Designation: C 1318 – 95 (Reapproved 2001)

Standard Test Method for Determination of Total Neutralizing Capability and Dissolved Calcium and Magnesium Oxide in Lime for Flue Gas Desulfurization (FGD)[1]

This standard is issued under the fixed designation C 1318; 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.

1. Scope

1.1 This test method covers analysis of magnesian, dolomitic and high-calcium limes for total neutralizing capability and dissolved major oxides. Dissolved calcium and magnesium are the major species that neutralize acid under the conditions of the test.

1.2 The test conditions are chosen to measure the acid-neutralizing capacity of both calcium hydroxide and magnesium hydroxide contained in slaked lime. By controlling the neutralization pH at 6, magnesium hydroxide and magnesium oxide are titrated in addition to calcium hydroxide fraction.

1.3 This test method also determines the fraction of Mg ions present in the lime that will dissolve under lime flue gas desulfurization (FGD) conditions. Because the Mg^{++} ion alters FGD performance, it is important to know its concentration.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime[2]

C 50 Practice for Sampling, Sample Preparation, Packing, and Marking of Lime and Limestone Products[2]

C 51 Terminology Relating to Lime and Limestone (as Used by the Industry)[2]

3. Terminology

3.1 *Definitions*—Unless otherwise specified, for definitions of terms used in these test methods refer to Terminology C 51.

4. Summary of Test Method

4.1 Lime is slaked by boiling and is reacted with acid at a pH and residence time similar to those found in full-scale FGD reaction tanks.

4.2 A sample of lime is titrated with 1N hydrochloric acid, maintaining a pH of 6 for 30 min. After 30 min, the acid consumption is recorded. The total neutralizing capacity is calculated from the acid consumption and reported as CaO. Dissolved magnesium is determined by atomic absorption spectrometry or by EDTA titration and reported as percent dissolved Magnesium Oxide (as MgO).

5. Significance and Use

5.1 There are existing lime-based flue gas desulfurization units in operation that require a method to measure the oxides available for sulfur dioxide absorption. Dissolved magnesium oxide varies among limes depending on the limestone sources and calcination conditions.

6. Interferences

6.1 Any substance reacting with acid under the conditions of the test will contribute to the total oxide and dissolved oxide values.

6.2 Magnesium in forms other than MgO, which dissolve under test conditions, may affect the dissolved MgO and total oxide value.

7. Apparatus

7.1 *Digital Readout pH Meter*, with combination electrode readable to 0.01 pH units, or an autotitrator with an automatic temperature compensator capable of titrating to a preset endpoint (Note 1).

NOTE 1—Use of an automatic titrator is recommended.

7.2 *Stirrers and Stir Bars*.

7.3 *Atomic Absorption Spectrometer*, if not using EDTA titration.

7.4 *Filtering Funnel*, Type AE glass fiber paper, volumetric flasks (size as needed), 500 mL volumetric flasks, and various Class A pipettes.

8. Reagents

8.1 *1.0 Normal Hydrochloric Acid*, standardize using procedures in Test Methods C 25, Section 28.

[1] This test method is under the jurisdiction of ASTM Committee C07 on Lime and is the direct responsibility of Subcommittee C07.05 on Chemical Uses.

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[2] *Annual Book of ASTM Standards*, Vol 04.01.

8.2 *Distilled Water*, CO_2 free.

8.3 *Calcium and Magnesium Standard Solutions* (commercially available or use methods in Test Methods C 25, Section 31).

8.4 *EDTA Standard Solution* (0.4 %), standardize according to Test Methods C 25, Section 31.5, Standardization of EDTA.

9. Sample Preparation

9.1 The sampling and grinding of any lime sample shall be carried out as rapidly as possible, so that the absorption of moisture and carbon dioxide is held to a minimum.

9.2 The sample as received at the laboratory shall be thoroughly mixed and a representative sample with minimum weight of 100 g shall be taken and pulverized to pass a No. 50 mesh sieve for analysis.

9.3 The prepared sample shall be stored in a tightly capped container.

10. Procedure

10.1 *Slaking of Lime Sample*:

10.1.1 Weigh rapidly 1.402 g of the finely pulverized sample and brush carefully into a 250 mL Erlenmeyer flask containing about 50 mL of CO_2 free water and immediately stopper the flask.

10.1.2 Remove the stopper. Swirl the flask, place on a hot plate and boil actively 1 min for complete slaking. Remove from the hot plate, stopper the flask loosely and place in a cold-water bath to cool to room temperature.

