

APR 19 1929

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS

CIRCULAR OF THE BUREAU OF STANDARDS, No. 372

RECOMMENDED SPECIFICATION FOR
QUICKLIME AND HYDRATED LIME
FOR USE IN SOAP MAKING

MARCH 19, 1929



UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON : 1929

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY

RECOMMENDED SPECIFICATION FOR QUICKLIME AND HYDRATED LIME FOR USE IN SOAP MAKING

[This is the twelfth of a series of specifications for the lime used in various chemical processes. 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, of the Interior Department; the Bureau of Mines, of the Commerce Department; Bureau of Chemistry and Soils and the Forest Service, of the Department of Agriculture; and the Chemical Warfare Service, of the War Department. The present specification is based on a draft originally prepared by J. M. Porter, of the lime section, Bureau of Standards, and has been unanimously approved by the above conference and the National Lime Association.]

ABSTRACT

In the soap-making industry lime is used for causticizing and for neutralizing glycerine sweet waters. For these purposes lime should contain less than 2 per cent magnesia, less than 1.0 per cent insoluble material, and the available lime content of quicklime, or the available calcium hydroxide content of hydrated lime, should be at least 90 per cent.

CONTENTS

	Page
I. General.....	2
1. Definition of lime.....	2
2. Use of lime in soap making.....	2
3. Quality.....	2
4. Packing.....	2
II. Requirements.....	2
1. Composition.....	2
(a) Available lime.....	2
(b) Magnesia.....	3
(c) Insoluble material.....	3
2. Basis.....	3
III. Sampling and testing.....	3
1. Sampling.....	3
2. Testing.....	3
(a) Available lime.....	3
(b) Magnesia.....	4
(c) Insoluble material.....	6
(d) Nonvolatile matter.....	6
IV. Retesting.....	6

I. GENERAL

1. DEFINITION OF LIME

Quicklime is the product resulting from the calcination of limestone. Hydrated lime is a fine, white powder produced by the action of water on quicklime. It consists essentially of calcium hydroxide naturally associated with more or less magnesium oxide or hydroxide.

2. USE OF LIME IN SOAP MAKING

Lime is used for neutralization of glycerine sweet waters in the soap-making process. The glycerine water is obtained by steaming the mass of melted and purified fat with about one-third its volume of water in the presence of a predetermined quantity of saponifier reagent. The mixture is kept slightly acid with sulphuric acid during the boiling process. After settling, the glycerine water is drawn off, neutralized with lime, and evaporated to crude glycerine. The residue of fatty acids is again neutralized with lime to remove all soluble acids before the fatty acids are stored for later use in soap-making.

Lime is also used in soap making in the causticizing operation, where it is added to solutions containing alkali carbonates for the purpose of forming the corresponding hydroxides. These hydroxides remain in solution, the calcium carbonate precipitating.

3. QUALITY

For neutralization of glycerine water lime should be free from grit to prevent undue wear on pump linings. Magnesium compounds tend to form scale formations in evaporator tubes.

The impurities ordinarily found in lime, together with underburned and overburned lime, retard the slaking process, and increase the volume of the sludge produced in the causticizing process. Magnesium compounds do not settle readily in the solution of caustic.

4. PACKING

Quicklime is shipped in bulk in carload lots, in wooden barrels or metal drums holding 180 or 280 pounds each, net, or in waterproof bags holding 180 pounds net. Hydrated lime is shipped in paper bags holding 40 or 50 pounds net.

II. REQUIREMENTS

1. COMPOSITION

(a) AVAILABLE LIME.—Quicklime shall contain not less than 90 per cent available lime; hydrated lime shall contain not less than 90 per cent available calcium hydroxide.

(b) **MAGNESIA.**—Quicklime or hydrated lime shall contain not more than 2 per cent magnesium oxide.

(c) **INSOLUBLE MATERIAL.**—Quicklime or hydrated lime shall contain not more than 1.0 per cent insoluble material.

