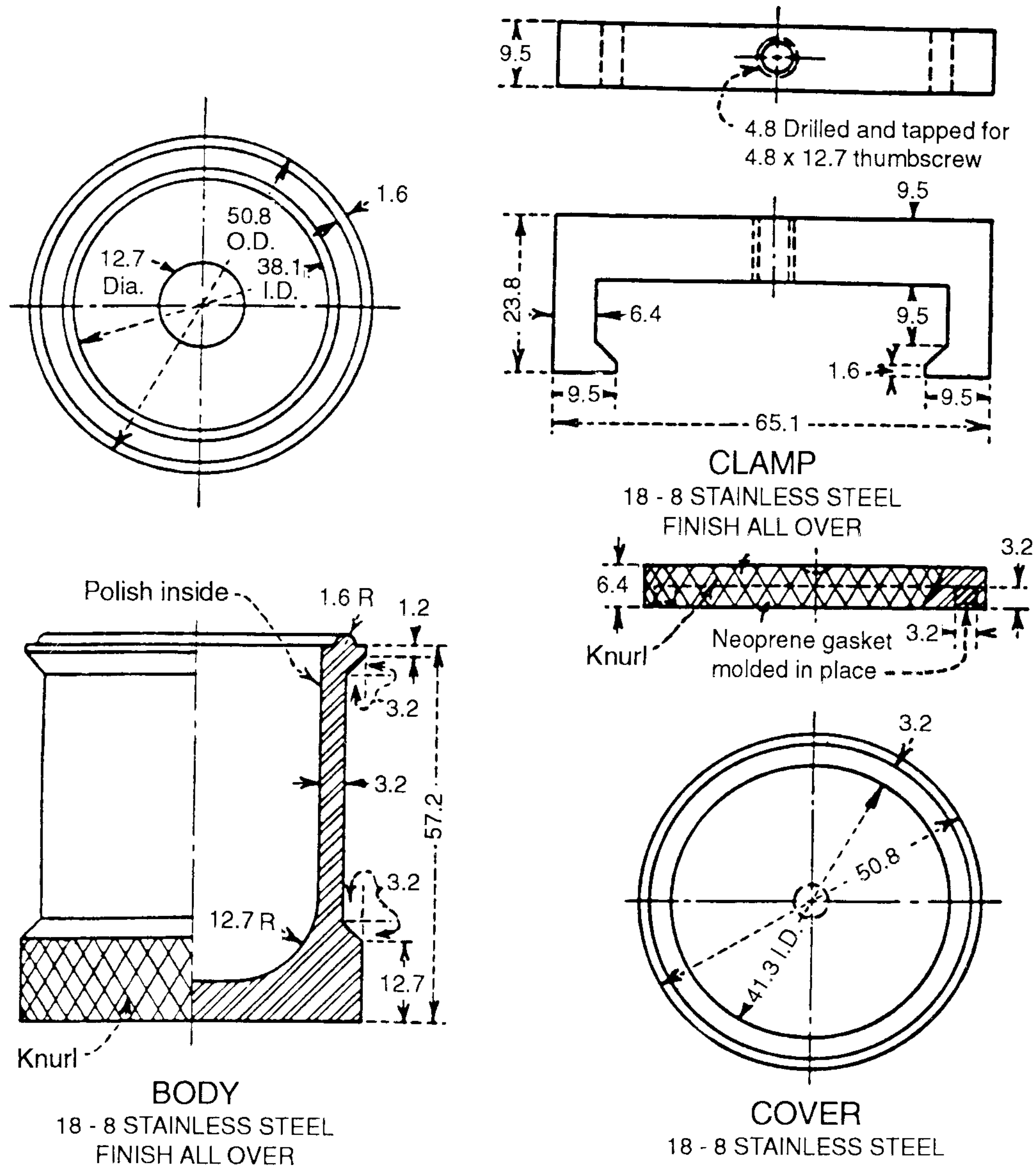
[2] *Annual Book of ASTM Standards*, Vol 04.01.
[3] *Annual Book of ASTM Standards*, Vol 04.02.
[4] *Annual Book of ASTM Standards*, Vol 11.01.
[5] *Annual Book of ASTM Standards*, Vol 08.01.
[6] *Annual Book of ASTM Standards*, Vol 14.02.
[7] *Annual Book of ASTM Standards*, Vol 03.05.



