



Designation: C641 – 17

Standard Test Method for Iron Staining Materials in Lightweight Concrete Aggregates¹

This standard is issued under the fixed designation C641; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method covers the testing of lightweight concrete aggregates to evaluate the potential degree of staining from iron compounds.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound values given in parentheses are provided for information only.

1.3 *This standard does not purport to address the safety problems 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.*

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

C125 Terminology Relating to Concrete and Concrete Aggregates

C330/C330M Specification for Lightweight Aggregates for Structural Concrete

C331/C331M Specification for Lightweight Aggregates for Concrete Masonry Units

¹ This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.21 on Lightweight Aggregates and Concrete.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C702/C702M Practice for Reducing Samples of Aggregate to Testing Size

D75/D75M Practice for Sampling Aggregates

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E832 Specification for Laboratory Filter Papers

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology **C125**.

4. Significance and Use

4.1 This test method evaluates the potential degree of staining attributable to the presence of iron compounds in a lightweight aggregate sample primarily by means of a visual classification method. Such compounds may or may not produce stains on the surface of the concrete in which the aggregate is incorporated.

5. Apparatus

5.1 **Balance**—A balance or scale accurate to within 0.1 % of the test load at any point within the range of use.

5.2 **Sieves**—9.5-mm (3/8-in.) and 600-μm (No. 30) sieves conforming to Specification **E11**.

5.3 **Filter Paper**—shall comply with Specification **E832** class E2 with a minimum diameter of 240 mm.

5.4 **Cheesecloth Wrapping**—Two thicknesses, reagent grade cheesecloth, approximately 457 mm (18 in.) square is sufficient for wrapping each sample.

5.5 **Steam Bath**—Any suitable apparatus that will meet the requirement of the test procedure. Water in the steam bath, and make-up water, shall be iron-free water or distilled water.

NOTE 1—An oven top glassware sterilizer made of nonferrous materials is satisfactory.

*A Summary of Changes section appears at the end of this standard

6. Reagents

6.1 *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.³

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

6.3 Concentration of Reagents:

6.3.1 *Concentrated Acid and Ammonium Hydroxide*—When reagents are specified by name it shall be understood that concentrated reagents of the following specific gravity are intended:

Hydrochloric acid (HCl)	sp gr 1.19
Ammonium hydroxide (NH ₄ OH)	sp gr 0.90

6.3.2 *Diluted acid* is described in terms of the number of volumes of the concentrated reagent to be added to a given number of volumes of water. Thus HCl (1 + 2) means 1 volume of HCl (sp gr 1.19) added to 2 volumes of water.

7. Sampling

7.1 Sample in accordance with Practice **D75/D75M**.

7.2 After reducing a field sample to an appropriate size in accordance with Practice **C702/C702M**, thoroughly dry the

aggregate and prepare by sieving material to pass the 9.5-mm (3/8-in.) sieve and is retained on the 600-μm (No. 30) sieve.

8. Procedure

8.1 Select two portions each weighing 100 g from the aggregate sample prepared for test.

8.2 Crimp the edges of two filter papers to form cup-shaped receptacles approximately 130 mm (5 in.) in diameter and 60 mm (2½ in.) in depth. Place one of the 100-g portions in each filter cup, spreading to a uniform depth. Fold the sides of the cup to the center and press in that position.

8.3 Wrap both portions of the prepared sample, one on top of the other, in cheesecloth. Saturate with distilled water and expose to steam in the steam bath for 16 h ± 30 min, adding distilled water as make-up water as required.

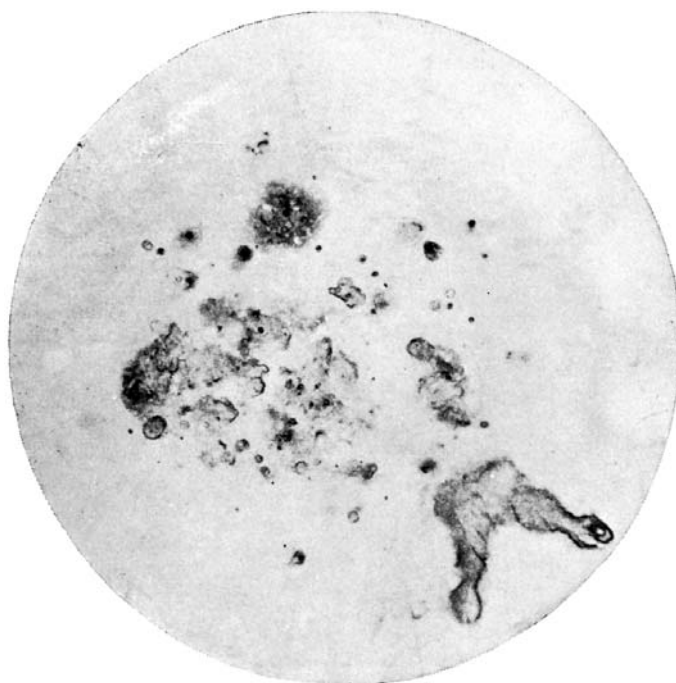
8.4 Remove from the steam bath, and carefully remove the aggregate from the filter papers. Wash both papers in water, place on a watch glass, and oven dry at a temperature of 110 ± 5 °C (230 ± 9 °F). The insoluble products of the decomposition of iron compounds in the aggregate will be deposited on the filter paper as red, green, or black stains.