2. BASIS

The percentages enumerated herein are based on the nonvolatile portion of a sample taken at the point of shipment.

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, thus avoiding a disproportionate share of the top and bottom layers, which are most subject to changes. The sample shall comprise at least 10 shovelfuls taken from different parts of the shipment. The total sample shall weigh at least 100 pounds and shall be crushed to pass a 1-inch ring, mixed thoroughly, and "quartered" to provide a 15-pound sample for the laboratory.

When quicklime is shipped in barrels or waterproof bags at least 15 per cent of the number of containers shall be sampled. They shall be taken from various parts of the shipment, dumped, mixed, and sampled as in the above paragraph.

The sample of hydrated lime shall be a fair average of the shipment. Three per cent of the packages shall be sampled. The sample shall be taken from the center to the surface of the package. The material so obtained shall be mixed thoroughly 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 be placed immediately 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.

2. TESTING

Details regarding the complete analysis of lime are given in other papers of this series and in Bulletin No. 700 of the United States Geological Survey.

(a) **AVAILABLE LIME, OR CALCIUM HYDROXIDE.**—Place 1.40 g of the carefully prepared and finely ground (passing a No. 100 sieve) lime in a 400 ml beaker, add 200 ml of hot water, cover, heat carefully, and then boil for three minutes.

Cool, wash down the 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 the pink color disappears in streaks retard the rate of addition of acid somewhat, but continue until the pink color disappears entirely and does not reappear for one or two seconds. Note the reading and ignore the return of color.

Repeat the test, substituting for the 400 ml beaker a 1-liter graduated flask carrying a one-hole stopper fitted with a short glass tube drawn out to a point. Cool and add dropwise with vigorous stirring 5 ml less acid than before. Call the number of milliliters used "A." Grind up any small lumps with a glass rod flattened at one end, dilute to the mark with freshly boiled distilled water, close with a solid stopper, mix thoroughly for four or five minutes, and let settle for half an hour.

Pipette a 200 ml portion, add phenolphthalein and titrate slowly with half-normal hydrochloric acid until colorless on standing one minute. Call this additional number of milliliters of acid "B." Then the per cent of available lime in the quicklime or of available calcium hydroxide in the hydrated lime as received is $2A + 5B$. Recalculate this to the nonvolatile basis.

(b) **MAGNESIA.**—To 1.0 g of the sample in an evaporating dish add 10 ml of water to prevent lumping and then 10 ml of concentrated hydrochloric acid (specific gravity 1.19). The material should be ground with the flattened end of a glass rod until attack is complete. Then evaporate the solution to complete dryness on a steam or water bath. To hasten dehydration the residue may be heated to 150° C. or even 200° C. for one-half to one hour. Treat the residue with 10 ml of hydrochloric acid diluted with an equal amount of water. Cover the dish and digest the solution for 10 minutes on a steam bath or water bath. Dilute the solution to 75 ml, filter into a beaker, and wash the separated silica thoroughly with hot water.¹ Add 5 ml of concentrated hydrochloric acid and two drops of methyl red indicator (0.2 per cent alcoholic solution) to the filtrate (about 250 ml).²

Add dilute ammonium hydroxide dropwise until the color of the solution changes to a distinct yellow. Boil the solution for one or two minutes and filter at once. Wash the precipitate slightly with

¹ Since this procedure does not involve the determination of silica, a second evaporation is unnecessary.

