



Standard Test Method for Insoluble Residue in Carbonate Aggregates¹

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1. Scope

1.1 This test method covers determination of the percentage of insoluble residue in carbonate aggregates using hydrochloric acid solution to react the carbonates.

1.2 The values stated in inch-pound 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.* Specific precaution statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates²

C 294 Descriptive Nomenclature for Constituents of Natural Mineral Aggregates²

C 670 Practice for Preparing Precision and Bias Statements for Test Methods of Construction Materials²

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and Related Construction Materials³

E 11 Specification for Wire-Cloth and Sieves for Testing Purposes⁴

3. Significance and Use

3.1 The amount and size distribution of noncarbonate (insoluble) material in carbonate aggregates is of interest to those conducting research on the polish susceptibility of stone used in pavements or on the friction properties of pavement surfaces. In addition, some agencies utilize the test for acceptance purposes to delineate carbonate aggregates that may polish excessively and become slippery and those that will not.

4. Apparatus

4.1 *Sieves*, U.S. standard 8-in. (203.2-mm) diameter, con-

forming to Specification E 11 in the following sizes:

$\frac{3}{8}$ -in.	(9.5-mm)
No. 4	(4.75-mm)
No. 8	(2.36-mm)
No. 16	(1.18-mm)
No. 30	(600- μ m)
No. 50	(300- μ m)
No. 100	(150- μ m)
No. 200	(75- μ m)

4.2 *Balance*—A balance having a minimum capacity of 15 kg and meeting the requirements of Specification D 4753, Class GP5.

4.3 *Oven*, of appropriate size capable of being maintained at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$).

4.4 *Agitation Equipment*, such as a jar mill or a magnetic stirrer.

4.5 *Bunsen Burner or Hot Plate*.

4.6 *Receiving Tank*, of nonreactive material to receive wash water and residue washed through the 8-in. (203.2-mm) diameter sieves.

4.7 *Rapid Filter Paper*, acid-resistant.

4.8 *Safety Eyeglasses, Rubber Gloves, Aprons, etc.*

4.9 *Borosilicate Glass Containers*, for performing acid leaching test.

4.10 *pH Paper*.

5. Reagent

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ 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 *Hydrochloric Acid (6 N)*—Prepare a 6 N solution of hydrochloric acid (HCl).

6. Samples

6.1 Samples shall be representative of the sources from which they are obtained and shall be reduced to appropriate

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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 14.02.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. 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."

size by use of sample splitters or by quartering in accordance with the Sampling Section of Test Method C 136.

6.1.1 Normally, the aggregate used in the test sample shall be retained after washing over the No. 4 (4.75-mm) sieve, and passing the $\frac{3}{8}$ -in. (9.5-mm) sieve).

6.1.2 Separate fractions or combined aggregate gradations may be tested as appropriate.

6.2 Sample aggregates shall be dried to constant mass at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$).

6.3 An oven-dry sample weighing a minimum of 500 g shall be used for the test.

7. Procedure for Determining Only the Plus No. 200 Size Fraction of Insoluble Residue

7.1 For many applications, only the plus No. 200 (75- μm) size insoluble residue fraction is of interest. This section provides a procedure for determination of the amount and size distribution of only the plus No. 200 (75- μm) size insoluble residue. If the total amount of insoluble residue is desired, the procedure given in Section 8 should be followed and the remainder of this section should be omitted.

7.2 Prepare and test samples in triplicate for each aggregate sampled.

7.3 **Warning**—Hydrogen chloride gas is released during this procedure. Operators shall be required to wear a mask and should perform this test using the procedures set forth in 7.4.

7.4 Place an aggregate sample weighing a minimum of 500 g in a suitable borosilicate glass container. The container shall be vented to allow gas from the reaction to escape and to be drawn away. Exercise extreme care to prevent corrosive gas from entering the laboratory atmosphere. The hydrogen chloride gas that may be produced in the chemical reaction can be destructive to laboratory equipment and can also be a definite safety hazard to laboratory personnel. Under no circumstances shall the test be performed without proper and adequate ventilation. Slowly add 1000 mL of 6 *N* hydrochloric acid (HCl) allowing for excessive effervescence or foaming to subside before further addition of solution. Agitate the container and contents gently by hand until excessive effervescence has subsided.