8.5 Rate the extent of staining on the filter papers by the Visual Classification Method.

8.5.1 *Visual Classification Method*—Evaluate the extent and intensity of the stains on the filter paper in accordance with the photographic stain index reference standards shown in **Figs. 1-5**: The photographic stain index ranges from No Stain (stain index = 0) to a Very Heavy Stain (stain index = 100).

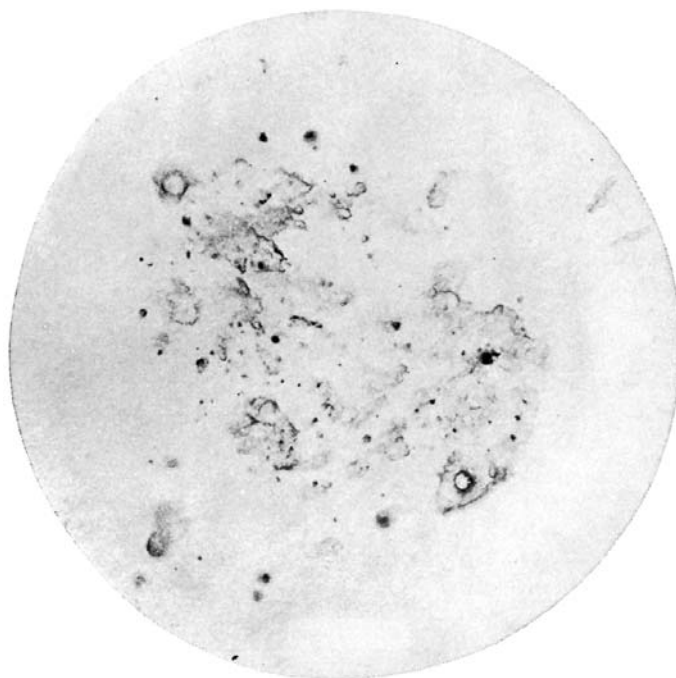
8.6 When required by Specification **C330/C330M** and Specification **C331/C331M**, follow the procedure of the Chemical Analysis Method.

³ "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."