NOTE 1—All dimensions are in mm.

FIG. 1 Reaction Container

(R_c, Section on Reduction in Alkalinity) when used alone as a blank in the container in question, of less than 10 mmol/L.

4.6 *Constant-Temperature Bath*—A liquid bath capable of maintaining a temperature of 80 ± 1°C for 24 h.

4.7 *Spectrophotometer or Photometer*—A spectrophotometer or photoelectric photometer capable of measuring the transmission of light at a constant wavelength of approximately 410 nm (See Practice E 60).

4.8 *Glassware*—All glass apparatus and vessels should be carefully selected to meet the particular requirements for each operation. Standard volumetric flasks, burets, and pipets should be of precision grade.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

5.3 *Ammonium Molybdate Solution*—Dissolve 10 g of ammonium molybdate ($(NH_4)_6 Mo_7O_{24}\cdot 4H_2O$) in 100 mL of water. If the solution is not clear, filter through a fine-texture paper. Store the solution in a polyethylene container (Note 2).

5.4 *Hydrochloric Acid* (1.19 kg/L)—Concentrated hydrochloric acid (HCl). Store the solution in a chemically resistant glass or suitable plastic container (Note 2).

5.5 *Hydrochloric Acid, Standard* (0.05 *N*)—Prepare approximately 0.05 *N* HCl and standardize to ±0.0001 *N*. Store the solution in a chemically resistant glass or suitable plastic container (Note 2).

5.6 *Hydrochloric Acid* (1 + 1)—Mix equal volumes of concentrated HCl (1.19 kg/L) and water. Store the solution in a chemically resistant glass or suitable plastic container (Note 2).

5.7 *Hydrofluoric Acid* (approximately 50 % HF)—Concentrated hydrofluoric acid. Store in a polyethylene bottle (Note 2).

5.7.1 **Precaution**—Before using HF, review (*1*) the safety precautions for using HF, (*2*) first aid for burns, and (*3*) the emergency response to spills, as described in the manufacturer's Material Safety Data Sheet or other reliable safety literature. HF can cause very severe burns and injury to unprotected skin and eyes. Suitable personal protective equipment should always be used. These should include full-face shields, rubber aprons, and gloves impervious to HF. Gloves should be checked periodically for pin holes.

5.8 *Oxalic Acid Solution*—Dissolve 10 g of oxalic acid dihydrate in 100 mL of water. Store the solution in a chemically resistant glass or suitable plastic container (Note 2).

5.9 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (1 + 1). Store the solution in a chemically resistant glass or suitable plastic container (Note 2).

5.10 *Silica Standard Solution*—Prepare a standard silica solution containing approximately 10 mmol of silica (SiO_2)/L by dissolving sodium metasilicate in water. Store the solution in a polyethylene bottle. Use a 100-mL aliquot of the solution to determine its SiO_2 content by the procedure described in 8.1.1-8.2.1. Do not use a standard silica solution older than 1 year, since dissolved ionic silica in such a solution slowly polymerizes, causing spuriously low photometric readings (Note 2).

5.11 *Sodium Hydroxide, Standard Solution* (1.000 ± 0.010 *N*)—Prepare a 1.000 ± 0.010 *N* sodium hydroxide (NaOH) solution and standardize to ±0.001 *N*. Store the solution in a polyethylene bottle (Note 2). Protect the dry reagent and solution from contamination by carbon dioxide.

5.12 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4). Store the solution in a chemically resistant glass container (Note 2).

NOTE 2—In selecting the container, take care to ensure that the reagent will not be modified by reaction with the material composing the container, including pigments or other additives, or by transpiration of phases through the walls of the container. Containers with wall thickness not less than 0.51 mm and composed of high-density polyethylene meeting the requirements of Specification D 1248, for materials of Type III, Class A, are suitable.

6. Selection and Preparation of Test Samples

6.1 The test can be used for either fine or coarse aggregate, and when the fine and coarse aggregate are of the same material it can be used for the total aggregate.