7.5 Place the container and contents on an agitating device.

7.6 Check the container periodically, and when the reaction appears to have subsided (no obvious bubbling) decant and add approximately 300 mL of the 6 *N* HCl and check for a reaction. Collect all decant solution in a receiving tank for future use only if determination of the total residue content is desired as in Section 8.

7.6.1 If a reaction (bubbling) is observed, continue agitation until the reaction subsides and repeat the procedure beginning at 7.6.

7.7 When no reaction is observed, heat the container gently over a bunsen burner or hot plate. Some carbonate materials will require heating to temperatures of up to 230°F (110°C) and maintaining this temperature level for approximately 1 h in order to completely react the carbonate fraction.

7.7.1 If no reaction is observed upon heating, proceed with the remainder of the test procedure.

7.7.2 If a reaction is observed upon heating, continue the agitation and repeat the procedure beginning at 7.7.

7.8 When the reaction has stopped, decant the acid solution and add water to the aggregate in the glass container to thoroughly dilute the acid. The residue in the container should have a pH as indicated by pH paper of more than 5.5.

7.9 Wash the diluted solution and residue from the aggregate sample over a preweighed No. 200 sieve placed over a receiving tank.

7.10 Thoroughly rinse the material on the No. 200 (75- μm) sieve and into the receiving tank. Dry the sieve and the residue in a $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) oven.

7.11 When the sieve and residue have cooled to room temperature, weigh the sieve plus the residue and subtract the original mass of the dry clean sieve to obtain the mass of the plus No. 200 (75- μm) sieve size residue.

7.12 Visually inspect the residue to determine whether or not particle agglomeration because of the presence of plastic clay fractions has occurred.

7.12.1 If agglomeration has occurred, physical reduction of the agglomerated particles should be performed by use of a mortar and rubber-tipped pestle. Care should be exercised to prevent crushing of individual particle grains during this process.

7.12.2 If no agglomeration has occurred, proceed with the remainder of the test procedure.

7.13 Dry sieve the residue from the aggregate sample over preweighed nested sieves. The nested sieves shall be of the following series:

No. 4	(4.75-mm)
No. 8	(2.36-mm)
No. 16	(1.18-mm)
No. 30	(600- μm)
No. 50	(300- μm)
No. 100	(150- μm)
No. 200	(75- μm)

8. Procedure for Determining the Total Acid Insoluble Residue Content

8.1 Perform the steps in 7.1 through 7.8.

8.2 Wash the diluted solution and residue from the aggregate sample over pre-weighed nested sieves placed over a receiving tank. The nested sieves shall be of the following series:

No. 4	(4.75-mm)
No. 8	(2.36-mm)
No. 16	(1.18-mm)
No. 30	(600- μm)
No. 50	(300- μm)
No. 100	(150- μm)
No. 200	(75- μm)

8.3 Rinse each sieve with water individually and thoroughly over the lower sieves and into the receiving tank. Dry the sieves and the residue in a $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) oven.

8.4 When the sieves have cooled to room temperature, weigh each sieve plus the residue and subtract the original mass of the dry clean sieve to obtain the mass of the residue.

8.5 Dilute the decant acid solution in the receiving tank with water. The solution in the receiving tank should have a pH as indicated by pH paper (or a pH meter) of more than 5.5.

8.6 Pour the solution contained in the receiving tank through a No. 200 sieve (75- μm) and then through rapid filtering paper placed in glass funnels. Thoroughly rinse with

additional water. Preweigh the filter papers and the evaporating dish.

8.7 When filtration is complete, rinse by filtering clean water, then transfer the paper and the residue into an evaporating dish and place in the oven at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) for 24 h.

8.8 Weigh the dish, dried filters, and residue. Subtract the mass of the clean filters and dish from the mass of the dish, filters, and residue to obtain the mass of the residue passing the No. 200 (75- μm) sieve.

8.9 Dry the No. 200 (75- μm) sieve and residue obtained during filtering process (see 8.6) in a $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) oven.

