DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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RECOMMENDED SPECIFICATION FOR QUICKLIME AND HYDRATED LIME FOR USE IN THE ABSORPTION OF CARBON DIOXIDE

[This is the eighth 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 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 W. E. Emley, of the lime section, Bureau of Standards, at the request of the Bureau of Aeronautics, of the Navy Department, has been unanimously approved by the above conference, by the National Lime Association, and by the Bureau of Aeronautics, of the Navy Department.]

ABSTRACT

Carbon dioxide may be removed from mixtures of gases by absorption, using lime. Two methods are employed: Hydrated lime may be moistened and spread out in thin layers, over which the gas to be purified passes, or the gas may be scrubbed with a milk of lime made from either quicklime or hydrated lime. Quicklime should contain not less than 90 per cent available lime and hydrate not less than 85 per cent available lime. Hydrate should have a fineness of 97 per cent through a No. 200 screen.

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1. GENERAL

(*a*) 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.

(*b*) 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 mixture of calcium hydroxide and magnesium oxide or hydroxide.

(*c*) Use of Lime in the Absorption of Carbon Dioxide.—Owing to the slowness with which it reacts with dry carbon dioxide, quicklime is not used as such for the absorption of this gas. It is frequently purchased, however, to be made into the hydrated lime or milk of lime used for this purpose.

Hydrated lime may be moistened and spread out in thin layers, over which the gas to be purified passes. Milk of lime, made from either quicklime or hydrated lime, may be used as the liquid with which the gas is scrubbed.

Whichever process is used, the net result is that the carbon dioxide in the gas reacts chemically with the calcium hydroxide to form calcium carbonate. Because of the limited solubility of magnesium hydroxide, the effectiveness of this ingredient is doubtful.

(*d*) Quality.—The constituents other than calcium oxide ordinarily found in lime, as well as underburned and overburned lime, must be regarded as diluents, increasing the handling charges per unit of effective material. The fineness of the hydrate is important, in the dry process because the finer the hydrate the greater the surface presented to the gas and in the milk of lime process because the finer the hydrate the less the possibility of trouble from clogged pipes, pumps, or sprays.

(*e*) Packing.—Quicklime is shipped either in bulk in carload lots or in barrels holding 180 or 280 pounds net. Hydrated lime is shipped in paper bags holding 50 pounds net each. In Texas the law requires hydrated lime to be shipped in paper bags holding 40 pounds net each.

2. REQUIREMENTS

(*a*) Composition.—Quicklime shall contain not less than 90.0 per cent of available lime and hydrated lime not less than 85.0 per cent of available lime, computed on the basis of the nonvolatile matter contained therein. Quicklime or hydrated lime shall contain not more than 5 per cent of carbon dioxide, computed on the basis of the sample as received.

(*b*) Fineness.—Hydrated lime shall be of such fineness that 99.5 per cent of it will pass a No. 50 sieve and 97.0 per cent will pass a No. 200 sieve.

(*c*) 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 information is in addition to that required by the Federal lime-barrel law.

(*d*) 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 five days of receipt of the notice of rejection. The consignee shall provide all reasonable facilities to permit the consignor to resample the material. This retest shall be at the expense of the consignor.

3. SAMPLING AND TESTING

(*a*) 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 one 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, the sample shall be a fair average of the shipment. Three 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 the point of destination, as agreed upon by the contracting parties.

(*b*) TESTING.—Details regarding the complete analysis of lime are given in other papers of this series and in Bulletin 700 of the U. S. Geological Survey.

Available lime.—1. To 1.4 g of the carefully prepared and finely ground (passing a No. 100 sieve) lime, in a 400 cc beaker add 200 cc of hot water, cover, heat carefully, and then boil for three minutes.

2. Cool, wash down cover, add two drops of phenolphthalein and titrate with normal hydrochloric acid, adding the acid dropwise as rapidly as possible, stirring vigorously to avoid local excess of acid. When white spots appear, retard the rate of addition of acid somewhat, but continue until the pink color fades out throughout the solution for a second or two. Note the reading and ignore the return of color.

3. Repeat the procedure of paragraph 1 above, using (instead of a beaker) a liter graduated flask carrying a one-hole stopper fitted with a short glass tube drawn out to a point. Cool, add slowly 5 cc less acid than before, stirring vigorously. Call the number of cc used "A." Grind up any small lumps with a glass rod slightly flattened at one end, dilute to the mark with freshly boiled distilled water, stopper, mix thoroughly for four or five minutes, and let settle for half an hour.

4. Pipette off a 200 cc portion, add phenolphthalein, and titrate slowly with half-normal hydrochloric acid until colorless on standing one minute. Call this additional number of cc "B." Then percentage of available lime=2A+5B.

Volatile matter.—Heat 1 g of the sample in a platinum crucible to constant weight, using a blast lamp or an electric muffle, and a temperature sufficiently high to insure decomposition of the carbonates. The loss of weight is calculated as a percentage of the original weight. This figure is used to correct the percentage of available lime found to the nonvolatile basis.

Carbon dioxide.—Place 5 g of the sample of quicklime or hydrated lime in a small Erlenmeyer flask and cover with hot distilled water. Connect this flask into a carbon-dioxide train, set up as follows: Next to the flask is a reflux condenser, to which is connected a calcium-chloride drying tube, followed by a tube containing anhydrous $CuSO_4$, then another tube of $CaCl_2$, then by two tubes filled with soda lime, and finally by another tube of $CaCl_2$. The entire train must be so arranged that a stream of air free from CO_2 can be kept passing through it. Start this stream of air. Weigh the tubes containing soda lime and replace them in the train. Add to the sample in the flask about 25 cc of 1:1 HCl, being careful that

no gas is lost and that the effervescence is not too violent during the operation. When the effervescence diminishes, heat the flask, bringing the liquid gradually to boiling. Boil for 10 minutes. Remove the flame and allow the flask to cool while the stream of air is still flowing for 15 minutes. Disconnect and weigh the soda-lime tubes. Their increase in weight is recorded as carbon dioxide.

Fineness.—Fineness of hydrated lime shall be determined as follows: One hundred grams of the sample as received shall be placed on a No. 50 sieve, which shall be nested above a No. 200 sieve. The material shall be washed by means of a stream of water from a faucet.[1] The washing shall be continued until the water coming through the sieve is clear. The residue upon the No. 50 sieve shall be dried to constant weight in an atmosphere free from carbon dioxide in a drying oven whose temperature is maintained between 100 and 120° C. The weight of this residue shall be calculated as percentage of the original sample.

The material which has passed the No. 50 sieve and remained on the No. 200 sieve shall be then washed through the latter as above described. The residue on this sieve shall be treated in the same manner as described above for that retained on the No. 50 sieve and the percentage retained added to the percentage of residue on the No. 50 sieve. The sum of these two shall be reported as the percentage of residue on the No. 200 sieve.

[1] A small piece of rubber tubing attached to a water faucet will be found convenient. The velocity of the stream of water may be increased by pinching the tubing, but the velocity should not be sufficient to cause any danger of splashing the sample over the sides of the sieve.

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