6.2 The test sample shall be prepared from a representative portion of the aggregate by crushing so as to pass a 300-µm sieve, according to the following procedure (Note 3): Reduce the coarse aggregate to pass a 4.75-mm sieve by means of a small jaw crusher. Sieve the crushed coarse aggregate and likewise the sand to recover the 150-µm particles. Discard the material passing the 150-µm sieve. Reduce the material retained on the 300-µm sieve by repeated passes through a disk-type pulverizer, with sieving after each pass. The separation of the plates shall be about 3 mm for the first pass and shall be progressively diminished until all the material passes the 300-µm sieve. Every effort shall be made to reduce as much as possible the proportion of fines passing the 150-µm sieve.

NOTE 3—It is recommended that each size fraction of coarse aggregate be separately processed according to 6.2, and that the 300-µm to 150-µm material obtained from each size fraction be combined in the proportions in which those fractions are to be used in the concrete. It is recommended that, wherever possible, the sand be screened and the several size fractions recombined in the proportions to be used in the concrete, prior to processing according to 6.2.

6.3 To ensure that all material finer than the 150-µm sieve has been removed, wash the sample over a 150-µm sieve. Do not wash more than 100 g over a 203-mm diameter sieve at one time. Dry the washed sample at 105 ± 5°C for 20 ± 4 h. Cool the sample and again sieve on the 150-µm sieve. If inspection of the sample indicates the presence of silty or clayey coatings on particles, repeat the washing and drying procedure, and sieve as before over the 150-µm sieve. Reserve the portion retained on the 150-µm sieve for the test sample.

7. Reaction Procedure

7.1 Weigh out three representative 25.00 ± 0.05-g portions of the dry 150-µm to 300-µm test sample prepared in accordance with Section 6. Place one portion in each of the three of the reaction containers, and add by means of a pipet, 25 mL of the 1.000 *N* NaOH solution. To a fourth reaction container, by means of a pipet, add 25 mL of the same NaOH solution to serve as a blank. Seal the four containers and gently swirl them to liberate trapped air.

7.2 Immediately after the containers have been sealed, place them in a liquid bath maintained at 80 ± 1.0°C. After 24± ¼ h, remove the containers from the bath and cool them, for 15 ± 2 min, under running tap water having a temperature below 30°C.

7.3 Immediately after the containers have been cooled, open them and filter the solution from the aggregate residue. Use a porcelain Gooch crucible (Note 4) with a disk of rapid, analytical-grade filter paper cut to fit the bottom of the crucible, setting the crucible in a rubber crucible holder in a funnel. Place a dry test tube, 35 to 50-mL capacity, in the filter flask to collect the filtrate, and seat the funnel in the neck of the filter flask. With the aspirator in operation or the vacuum line open, decant a small quantity of the solution onto the filter paper so it will seat properly in the crucible. Without stirring the contents of the container, decant the remaining free liquid into the crucible. When the decantation of the liquid has been completed, discontinue the vacuum and transfer the solids remaining in the container to the crucible and pack in place with the aid of a stainless-steel spatula. Then apply and adjust the vacuum to approximately 51 kPa. Continue the filtration until further filtration yields filtrate at the approximate rate of 1 drop every 10 s; reserve the filtrate for further tests. Record the total amount of time during which the vacuum is applied as the filtration time; make every effort to achieve an equal filtration time for all samples in a set, by uniformity of procedure in the assembly of the filtration apparatus and the packing of the solids in the crucible.

NOTE 4—Coors Size No. 4 Gooch crucibles, or equivalent, have been found satisfactory for this purpose.

7.4 Filter the blank according to the procedure described in 7.3. Apply the vacuum for a length of time equal to the average filtration time for the three specimens.

7.5 Immediately following the completion of filtration, stir the filtrate to assure homogeneity, then take by pipet an aliquot of 10 mL of the filtrate and dilute with water to 200 mL in a volumetric flask. Reserve this diluted solution for the determination of the dissolved SiO_2 and the reduction in alkalinity.

7.6 If the diluted filtrate is not to be analyzed within 4 h following completion of the filtration, transfer the solution to a clean, dry polyethylene container and close the container by means of a stopper or tight-fitting cap or lid.

