Standard Test Methods for
Quicklime and Hydrated Lime for Neutralization of Waste Acid[1]

This standard is issued under the fixed designation C 400; 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 the testing of quicklime and hydrated lime for use in the treatment of waste acid solutions and includes the characterization of the liming material and of the acid waste.

NOTE 1—When agreed upon by the manufacturer and the purchaser, this method may be used directly to evaluate the lime requirement for a plant waste acid and in the preparation of the lime slurry; the pH, the time, temperature, and other conditions of treatment may be adjusted to conform to plant practice. Otherwise the test shall be performed as described in this method.

NOTE 2—Under some conditions of test the lime requirement may vary substantially from that indicated by ultimate chemical analysis or by the use of a determination of available alkalinity by titration to a stoichiometric end point.

1.2 *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 50 Methods of Sampling, Inspection, Packing, and Marking of Lime and Limestone Products[2]
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *neutralization coefficient*—the number of parts of lime required to neutralize one million parts by weight of sulfuric acid solution (1.5 %) to a pH of 4.4 in 30 min (see Note 1).

3.1.2 *pH*—a measure of the hydrogen ion concentration of a sample and representing the logarithm of the reciprocal (negative logarithm) of the activity of hydrogen ions, calculated as follows:

$$pH = \log \frac{1}{(H)^+} = -\log(H^+)$$

where:
H^+ = activity of hydrogen ions.

4. Significance and Use

4.1 Since all limes and waste acid solutions are different, this test method evaluates the relative quantity of a given lime needed for the neutralization of a specific quantity of acid.

4.2 Liming materials have a specific basicity factor or measure of alkalinity which can be used for comparing their relative neutralizing power. It avoids dependence on chemical analysis and is determined as grams of calcium oxide equivalents per kilogram of liming material.

4.3 Likewise, specific waste acids have an acid value that can be expressed as grams of hydrochloric acid equivalent per litre of acid waste. This value is related on a stoichiometric basis to lime neutralization requirements of a liquid acid waste.

5. Apparatus

5.1 *pH Measuring Apparatus,* conforming to the requirements of Section 5, Apparatus, and Section 6, Reagents and Materials, of Test Method E 70. A time-pH recording device or attachment is desirable but not required.

5.2 *Analytical Balance,* capable of reproducing results within 0.005 g.

5.3 *Stirrer,* motorized, capable of rotating at speeds from 120 to 600 r/min.

5.4 *Miscellaneous Equipment*—Chemically resistant glassware,[4] metal stands for support of the equipment, etc.

6. Purity of Reagents

6.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall

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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.

6.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

7. Test Method A—for Determining Lime Requirement for the neutralization of a standard sulfuric acid or waste acid solution within a time limit of thirty minutes.

7.1 *Reagents*:

7.1.1 *Sulfuric Acid, Standard Solution* (1.5 ± 0.05 %)— Thoroughly mix 15.8 g of concentrated sulfuric acid (95 % H_2SO_4) with water, dilute to 1 L, analyze to within 0.005 % H_2SO_4, and store in a suitable stoppered bottle.

7.2 *Preparation of Sample*:

7.2.1 Take samples of quicklime and hydrated lime in accordance with Methods C 50.

7.2.2 *Quicklime*—Weigh a sample of quicklime (2.50 ± 0.01 g) ground to pass a No. 50 mesh sieve. Add to a 500-mL tall-form beaker containing 50 mL of boiling water and continue to boil for 2 min. Remove from hot plate and continue stirring with a magnetic stirrer until the slurry reaches room temperature (at least 10 min).

7.2.3 *Hydrated Lime*—Mix a sample of hydrated lime (2.50 ± 0.01 g) with 50 mL of water in a 500-mL tall-form beaker for at least 10 min.

7.3 *Procedure*:

7.3.1 Place the pH electrodes within the beaker about ½ in. (13 mm) above the surface of the lime slurry sample. Set the motor stirrer to the highest speed at which splattering or immersion of the electrodes does not occur. Measure 250 g of H_2SO_4 (1.5 ± 0.05 %) into a separate 500-mL beaker. Add this acid to the sample in a single motion and record the pH as a function of time for a period of 30 min (see Note 1).

