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BUREAU OF STANDARDS.

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RECOMMENDED SPECIFICATION FOR QUICKLIME AND HYDRATED LIME FOR THE MANUFACTURE OF SILICA BRICK.

ABSTRACT.

A brief description of the way in which lime is used in the manufacture of silica brick is followed by a general statement as to the quality of lime required.

The standard of quality is set at 92 per cent, based on the nonvolatile matter, with maximum limits of 5 and 10 per cent carbon dioxide, depending upon whether the sample is taken at point of shipment or of destination.

Complete directions for sampling and testing are included.

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This is the seventh of a series of specifications for the lime used in various chemical industries. To assist in the development of these specifications the bureau has called together an Interdepartmental Conference on Chemical Lime, composed of representatives of the Geological Survey and Bureau of Mines of the

Interior Department; the Bureau of Soils, Bureau of Chemistry, Forest Service, and Fixed Nitrogen Research Laboratory of the Department of Agriculture; and the Chemical Warfare Service of the War Department. The present specification, based on a draft originally prepared by M. E. Holmes, chemical director, National Lime Association, has been unanimously approved by the above conference, the National Lime Association, and a representative group of consumers. It contains the same requirements as a similar specification published as tentative by the American Society for Testing Materials.

I. GENERAL.

1. DEFINITION OF QUICKLIME.

Quicklime is the product resulting from the calcination of limestone and consists essentially of calcium oxide, or of calcium and magnesium oxides. It will slake when water is added to it, and this slaking is accompanied by an evolution of heat and an increase in volume.

2. DEFINITION OF HYDRATED LIME.

Hydrated lime is a dry powder which is made by treating quicklime with enough water to satisfy its chemical affinity under the conditions of manufacture. It consists essentially of calcium hydroxide, or of a mixture of calcium hydroxide and magnesium oxide or hydroxide.

3. USE OF LIME IN MANUFACTURE OF SILICA BRICK.

In the manufacture of silica brick, silica, preferably quartzite, is ground in a wet pan until the particles are of suitable size. Milk of lime is then added in quantities varying from 1.5 to 4 per cent CaO (based on the weight of the silica), and the shapes are molded and dried. When bone dry they are burned until most of the quartzite has been converted into tridymite and cristobalite. Either quicklime or hydrated lime may be used.

4. QUALITY.

The lime shall be substantially free from ash, dirt, and core. Fineness of hydrate and rapidity of slaking of quicklime are desirable qualities.

5. PACKING.

Quicklime is shipped either in bulk in carload lots or barrels holding 180 pounds or 280 pounds. Hydrated lime is shipped in paper bags holding 50 pounds.

II. REQUIREMENTS.

1. COMPOSITION.

The quick lime or hydrated lime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

	Per cent.
Calcium oxide (CaO), minimum.....	92.0
Magnesium oxide (MgO), maximum.....	3.0
Combined iron and aluminum oxides (Fe ₂ O ₃ and Al ₂ O ₃), maximum.....	1.5
Silica (SiO ₂) and insoluble matter, maximum.....	3.0
Carbon dioxide (CO ₂), maximum:	
(a) If sample is taken at place of manufacture.....	5.0
(b) If sample is taken at other than place of manufacture.....	10.0

2. MARKING.

Each carload of material, or fraction thereof, shall be legibly marked with the names of the consignor and consignee, and with some means of identifying the particular contract on which the shipment is made.

This marking is in addition to that required by the Federal lime-barrel law.

3. RETESTING.

Notice of the rejection of a shipment based on these specifications must be in the hands of the consignor within 10 days after the receipt of the shipment at the point of destination. If the consignor desires a retest, he shall notify the consignee within 5 days of receipt of the notice of rejection. The consignee shall provide all reasonable facilities to permit the consignor to re-sample the material. This retest shall be at the expense of the consignor.

III. SAMPLING AND TESTING.

1. SAMPLING.

The purchaser will bear all expense of sampling and testing. When quicklime is shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom, and shall not contain a disproportionate share of the top and bottom layers, which are most subject to changes. The sample shall consist of 1 shovelful for each 3 tons of material, but not less than 10 shovelfuls, taken from different parts of the shipment. The total sample taken shall weigh at least 100 pounds, shall be mixed thoroughly, and "quartered" to provide a 15-pound sample for the laboratory. In case a shipment consists of more than 1 car, a separate sample shall be taken from each car.