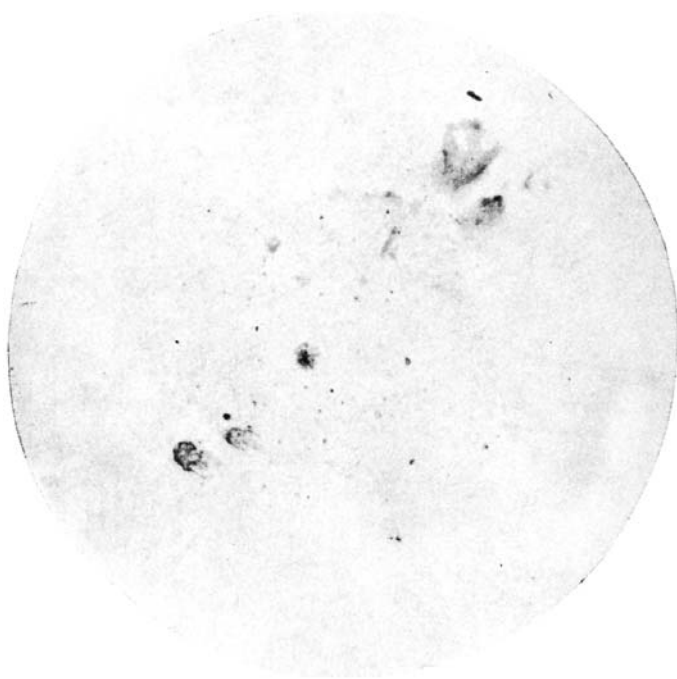
Stain Index = 100

FIG. 1 Visual Degree of Staining



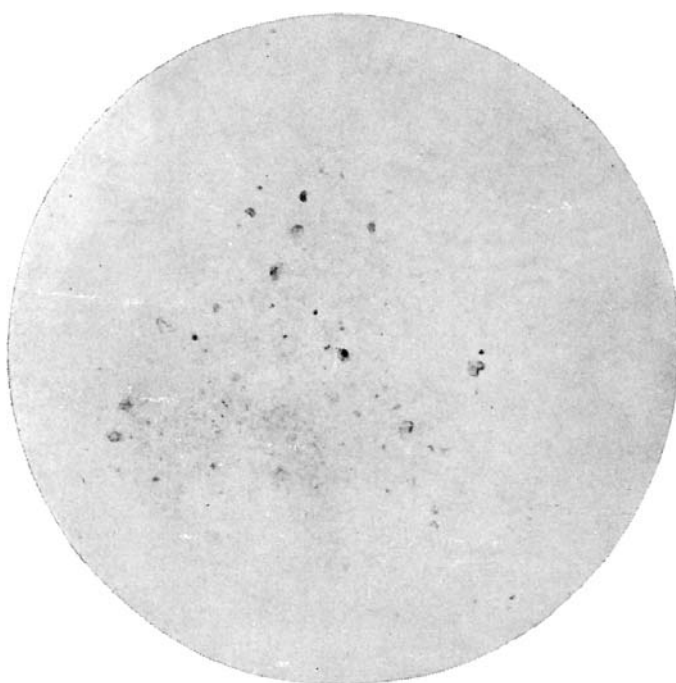
Stain Index = 80

FIG. 2



Stain Index = 60

FIG. 3



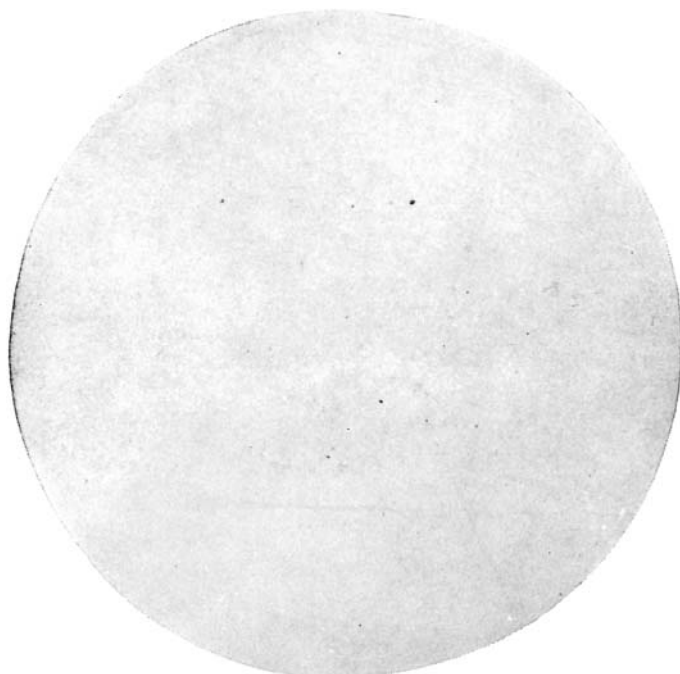
Stain Index = 40

FIG. 4

8.6.1 Chemical Analysis Method—The iron deposits may be dissolved from the filter papers by careful application of HCl from a dropping bottle and rinsing with hot distilled water from a wash bottle. Otherwise, dissolve the iron compound on the washed and dried filter papers by digesting in HCl (1 + 1) and filtering out the residue of filter pulp washing thoroughly with hot water. Precipitate the iron in the filtrate as ferric hydroxide $\text{Fe}(\text{OH})_3$ by adding NH_4OH dropwise to neutralize

the acid using methyl red indicator solution. Redissolve the $\text{Fe}(\text{OH})_3$ precipitate using 10 cm^3 of HCl (1 + 1) and determine the iron quantitatively as Fe_2O_3 by standard titration procedures.

NOTE 2—The iron may be determined by using other standard quantitative procedures.



Stain Index = 20

FIG. 5

9. Calculation

9.1 Calculate the determined Fe_2O_3 to the nearest 0.01 mg (to be reported to the nearest 0.1) as follows:

$$\text{Fe}_2\text{O}_3, \text{ mg}/200 \text{ g} = E \times V \quad (1)$$

where:

E = Fe_2O_3 equivalent of standard titration solution, mg/mL,

V = millilitres of standard titration solution required by the stains from the 200-g sample.

10. Report

10.1 The report shall include the following:

10.1.1 Identification of the sample,

10.1.2 Stain index, as evaluated by the Visual Classification Method, when required, and

10.1.3 Iron content as milligrams of Fe_2O_3 per 200 g of sample evaluated by the Chemical Analysis Method.

11. Precision and Bias

11.1 *Precision*—The precision of this test method has not yet been determined, but an interlaboratory testing program is underway. The precision statements will be included when the data are compiled and reviewed.

11.2 *Bias*—Bias for this test method cannot be determined since there is no reference standard available for comparison.

12. Keywords

12.1 iron; lightweight aggregate; staining

SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this test method since the last issue, C641 – 09, that may impact the use of this test method. (Approved June 15, 2017.)

(1) Revised minimum filter paper size in 5.3.

(2) Added tolerances to time in 8.3.

(3) Added terminology section.

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