10.2 *Titration of Sample*:

10.2.1 Set the pH electrode, burette tip, and temperature sensing device in place, while maintaining agitation.

10.2.2 Begin the titration by adding standard 1.0 normal hydrochloric acid solution. Titrate to and maintain a pH of 6 within 0.4 pH units for 30 min (Note 2). Time begins from initial addition of acid.

NOTE 2—When doing a manual titration, the increment of acid addition may require the use of partial drops (suspend a small amount of titrant on the buret tip and wash into the titration flask with CO_2 free distilled water) to avoid exceeding the 0.4 unit limit of pH 6.

10.2.3 Record the amount of acid consumed after 30 min.

10.2.4 Filter the solution and rinse the flask thoroughly with CO_2 free distilled water.

10.2.5 Carefully transfer the solution to an appropriately sized volumetric flask, add approximately 10 mL of 1:1 hydrochloric acid, and dilute to volume.

10.3 *Determination of Dissolved Oxides*:

10.3.1 *Atomic Absorption*:

10.3.1.1 From the diluted solution make the required dilutions and determine the dissolved magnesium by standard atomic absorption spectroscopy techniques.

10.3.1.2 Calculate the dissolved magnesium as MgO according to 11.2.

10.3.2 *EDTA Titration*:

10.3.2.1 From the diluted solution pipette 20 mL of sample into a 500 mL Erlenmeyer flask and dilute with CO_2 free distilled water to an approximate volume of 100 mL.

10.3.2.2 Determine the total dissolved calcium according to Test Methods C 25, Paragraph 31.6.4. Record mL of EDTA required for titration.

10.3.2.3 Repeat 10.3.2 and determine the total dissolved oxides according to Test Methods C 25, Paragraph 31.6.5. Record mL of EDTA required for this titration.

11. Calculation of Results

11.1 *Percent Total Neutralizing Capability (TNC)*:

$$\%\ TNC\ (\text{as CaO}) = \frac{A \times B \times C_1}{W} \tag{1}$$

where:

A = mL HCl used in 10.2.3,
B = normality HCl,
C_1 = chemical factor = 2.804 (mequi of CaO/10), and
W = sample weight, g.

11.2 *Atomic Absorption*:

11.2.1 *Dilution Factor*:

$$\text{Dilution factor} = \text{Initial Volume (see 10.2.5)/Pipette volume for final dilution} \tag{2}$$

11.2.2 *Percent Dissolved Magnesium*:

$$\%\ \text{Dissolved Mg++ (as MgO)} = \frac{A \times B \times C_2 \times D}{W \times 1000} \tag{3}$$

where:

A = concentration of magnesium from AA, ppm Mg^{++},
B = dilution factor,
C_2 = chemical factor = 1.658 (MgO/Mg^{++}),
D = final dilution volume, mL, and
W = sample weight, g.

11.3 *EDTA Titration*:

11.3.1 *Dilution Factor*:

$$\text{Dilution factor} = \text{Initial Volume (see 10.2.5)/titration volume (20 mL)} \tag{4}$$

11.3.2 *Percent Dissolved Calcium*:

$$\%\ \text{Dissolved Ca++ (as CaO)} = \frac{A \times B \times C_3 \times D}{W \times 10} \tag{5}$$

where:

A = mL of EDTA standard solution used,
B = dilution factor,
C_3 = chemical factor = 1.399 (CaO/Ca^{++}),
D = EDTA titer, mg Ca^{++}/mL EDTA, and
W = sample weight, g.

11.3.3 *Percent Total Dissolved Oxides (TDO)*:

$$\%\ TDO\ (\text{as CaO}) = \frac{A \times B \times C_3 \times D}{W \times 10} \tag{6}$$

where:

A = mL of EDTA standard solution used,
B = dilution factor,
C_3 = chemical factor = 1.399 (CaO/Ca^{++}),
D = EDTA titer, mg Ca^{++}/mL EDTA, and
W = sample weight, g.

11.3.4 *Percent Dissolved Magnesium*:

$$\%\ \text{Dissolved Mg++ (as MgO)} = (A - B) \times C_4 \tag{7}$$

where:

A = % total dissolved oxides,
B = % dissolved calcium, and

C_4 = chemical factor = 0.7188 (MgO/CaO).

12. Precision and Bias

12.1 The precision and bias of this test method have not been determined.

12.2 When sufficient data has been obtained and analyzed, a statement of precision will be provided.

12.3 The user is cautioned to verify by the use of reference materials, if available, that the bias of the test method is adequate for the contemplated use.

13. Keywords

13.1 dissolved calcium; dissolved magnesium; flue gas desulfurization (FGD); lime; pH determination; total neutralizing capability