8. Dissolved Silica by the Gravimetric Method

8.1 *Procedure*:

8.1.1 Transfer by pipet 100 mL of the dilute solution (7.5) to an evaporating dish, preferably of platinum for speed in evaporation, add 5 to 10 mL of HCl (1.19 kg/L), and evaporate to dryness on a steam bath. Without heating the residue further, treat it with 5 to 10 mL of HCl (1.19 kg/L) and then an equal amount of water, or at once pour 10 to 20 mL of HCl (1 + 1) upon the residue. Cover the dish and digest for 10 min on the steam bath or a hot plate. Dilute the solution with an equal volume of hot water, filter immediately through quantitative-grade, low–ash filter paper, and wash the separated silica (SiO_2) thoroughly with hot water (Note 5) and reserve the residue.

NOTE 5—The washing of the SiO_2 precipitates can be made more effective by using hot HCl (1 + 99) and then completing the washing with hot water.

8.1.2 Again evaporate the filtrate to dryness, baking the residue in an oven for 1 h at 105 to 110°C. Take up the residue with 10 to 15 mL of HCl (1 + 1) and heat on the bath or hot plate. Dilute the solution with an equal volume of hot water and catch and wash the small amount of SiO_2 it contains on another filter paper. This second evaporation is necessary only when determining the concentration of the standard sodium metasilicate solution in 5.10. For the other test solutions, it can be eliminated.

8.1.3 Transfer the papers containing the residue (8.1.1 and 8.1.2) to a platinum crucible (). Dry and ignite the papers, first at a low heat until the carbon of the filter paper is completely consumed without inflaming, and finally at 1100 to 1200°C until the mass remains constant.

NOTE 6—The mass of the empty crucible may be determined if one wishes to know the magnitude of impurities in the residue of SiO_2.

8.1.4 Treat the SiO_2 thus obtained, which will contain small amounts of impurities, in the crucible with a few drops of water, about 10 mL of HF, and one drop of H_2SO_4, and evaporate cautiously to dryness on the steam bath. Finally, heat the small residue at 1050 to 1100°C for 1 to 2 min, cool, and determine the mass. The difference between this determination and that previously obtained represents the amount of SiO_2.

8.2 *Calculation*:

8.2.1 Calculate the SiO_2 concentration of the NaOH solution filtered from the aggregate material, as follows:

$$S_c = 3330 \times W \quad (1)$$

where:

S_c = concentration of SiO_2 in mmol/L in the original filtrate, and

W = grams of SiO_2 found in 100 mL of the dilute solution.

9. Dissolved Silica by the Photometric Method

9.1 *Application*:

9.1.1 This method is applicable to the determination of crystalloidal (noncolloidal) silica (Note 7) in all aqueous solutions except those with excessive color interferences (tannin, etc.), but it will not determine total silica. The method is particularly applicable to rapid control analysis of crystalloidal silica below 10 ppm.

NOTE 7—Crystalloidal (noncolloidal) silica reacts with molybdate ion in acid solution (optimum pH 1.2 to 1.5) to form a greenish yellow silico-molybdate color complex the intensity of which is approximately proportional to the silica concentration of the solution, but does not follow Beer's law perfectly.

10. Preparation of Calibration Curve

10.1 Prepare a series of solutions of known silica concentration varying from 0.0 to 0.5 mmol/L by diluting portions of the stock solution of sodium silicate (5.10). Transfer the portions of sodium silicate solution to 100-mL volumetric flasks about half filled with water.

10.2 Add 2 mL of the ammonium molybdate solution and 1 mL of HCl (1 + 1), and agitate by swirling the flask. Allow the solution to stand for 15 min at room temperature. Add 1.5 ± 0.2 mL of the oxalic acid solution, fill the flask to the mark with water, and mix thoroughly. Allow the solution to stand for 5.0 ± 0.1 min. Read the transmittance of the various solutions on the photometer at 410 nm, in comparison with that of water.

10.3 Prepare a calibration curve by plotting the percent transmittance or absorbance readings against the known concentrations of silica in each solution.

11. Determination of Dissolved Silica

11.1 Transfer by pipet a 10-mL aliquot of the dilute solution to a 100-mL volumetric flask half filled with water and proceed as directed in 10.2 and 10.3. Read the concentration of silica in the solution directly from the previously prepared calibration curve correlating transmission of light of this wave length with silica concentration. If the transmittance is below 30 % or above 50 %, a smaller or larger aliquot of the diluted solution shall be used.