When quicklime is shipped in barrels, at least 3 per cent of the number of barrels shall be sampled. They shall be taken from various parts of the shipment, dumped, mixed, and sampled as specified in the above paragraph.

In the case of hydrated lime, 3 per cent of the packages shall be sampled. The sample shall be taken from the surface to the center of the package. The material so obtained shall be thoroughly mixed and quartered to provide a 2-pound sample for the laboratory.

When sampling quicklime or hydrated lime, it is essential that the operation be conducted as expeditiously as possible, in order to avoid undue exposure of the material to the air. The sample to be sent to the laboratory shall immediately be placed in an air-tight container, in which the unused portion shall be stored until the shipment has been finally accepted or rejected by the purchaser.

The sample may be taken either at the point of shipment or at destination, as agreed upon by the contracting parties.

2. TESTING.

The following directions for analytical work are adapted from American Society for Testing Materials, Tentative Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime, C25-22T. For further information on this subject, reference should be made to "The analysis of silicate and carbonate rocks," by W. F. Hillebrand, U. S. Geological Survey Bulletin No. 700.

(a) SILICA AND INSOLUBLE MATTER.—Weigh out 0.5 g of the substance. Transfer to an evaporating dish, preferably of platinum (porcelain may be used, but not glass) for the sake of celerity in evaporation, mix to a thin slurry with distilled water, add 5 to 10 cc of HCl (sp. gr. 1.20) and digest with aid of gentle heat and agitation until solution is complete. Solution may be aided and the time shortened by light pressure with the flattened end of a glass rod upon resistant lumps. Then evaporate the solution to dryness so far as this may be possible on the water bath. When dry or nearly so, place the dish and its contents in an air bath or (covered) on a platinum triangle resting on a hot plate. Drench the cooled mass with HCl (sp. gr. 1.20) and allow to stand for a few minutes. Add an equal volume of water, cover the dish, and place on the water bath for 10 minutes. Separate the silica by filtration on a filter of suitable size. Wash thoroughly with dilute HCl and then wash twice with cold water.

Evaporate the filtrate to dryness. Extract the residue with HCl as before, but allowing only a few minutes' time. Filter this solution through a second and smaller paper. Transfer, wet, the papers containing the separated residues to a platinum crucible. Char carefully without allowing the paper to inflame and ignite in an electric muffle (a blast lamp may be used) and weigh. Correct the silica for foreign matter by evaporating with 5 cc of HF and one or two drops of H_2SO_4 . Heat the residue for two or three minutes in an electric muffle (a blast lamp may be used).

(b) COMBINED IRON AND ALUMINUM OXIDES.—Fuse the residue from the silica determination using a little Na_2CO_3 . Dissolve the cooled melt in HCl and add the solution to the filtrate from the second evaporation for silica. Add a few drops of bromine water or HNO_3 and boil the solution until all trace of bromine or chlorine is gone. Then add HCl, if necessary, sufficient to insure a total volume of 10 to 15 cc of HCl (sp. gr. 1.20). Add a few drops of methyl red solution and after diluting to 200 to 250 cc heat the liquid to boiling. Neutralize by means of NH_4OH (dilute toward the end) until the color of the liquid changes to a distinct yellow. Boil for one or two minutes, allow to settle, filter and wash the precipitate at once two or three times with hot NH_4Cl (2 per cent) solution and suck dry. Set the filtrate aside. Dissolve any precipitate from the paper in hot, dilute HCl, the solution passing into the beaker in which the precipitation was made, and wash the paper thoroughly with hot water. Boil the solution to expel any trace of chlorine and again precipitate the hydroxides with NH_4OH as described above. Ignite and weigh as R_2O_3 .