7.3.2 Test a second slurry sample using more or less than 250 g of H_2SO_4 (1.5 ± 0.05 %), as indicated by the pH value observed after 30 min reaction in the first test. Repeat the procedure at least four times or until sufficient data are available to give a family of curves that indicate pH at 30 min as a function of grams of lime per 1000 g of H_2SO_4 (1.5 %), with particular emphasis in the region of pH equals 4.4.

7.4 *Results*:

7.4.1 *Neutralization Rate Curves*:

7.4.1.1 Plot pH versus time in minutes for each of the lime-acid ratios used in the initial series of tests. This family of curves serves as the basis for calculation of the lime requirements.

7.4.2 *Lime Requirement Curve*:

7.4.2.1 Plot the pH in 30 min, as determined from the neutralization rate curves described in Section 10, against grams of lime used per 1000 g of H_2SO_4 (1.5 %).

[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."

7.4.3 *Neutralization Coefficient*:

7.4.3.1 Interpolate the quantity of lime required to achieve a pH of 4.4 in 30 min from the lime requirement curve (Section 11), correct for the actual acid concentration, and calculate as parts of lime required per million parts of H_2SO_4 (1.5 %) as follows:

$$N = (1.5/Z) \times X \times 1000$$

where:
N = neutralization coefficient (see 3.1.1),
X = grams of lime per 1000 g of H_2SO_4(1.5 %), and
Z = concentration of the H_2SO_4 to the nearest 0.005 %, as determined by analysis.

7.5 *Precision and Bias*:

7.5.1 The precision and bias of this test method has not been determined at this time. Replicate samples are recommended.

8. Test Method B—For Determining Acid Value-Basicity Factor Relationship

8.1 *Procedure*:

8.1.1 *Basicity Factor*—Weigh 2.804 g ± 1 mg of the sample that has been prepared to pass a No. 100 (150-µm) sieve and transfer to a 500-mL Erlenmeyer flask containing approximately 20 mL of cold water. (For quicklime samples, heat to boiling to ensure complete slaking). Add from a buret 100 mL of 1.0 *N* hydrochloric acid and stopper with a two-hole rubber stopper (Note 3). The quantity of acid may be increased or decreased depending on the equivalent strength of the material being tested. Place the flask on a hot plate and boil for 15 min. (Glass beads may be added to prevent bumping.) Remove the flask from the hot plate and cool in water. Add several drops of phenolphthalein indicator solution and titrate the excess acid with 0.5 *N* sodium hydroxide solution. Calculate the basicity factor as follows:

$$\text{Basicity factor} = [(V_1 N_1 - V_2 N_2)/W] \times 0.02804$$

where:
V_1 = HCl (mL) required for titration of the sample,
N_1 = normality of HCl,
V_2 = NaOH solution (mL) required for titration of the excess acid,
N_2 = normality of NaOH solution, and
W = sample (g).

Note 3—A water-cooled condenser with a tapered, ground-glass joint and a magnetic stir plate may be used during the boiling step.

8.1.2 *Acid Value*—Pipet 10 mL of the sample of acidic waste into a 250-mL Erlenmeyer flask. This quantity may be increased or decreased depending on the strength of the acidic waste. Add an excess of 25 to 30 mL of 0.5 *N* sodium hydroxide solution and boil for 3 min. Filter through a small Büchner funnel with the aid of vacuum. Wash the residue on the funnel with water. Add several drops of phenolphthalein indicator solution to the filtrate and titrate the excess sodium hydroxide with 1.0 *N* hydrochloric acid solution. Calculate acid value as follows:

$$\text{Acid value} = [(V_1 N_1 - V_2 N_2)/V] \times 0.03646$$

where:

V_1 = NaOH solution (mL) required for titration of the sample,
N_1 = normality of NaOH solution,
V_2 = HCl (mL) required for titration of the excess NaOH solution,
N_2 = normality of HCl, and
V = sample (mL).

8.1.3 *Relationship of Acid Value to Basicity Factor*—Calculate the acid value-basicity factor relationship as follows:

$$\frac{\text{Acid value}}{\text{Basicity factor}} \times 0.7691$$

= lime or limestone product (kg) per litre of Acidic Waste.

NOTE 4—When agreed upon between the manufacturer and the purchaser, Test Method A may also be used.

8.2 *Precision and Bias*:

8.2.1 The precision and bias of this test method has not been determined at this time. Replicate samples are recommended.

9. Keywords

9.1 acid value; alkalinity; basicity; lime requirement; liming material; neutralization; neutralization coefficient; pH; waste acid