12. Calculation

12.1 Calculate the SiO_2 concentration of the NaOH solution filtered from the aggregate material, as follows:

$$S_c = 20 \times (100/V) \times C \quad (2)$$

where:

S_c = concentration of SiO_2, mmol/L in the original filtrate,
C = concentration of silica in the solution measured in the photometer, mmol/L, and
V = millilitres of dilute solution used from 7.5.

13. Reduction in Alkalinity

13.1 *Procedure*:

13.1.1 Transfer by pipet a 20-mL aliquot of the dilute solution (7.5) to a 125-mL Erlenmeyer flask, add 2 or 3 drops of phenolphthalein solution, and titrate with 0.05-*N* HCl to the phenolphthalein end point.

13.2 *Calculation*:

13.2.1 Calculate the reduction in alkalinity as follows:

$$R_c = (20N/V_1)(V_3 - V_2) \times 1000 \quad (3)$$

where:

R_c = the reduction in alkalinity, mmol/L,
N = normality of the HCl used for the titration,
V_1 = millilitres of dilute solution used from 7.5,
V_2 = millilitres of HCl used to attain the phenolphthalein end point in the test sample, and
V_3 = millilitres of HCl used to attain the phenolphthalein end point in the blank.

14. Precision and Bias

14.1 *Precision*—Information concerning the precision of this test method is being investigated and will be published when the proper data have been obtained and analyzed as prescribed in Practice C 670.

14.2 Preliminary data on precision indicate that the test results may be considered satisfactory if none of the three values of R_c (and of S_c) differs from the average of the three by more than the following amounts: (*1*) when the average is 100 mmol or less, 12 mmol/L, and (*2*) when the average is more than 100 mmol/L, 12 %.

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

15. Keywords

15.1 alkali; aggregate reactivity; alkali-silica reactivity; concrete aggregates

APPENDIX

(Nonmandatory Information)

X1. INTERPRETATION OF RESULTS

X1.1 Correlations between data obtained by this method, expansion of mortar bars containing high-alkali cement, petrographic examinations of aggregates, and performance of aggregates in concrete structures have been published **(1-7)**.[8] On the basis of these data, the solid curve shown in Fig. X1.1 has been established. A potentially deleterious degree of alkali reactivity is indicated if any of the three R_c, S_c points lie on the deleterious side of the curve in Fig. X1.1. However, potentially deleterious aggregates represented by points lying above the dashed line in Fig. X1.1 may give relatively low expansions in mortar or concrete even though they are extremely reactive with alkalies. These aggregates should be considered to indicate a potentially deleterious degree of reactivity until the innocuous character of the aggregate is demonstrated by service records or by supplementary tests in accordance with Test Method C 227, using several mixtures of the test aggregate and an essentially inert aggregate such as properly graded unstrained quartz or chert-free limestone. The mixtures should represent a series of proportions of the test aggregate and essentially inert aggregate ranging from about 5:95 to 50:50 by mass.

X1.2 Results of the test may not be correct for aggregates containing carbonates of calcium, magnesium, or ferrous iron, such as calcite, dolomite, magnesite, or siderite; or silicates of magnesium such as antigorite (serpentine) **(6,7)**. The error introduced by calcium carbonate is not significant unless S_c and R_c values indicate the potential reactivity is marginal. Examinations of the aggregate in accordance with Guide C 295, can be used to determine the presence of minerals of this type. In order to evaluate these possible effects, testing in accordance with Test Method C 227 is recommended.

X1.3 It is recommended that interpretations based upon this method be correlated with Guide C 295 and service records of the aggregate. The results of this test do not predict the late-slow silica-silicate reactivity in concrete that may result with aggregates containing strained or micro-granulated quartz, or aggregates composed of metagraywacke, metasiltstone, meta-quartz, and similar rocks.

[8] The boldface numbers in parentheses refer to the references appearing at the end of this test method.