(c) CALCIUM OXIDE.—Add a few drops of NH_4OH to the combined filtrate from the R_2O_3 precipitate, and bring the solution to boiling. To the boiling solution add 35 cc of a saturated solution of $(NH_4)_2C_2O_4$, and continue the boiling until the precipitated CaC_2O_4 assumes a granular form; then allow it to stand one hour, or until the precipitate has settled and the supernatant liquid is clear, filter and wash two or three times with boiling water. Place the wet filter and precipitate in a platinum crucible, and burn off the paper over the small flame of a Bunsen burner, or in a muffle. Ignite the residue, dissolve in hot dilute HCl, and make up the volume of the solution to 100 cc with water. Add NH_4OH in slight excess and boil the liquid. If a small amount of $Al_2(OH)_6$ separates, filter it out, wash with NH_4Cl (2 per cent solution), ignite, weigh, and add this weight to that found in the first determination. Then reprecipitate the lime by $(NH_4)_2C_2O_4$, allow to

stand until settled, filter, wash two or three times with boiling water and weigh as calcium oxide after ignition to constant weight in a covered platinum crucible.

(d) **MAGNESIUM OXIDE.**—Acidify the combined filtrates from the calcium precipitates with HCl and concentrate on the water bath to about 150 cc and heat to boiling. To the boiling solution add 10 cc of a saturated solution of $\text{NaNH}_4\text{HPO}_4$ and continue the boiling for several minutes. Then cool to room temperature, and add NH_4OH drop by drop with constant stirring until the crystallin ammonium-magnesium orthophosphate begins to form and then in moderate excess, the stirring being continued for several minutes. Allow the liquid to stand in a cool atmosphere for from 12 to 48 hours and filter. Dissolve the precipitate in hot dilute HCl, make up the solution with water to about 100 cc, add 1 cc of the saturated solution of $\text{NaNH}_4\text{HPO}_4$ and NH_4OH drop by drop with constant stirring until the precipitate is again formed as above described and the ammonia is present in moderate excess. Then allow it to stand in a cool atmosphere for from 12 to 48 hours, filter on a paper or a Gooch crucible, wash with dilute NH_4OH containing NH_4NO_3 , ignite, cool, and weigh as $\text{Mg}_2\text{P}_2\text{O}_7$. Multiply this weight by 0.3621 to find the weight of the MgO .

(e) **LOSS ON IGNITION.**—Place 1 g of the properly prepared sample in a weighed platinum crucible, cover with the lid, and heat gently for 5 minutes, gradually increasing the temperature to decompose carbonates, and maintain at this temperature until constant weight is obtained (usually about 15 minutes).

(f) **CARBON DIOXIDE.**—Carbon dioxide is to be determined upon the properly prepared sample according to the method given in U. S. Geological Survey Bulletin No. 700, page 217. An illustration showing the arrangement of the component parts of the necessary apparatus is shown on page 218 of that bulletin. Boil the 5 g sample with dilute HCl in a small Erlenmeyer flask attached to an upwardly inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulphate to retain hydrogen sulphide from decomposable sulphides and any HCl that may pass over, then calcium chloride again—carbon dioxide is caught by absorption tubes filled with soda lime followed by calcium chloride. The soda lime used for this determination must be porous, not hard, and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances. Of course, arrangement is made for a current of air free from CO_2 , with which to sweep out the apparatus before and

after the experiment, and for a slow current during its continuance. The manipulations are as follows: Pour hot water upon the powder in the flask fitted with a separatory funnel and delivery tube; attach this to the condenser, and force a current of air free from CO_2 through the whole system except the weighed, absorption tubes, until the original air has been displaced, the observation bulbs (containing H_2SO_4 to show the rate of gas flow) being attached directly to the drying system. Then close the stopcock in the separatory funnel, half fill the latter with HCl (1 : 1), replace the rubber stopper of the funnel, insert the absorption tubes between the drying system and the observation bulbs, and allow the acid to flow into the flask, slowly if there is much carbon dioxide, rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, light the burner under the flask and start the flow of water through the condenser. Keep the flame low, so as to secure steady but quiet ebullition, and do not interrupt the air current although it should be reduced to a slow rate. With much carbon dioxide the rate of absorption is very readily noted by holding the hand to the soda-lime tubes, which become hot or warm when absorption is taking place. Sufficient time having elapsed, extinguish the flame, and increase the air current. When cool, disconnect the soda-lime tubes from the apparatus and allow to stand in the balance case until two weights taken 30 minutes apart agree within 0.5 mg.

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