Standard Test Method for Iron Staining Materials in Lightweight Concrete Aggregates¹

This standard is issued under the fixed designation C 641; 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.

 ϵ^1 Note—Editorial corrections were applied to Figure 3 and 9.1.2.

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.

2. Referenced Documents

- 2.1 ASTM Standards:
- C 330 Specification for Lightweight Aggregates for Structural Concrete²
- C 331 Specification for Lightweight Aggregates for Concrete Masonry Units²
- C 702 Practice for Reducing Field Samples of Aggregate to Testing Size²
- D 75 Practice for Sampling Aggregates²
- E 11 Specification for Wire-Cloth and Sieves for Testing Purposes²

3. Significance and Use

3.1 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.

4. Apparatus

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

- ¹ This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregatesand is the direct responsibility of Subcommittee C09.21on Lightweight Aggregates.
- Current edition approved Nov. 10, 1998. Published May 1999. Originally published as C 641 69. Last previous edition C 641 82 (1991).
 - ² Annual Book of ASTM Standards, Vol 04.02.

- 4.2 *Sieves* 9.5-mm (¾-in.) and 600-μm (No. 30) sieves conforming to Specification E 11.
- 4.3 Filter Paper—250 ± 10-mm diameter, rapid filtering, high wet bursting strength, quantitative grade white filter paper.
- 4.4 *Cheesecloth Wrapping*—Two thicknesses, reagent grade cheesecloth, approximately 457 mm (18 in.) square is sufficient for wrapping each sample.
- 4.5 *Steam Bath*—Any suitable apparatus that will meet the requirement of the test procedure. Water in the steam bath, and makeup water, shall be iron-free water or distilled water.

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

5. Reagents

- 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.³
- 5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.
 - 5.3 Concentration of Reagents:
- 5.3.1 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:

 $\begin{tabular}{lll} Hydrochloric acid (HCI) & sp gr 1.19 \\ Ammonium hydroxide (NH_4OH) & sp gr 0.90 \\ \end{tabular}$

5.3.2 *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.

6. Sampling

- 6.1 Sample in accordance with Practice D 75.
- 6.2 After reducing a field sample to an appropriate size in

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."



accordance with Methods C 702, 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.

7. Procedure

- 7.1 Select two portions each weighing 100 g from the aggregate sample prepared for test.
- 7.2 Crimp the edges of two filter papers to form cup-shaped receptacles approximately 130 mm (5 in.) in diameter and 60 mm ($2\frac{1}{2}$ 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.
- 7.3 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, adding distilled water as make-up water as required.
- 7.4 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 $110 \pm 5^{\circ}$ C (230 \pm 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.
- 7.5 Rate the extent of staining on the filter papers by the Visual Classification Method.
- 7.5.1 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).
- 7.6 When required by Specification C 330 and Specification C 331, follow the procedure of the Chemical Analysis Method.
- 7.6.1 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 NH₄OH dropwise to neutralize the acid using methyl red indicator solution. Redissolve the $\text{Fe}(\text{OH})_3$ precipitate using $10~\text{cm}^3$ of HCl (1+1) and determine the iron quantitatively as Fe_2O_3 by standard titration procedures.

 ${\it Note}\ 2$ —The iron may be determined by using other standard quantitative procedures.

8. Calculation

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

$$Fe_2O_3$$
, $mg/200 g = E \times V$ (1)

where:

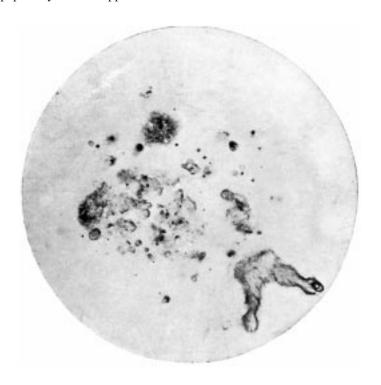
- $E = \text{Fe}_2\text{O}_3$ equivalent of standard titration solution, mg/mL.
- V = millilitres of standard titration solution required by the stains from the 200-g sample.

9. Report

- 9.1 The report shall include the following:
- 9.1.1 Identification of the sample,
- 9.1.2 Stain index, as evaluated by the Visual Classification Method, when required, and
- 9.1.3 Iron content as milligrams of Fe $_2$ O $_3$ per 200 g of sample evaluated by the Chemical Analysis Method.

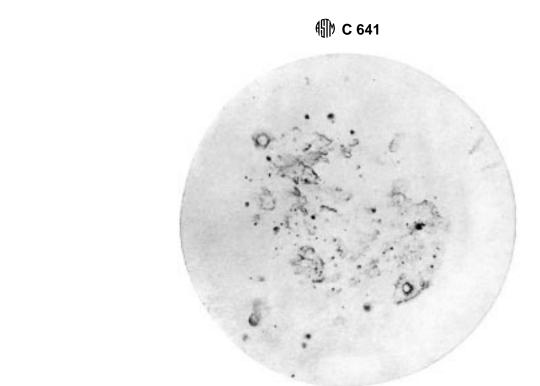
10. Precision and Bias

10.1 Classification Method—Not applicable.

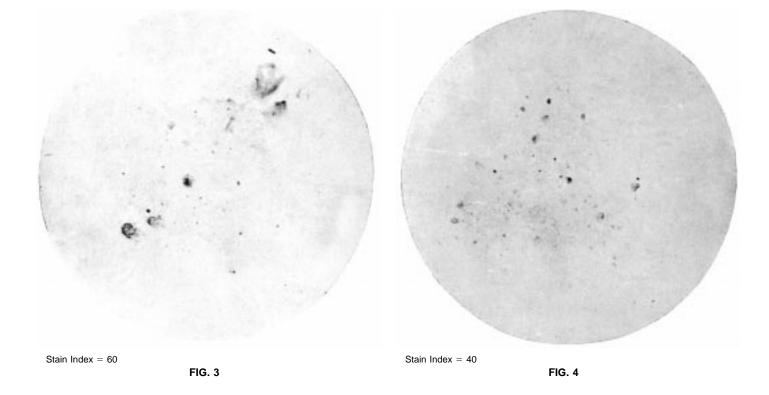


Stain Index = 100

FIG. 1 Visual Degree of Staining

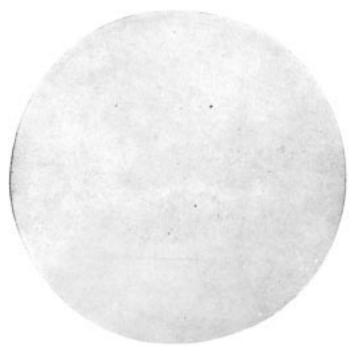


Stain Index = 80 FIG. 2



10.2 Chemical Analysis Method—Refer to the appropriate section of the method selected.





Stain Index = 20

FIG. 5

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