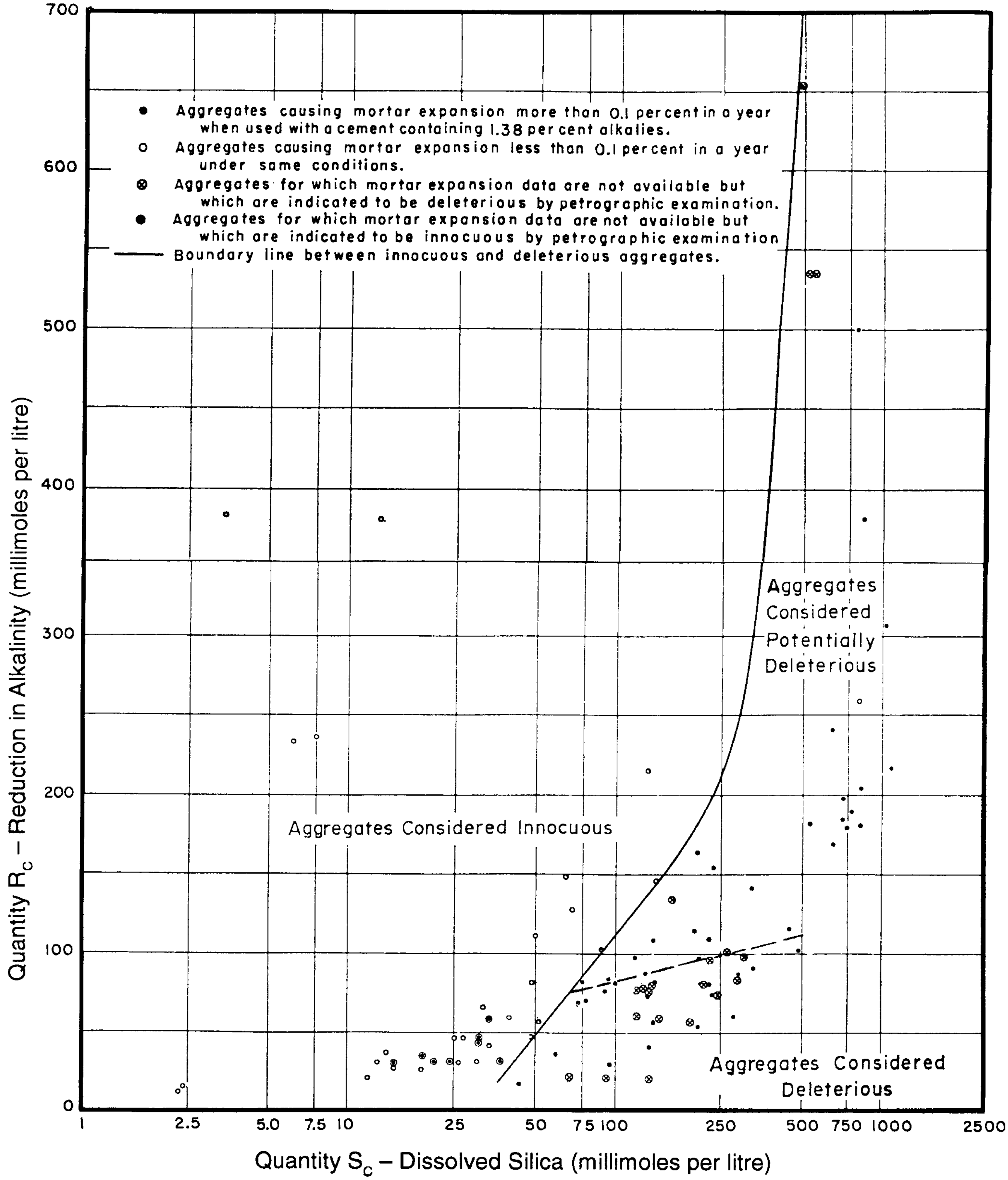


FIG. X1.1 Illustration of Division Between Innocuous and Deleterious Aggregates on Basis of Reduction in Alkalinity Test

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(**2**) Mielenz, R. C., Greene, K. T., and Benton, E. J., "Chemical Test for Reactivity of Aggregates with Cement Alkalies: Chemical Processes in Cement-Aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol 44, 1948, p. 193.

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(**7**) Chaiken, Bernard, and Halstead, W. J., "Correlation Between Chemical and Mortar Bar Tests for Potential Alkali Reactivity of Concrete Aggregates," *Public Roads*, Vol 30, 1959, p. 177.