8.10 When the No. 200 (75- μm) sieve and residue have cooled to room temperature, weigh the sieve plus the residue and subtract the original mass of the dry clean sieve to obtain the mass of the residue (if any) retained on the No. 200 sieve from the decant solution. This quantity should be added to the quantity established in 8.2.

8.11 Examine the residue and describe it with the aid of such mineral and rock identification tests as are necessary and appropriate. Refer to Descriptive Nomenclature C 294.

9. Calculation

9.1 Calculate the cumulative percent of insoluble residue retained on each of the sieves listed in 8.2 expressed as a percentage of that portion of the original sample mass retained on the No. 200 (75- μm) sieve.

9.2 Calculate the total percent of insoluble residue expressed as a percentage of the total original aggregate sample mass.

9.3 Calculate the cumulative percent of insoluble residue retained on each of the sieves listed in 8.2 expressed as a percentage of the total original aggregate sample mass.

10. Report

10.1 Report the following information:

10.1.1 Identification and description of the aggregate samples, and

10.1.2 Calculated results for each sample and average results for triplicate tests.

11. Precision and Bias ⁶

11.1 Precision has been established only for determination of the plus No. 200 (75- μm) size fraction.

11.2 Tests of three materials having widely different levels of insoluble residue indicated a non-linear relationship of insoluble residue between standard deviations and the amounts contained in the samples. These data are shown in Appendix X1.

11.3 *Precision Based on Weighted Average Variance for All Materials:*

11.3.1 *Single-Operator Precision*—The single-operator standard deviation (one-sigma limit) has been found to be 1.5 %.⁷ Therefore, results of two properly-conducted tests by the same operator should not differ by more than 4.2.⁷ The test method calls for reporting three test results. The range (difference between highest and lowest) of the three test results obtained by the same operator should not exceed 4.9.⁸

11.3.2 *Multilaboratory Precision*—The multilaboratory standard deviation (one-sigma limit) has been found to be 3.1 percent.⁷ Therefore, results of two properly-conducted tests in different laboratories on the same material should not differ by more than 8.9.⁷ The averages of three test results in two different laboratories should not differ by more than 5.1.⁸

11.4 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring the insoluble residue in carbonate aggregates, because no material having an accepted reference value is available.

12. Keywords

12.1 carbonate aggregates; friction; insoluble residue; polish susceptibility

⁶ A Research Report is available from ASTM headquarters. Request RR: D04-1009.

⁷ These numbers represent the (1s) and (d2s) limits as described in Practice C 670.

⁸ Calculated as described in the Descriptions of Terms of Practice C 670.

APPENDIX

(Nonmandatory Information)

X1. PRECISION AT DIFFERENT INSOLUBLE RESIDUE LEVELS

X1.1 Three carbonate aggregates, selected to represent low, intermediate, and high insoluble residue contents were tested. Data analysis was confined to the plus No. 200 size fraction of the insoluble residue.

X1.2 The precision was found to vary with insoluble residue level, with the intermediate level having the highest variability. Data analyses by individual material provides the results: described in Table X1.1 and Table X1.2.

TABLE X1.1 Single-Operator Precision

Average Insoluble Residue, %	Number of Labs	Single-Operator Standard Deviation (1s) Limit ^A	Acceptable Difference Between Two Results (d2s) ^A	Acceptable Range of Three Results ^B
4.7	15	0.9	2.6	3.0
25.3	19	2.0	5.6	6.6
48.2	19	1.2	3.4	4.0

^A These numbers represent the (1s) and (d2s) limits as described in Practice C 670.

^B Calculated as described in the Descriptions of Terms of Practice C 670.

TABLE X1.2 Multilaboratory Precision

Average Insoluble Residue, %	Number of Labs	Multilaboratory Standard Deviation (1s) Limit ^A	Allowable Differences Between Two Laboratories	
			Single Test Results (d2s) ^A	Average of Three Tests ^B
4.7	15	0.9	2.7	1.5
25.3	19	4.4	12.5	7.2
48.2	19	2.7	7.7	4.5

^A These numbers represent the (1s) and (d2s) limits as described in Practice C 670.

^B Calculated as described in the Descriptions of Terms of Practice C 670.

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