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T_____ 617

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WORKING GROUP
CHAIR_____ Todd Bolton

SUBJECT
CATEGORY_____ Chemical Properties

RELATED
METHODS_____ See "Additional Information"

CAUTION:

This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this test method, the user should determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Analysis of lime

(Proposed Reconfirmation of Classical Method T 617 cm-10)

(Underscores, notes, and strikethroughs show changes from Draft 1)

1. Scope

1.1 "Available lime index," expressed as a percentage of calcium oxide, is a measure of the alkaline content of high calcium quicklime and hydrated lime that reacts with hydrochloric acid under the specific conditions described in this method. In this method, procedures are given for sampling a shipment of lime and determining its available lime index. Procedures for determining the components of limestone are given in TAPPI T 618 "Analysis of Limestone."

1.2 Two forms of lime are used in the pulp and paper industry: quicklime or unslaked lime and hydrated or slaked lime. Quicklime is produced by incinerating limestone or chemically similar materials, such as oyster shells, leaving a residue consisting chiefly of CaO. It should contain a minimum of fine air-slaked dust which is practically worthless because the dust has taken up CO₂ from the air and formed the carbonate, CaCO₃. Quicklime, when slaked,

should disintegrate almost completely, leaving a minimum quantity of unslaked coarse particles. These may be overburnt oxides, uncalcined cores, or small stones.

1.3 Dry hydrated lime is made from quicklime by slaking with just enough water to cause the lumps of quicklime to form a powder, the heat of reaction being sufficient to dry the product. On a CaO basis, a ton of hydrated lime is equivalent to about three-quarters of a ton of quicklime.

1.4 Dolomitic limestone has a high magnesium content, so that its residue will consist of CaO and MgO and will have a relatively low available lime index.

2. Apparatus

- 2.1 *Shovel*, for sampling bulk materials and quartering.
- 2.2 *Thief*, a thin-walled metal tube about 25 mm (1 in.) ID and 91.4 cm (3 ft) long, for sampling bags.
- 2.3 *Sheet*, canvas or heavy plastic, large enough for mixing and quartering the sample.
- 2.4 *Mechanical grinder*, or agate mortar and pestle, for reducing material to pass a 100-mesh sieve.
- 2.5 *Sample bottles*, 177-mL (6-oz) wide-mouth glass bottles, with rubber stoppers to keep the samples in air-tight storage.
- 2.6 *Glassware*: Erlenmeyer flask, 300 mL, with rubber stopper; graduated cylinders, 25 and 250 mL; buret, 100 mL.

3. Reagents

- 3.1 *Hydrochloric acid*, 0.2000N or 0.1783N HCl. Each milliliter of the latter is equivalent to 1% CaO when a 0.5-g specimen is used, and it is convenient if many titrations are to be made. (See TAPPI T 610 "Preparation of Indicators and Standard Solutions" for guidance, but adjust procedure of T 610 to obtain 0.2000 or 0.1783 normality.)
- 3.2 *Indicator*, phenolphthalein (see TAPPI T 609 "Indicators for Volumetric Analysis").
- 3.3 *Granulated sugar* (sucrose).
- 3.4 *Distilled water*, CO₂-free. Prepare by boiling a supply and cooling it in a stoppered container.

4. Sampling

4.1 Shipments should be sampled within 24 h after receipt, and, in order to avoid undue exposure to air, the samples should be taken and enclosed as expeditiously as possible.

4.2 For shipments in bags, sample at least 1% of the bags or five bags, whichever is larger, from various locations in the shipment. In sampling a bag, either take about 0.5 kg (1 lb) from beneath the top layer, or, with the thief, take a core from the interior of the package. If possible, sample bulk material as it is being delivered, taking an aggregate sample of 0.9 or 1.4 kg (2 or 3 lb) per ton. For small bulk shipments, take portions totaling at least 22.7 kg

(50 lb). In any case, the total sample from a bulk car or other unit should be a composite of at least 10 roughly equal amounts taken from 10 locations not having been exposed to the air.

4.3 Break up any lumps larger than 25 mm (1 in.). Place the samples on a large sheet, on a flat surface, then mix and quarter them in accordance with TAPPI T 605 "Reducing a Gross Sample of Granular or Aggregate Material to Testing Size." In this way, secure a representative composite sample of approximately 2.3 kg (5 lb) and place it in an airtight container.

4.4 For analysis, mix the 2.3-kg (5-lb) sample thoroughly, breaking up any lumps and further reduce by quartering to give a minimum portion of 75 g. Grind this portion by mechanical means or by hand in an agate mortar, until it all passes a 100-mesh sieve. Then thoroughly mix it and place it in an airtight container.

