

METHOD OF TEST FOR DETERMINATION OF FREE LIME CONTENT OF STEEL SLAG AGGREGATES

1. SCOPE

1.1 This method covers the procedures for the determination of free lime content of steel slag aggregates.

Note 1: The determination of free calcium oxide (free lime) is based on soluble calcium glycolate formed by ethylene glycol and free lime without influencing the combined lime in silicates, aluminates, etc. The method has been adopted from the cement industry.

2. APPARATUS

2.1 CRUSHER: Small aggregate crusher capable of reducing 19.0 mm particles to pass through a 2.36 mm sieve.

2.2 PULVERIZER: Aggregate pulverizer or shatter box capable of reducing pass 4.75 mm material to a fine powder passing a 75 μ m sieve.

2.3 SPLITTER: Any suitable means of obtaining a representative sample may be used. It is preferable, where practical, to use a mechanical splitter. Riffle type splitters shall have an even number of equal width chutes, but not less than a total of eight for coarse aggregate or twelve for fine aggregate, which discharge alternately to each side of the splitter. The minimum width of the individual chutes shall be approximately 50 percent larger than the largest particles in the sample to be split. The splitter shall be equipped with two receptacles to hold the two halves of the sample following splitting. It shall also be equipped with a hopper or straightedged pan which has a width equal to or slightly less than the over-all width of the assembly of chutes by which the sample may be fed at a controlled rate to the chutes. The splitter and accessory equipment shall be so designed that the sample will flow smoothly without restriction or loss of material.

3. PREPARATION OF TEST SAMPLE - COARSE AGGREGATE

3.1 A representative sample of oven-dried aggregate shall be prepared in accordance with LS-609, Procedure for the Petrographic Analysis of Coarse Aggregate.

3.2 The test specimen is then spread evenly over a flat, non-magnetic surface, and the magnet is moved back and forth over the surface until all magnetic particles are removed.

3.3 The test specimen free of magnetic particles is then crushed using a small crusher so that the entire sample passes a 2.36 mm sieve. Care should be taken that the crusher is clean, and that no material is lost.

3.4 Repeat step 3.2 on the crushed specimen to remove any magnetic particles that may be released in the crushing operation.

3.5 The crushed specimen free of magnetic particles shall be mixed thoroughly, and further reduced using a sample splitter to obtain a specimen of approximately 150 g. This 150 g test specimen shall be pulverized using suitable equipment such as the shatter box, so that it passes a 75 μm sieve. Any material retained on the 75 μm sieve shall be further treated to pass the 75 μm sieve. Care should be taken that no material is lost.

3.6 The pass 75 μm sieve specimen shall be mixed thoroughly and free lime content determined following clause 5.

Note 2: All equipment used in the preparation of these test specimens must be thoroughly cleaned before preparing the next specimen. All possibilities of contamination must be reduced to ensure accurate results.

4. PREPARATION OF TEST SAMPLE - FINE AGGREGATE

4.1 A representative 250-300 g sample of oven-dried aggregate that has passed the 4.75 mm sieve shall be obtained by the use of a splitter, as described in Test Method LS-600, section 3.6.

4.2 The test specimen is then prepared in an identical manner as the coarse aggregate, steps 3.1-3.6, specimen for the determination of free lime content of steel slag, with the exception of step 3.3, the crushing operation.

5. DETERMINATION OF FREE CALCIUM OXIDE

5.1 Weigh out 1 g of finely ground steel slag (minus 75 μm), and transfer to a 200 ml flask, together with a pinch of dry standard sand (C109 Ottawa sand).

5.2 Add 30 ml ethylene glycol and 15 ml absolute methyl alcohol.

5.3 Bring the sample into suspension by shaking, and then place the flask in a water bath and boil for 30 minutes under a reflux condenser, with occasional shaking.

5.4 Filter the contents of the flask through a suction filter, using a SS-White filter paper. Wash the flask twice with 10 ml alcohol, and finally wash the filter once with 10 ml alcohol.

5.5 Add 10 drops bromo-thymol blue indicator and titrate with 0.1 N HCL until yellow-green, record the value of HCL used.

5.6 $\% \text{ free CaO} = \text{ml of HCL} \times 0.28 \%$

Note 3: To get a proper result, the sample has to be ground very finely. The ethylene glycol used must be anhydrous, neutral, and clear to suppress reaction with the combined lime. Furthermore, it must be very pure, and should not be regenerated more than twice. All equipment used for the determination has to be absolutely dry.

6. REPORT

The report shall include the following.

- 6.1 Type and source of steel slag.
- 6.2 Maximum sieve size of the aggregate.
- 6.3 The amount of free lime (CaO) present to the nearest 0.01 %.

7. PRECISION

This test method has not been submitted to a formal multi-laboratory study. Results of a limited between laboratory study are presented in Table 1.

Table 1

SAMPLE NO.	Free CaO % 8 hour extraction MTO Lab (mean of 2)	Free CaO % 0.5 hour extraction MTO Lab	Free CaO % 0.5 hour extraction Dofasco Lab	Difference between Dofasco and MTO Lab	Type of Material and Source
87-9031	3.26	1.76	1.80	+ 0.04	Lasco f.a.
9026	1.76	1.33	1.40	+ 0.07	Lasco f.a.
9051A	4.89	3.56	4.10	+ 0.54	Levy c.a.
9051B	4.88	3.62	4.10	+ 0.48	Levy c.a.
9033	13.90	7.17	6.40	- 0.77	Red-D f.a.
9046	6.02	4.09	5.20	+ 1.11	Red-D c.a.
9038	6.96	4.27	5.10	+ 0.83	Steetley c.a.
9039	9.98	3.40	2.00	- 1.40	Steetley f.a.
9047	13.80	4.98	6.00	+ 1.02	Red-D f.a.
9040	15.00	5.28	5.90	+ 0.62	Steetley f.a.
Average	8.05	4.00	4.20		