5. Procedure

5.1 Weigh a 0.5-g specimen of the prepared sample to the nearest 0.001 g and carefully brush it into a 300-mL Erlenmeyer flask containing about 20 mL of CO₂-free distilled water. Immediately loosely stopper the flask with a rubber stopper.

NOTE 1: It is important to have a little water in the flask before adding the specimen. This is especially true with quicklime, since by adding water on the top of a dry charge in a flask, the lime may cake and form lumps difficult to dissolve completely. On the other hand, if the lime is added to a little water, conditions are favorable for a thorough dispersion of fine particles. With quicklime, the slaking action facilitate its dispersion and solution.

5.2 Disperse the specimen thoroughly in the water by a swirling motion, heat to boiling, and boil the loosely-stoppered flask for 2 min. Remove the stopper, add 150 mL of CO₂-free distilled water at room temperature, and then add 15 g of granulated sugar.

NOTE 2: The amounts of sugar and water used are only approximate. It is not necessary to weigh the sugar each time. To save time when testing a large number of samples, a small measure can be employed which when level full holds 15 g. A little excess sugar always ensures at least a 10% sugar solution.

5.3 Stopper the flask, shake vigorously at intervals for a 5-min period, and allow to stand for 45 ± 15 min. Add 2 drops of phenolphthalein indicator, wash down the stopper and sides of the flask with CO₂-free distilled water, and titrate (in the original flask) with the 0.2000*N* or 0.1783*N* HCl as follows:

5.4 First add about 90% of the acid requirement from a 100-mL buret, without shaking. Then shake the flask vigorously and rapidly. Complete the titration to the first complete disappearance of the pink color. Note the volume of acid used and ignore the return of the color.

NOTE 3: Unless the operator is familiar with a previous analysis of the lime under test, it is good practice to carry out a preliminary titration.

6. Calculation

Calculate the equivalent percentage of CaO as: (a) milliliters 0.1783N HCl \times 1.0000, or (b) milliliters 0.2000N HCl \times 1.122.

7. Report

Report the equivalent percentage of CaO as the available lime index, to three significant figures.

Table 1. Results of precision study

Sample	Material	<u>Available lime index</u>		Difference between duplicates
		No. 1	No. 2	
A	Slaked lime	70.29	70.53	0.24
B	Slaked lime	70.69	70.87	0.18
C	Slaked lime	72.37	72.38	0.01
D	Quicklime	95.00	95.20	0.20

Table 2. ASTM Specifications

No.	ASTM designation, use and material	CaO + MgO, min. %	Avail- able CaO, %	SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃ , max. %	Fe ₂ O ₃ max. %
1	ASTM C45-25 For cooking rags				
	Quicklime ^a	...	90.0
	Hydrated lime ^a	...	64.3

2	ASTM C46-62				
	For sulfite pulping				
	Quicklime ^b	95.0	...	3.0	...
3	ASTM C433-63				
	For hypochlorite bleach				
	Quicklime	...	90.0	...	0.3
	Hydrated lime	...	68.0	...	0.3
4	ASTM C53-52T				
	In water treatment for				
	(A) softening				
	Quicklime ^c	...	90.0
	Hydrated lime	...	68.1
	(B) Boiler water SiO ₂				
	Removal: Color removal and clarification ^d				
	Quicklime ^c	93.0
	Hydrated lime	... ^e

^aASTM refers to this as a standard composition rather than a minimum specification. ASTM also states that the lime shall be clean and free from gritty substances. ^bPercentages on a nonvolatile basis. ^cASTM states that "Quicklime shall be reasonably free of unslakable residues and shall be capable of disintegrating in water to form a suspension of finely divided material." ^dASTM recommends dolomitic lime for silica removal, high calcium lime for most other water treatment purposes. ^eEquivalent to 93% in quicklime.

8. Precision

Duplicate determinations by one chemist on each of four samples of different limes showed the results given in Table 1.

9. Keywords

Lime, Calcium oxide

10. Additional information

10.1 Effective date of issue: **To be assigned.**

10.2 Available CaO is of principal importance for cooking rags, straw, etc., for causticizing in kraft recovery, and for the treatment of water. For the sulfite process and in kraft recovery, the presence of certain impurities is undesirable. Specifications adopted by the American Society of Testing and Materials are given in Table 2.

10.3 This method, formerly T 617 os-68, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable.

10.4 Related methods: ASTM C25 58; Canadian PAPTAC J.5.

10.5 Note 1 in Section 5.1, changed facilities to facilitate.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■