² Manganese, if present, is distributed between the precipitates of iron and alumina, calcium, and magnesium. In the amounts usually present in lime it may be neglected. If it is desired to determine the small amount of manganese that may be present in the magnesium pyrophosphate, this may be done colorimetrically as described in the U. S. Geological Survey Bulletin No. 700, p. 153. If present in larger amounts, manganese should be precipitated with the iron and alumina, preferably by the persulphate method (U. S. Geological Survey Bulletin No. 700, p. 112). If this method is used, more aluminum passes into solution than in the method above described.

a hot 2 per cent solution of ammonium chloride (or ammonium nitrate). Set aside the filtrate, transfer the precipitate by a jet of hot water to the precipitating vessel, and dissolve in 10 ml of hot hydrochloric acid. Extract the paper with acid, adding the solution and washings to the solution of the precipitate. Reprecipitate the aluminum and iron as described above, collect the second precipitate on the filter used in the first instance, if this is still intact, and wash with a hot 2 per cent solution of ammonium chloride (or ammonium nitrate). To the combined filtrates from the hydroxides of iron and aluminum, reduced in volume if need be, add 1 ml of ammonium hydroxide, bring the solution to boiling, add an excess (about 65 to 70 ml) of a saturated solution of boiling ammonium oxalate, and continue boiling until the precipitated calcium oxalate has assumed a well-defined granular form. After one hour filter the precipitate and wash with a cold 1 per cent ammonium oxalate solution and set the filtrate aside. Place the wet filter in a platinum crucible, and burn off the paper over a small flame of a Bunsen burner and ignite. Cautiously moisten the residue with water, redissolve in hydrochloric acid, and dilute the solution to 100 ml; add ammonia in slight excess, boil, and filter if a precipitate appears. Reprecipitate by ammonium oxalate, allow to stand till settled, filter and wash. Acidify the combined filtrates from the calcium precipitates with hydrochloric acid, concentrate on the steam bath to about 150 ml and make slightly alkaline with ammonium hydroxide, boil and filter (to remove a little iron and aluminum, and perhaps calcium). When cool acidify the solution with hydrochloric acid, add 10 ml of saturated solution of sodium-ammonium-hydrogen phosphate, and ammonia, drop by drop, with constant stirring. When the crystalline ammonium-magnesium orthophosphate has formed add 10 ml of ammonia in excess. Set the solution aside overnight in a cool place, filter, and wash with water containing 2.5 per cent NH_3 . Dissolve the precipitate in a small quantity of hot hydrochloric acid, dilute the solution to about 100 ml, add 1 ml of a saturated solution of sodium-ammonium-hydrogen phosphate and ammonia, drop by drop, with constant stirring, until the precipitate is again formed as described above and the ammonia is in moderate excess. Allow the precipitate to stand about two hours; filter and wash as before. Place the paper and contents in a weighed platinum crucible, char the paper slowly, and carefully burn off the resulting carbon. Ignite the precipitate to constant weight over a Meeker burner, or a blast not strong enough to soften or melt the pyrophosphate. The weight of magnesium pyrophosphate obtained multiplied by 36.2 gives the percentage of magnesia. Recalculate this to the nonvolatile basis. The pre-

cipitate so obtained always contains some calcium and usually small quantities of iron, aluminum, and manganese as phosphates.³

(c) **INSOLUBLE MATERIAL.**—Treat 1 g of the sample in a 250 ml beaker with 100 ml of hydrochloric acid (1:9), and grind with the flattened end of a glass rod until decomposition appears complete. Let stand on the steam bath one hour, filter, wash thoroughly with hot water, and ignite to constant weight in a platinum crucible. Calculate the residue as percentage insoluble material on the non-volatile basis.

(d) **NONVOLATILE MATTER.**—Weigh out a 1 g sample and ignite to constant weight in a platinum crucible. The weight of the material left is the weight of the nonvolatile matter.

IV. RETESTING

Notice of the rejection of a shipment based on these specifications must be in the hands of the consignor within 10 working 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 working days of receipt of the notice of rejection. The original testing laboratory shall keep the sample sealed air-tight from the time they have taken out enough material for the original test until the expiration of the 15 days noted. The original testing laboratory shall agree to transmit the sample to any other laboratory for a retest at the direction of both the contracting parties. The retest shall be at the expense of the consignor.

WASHINGTON, August 2, 1928.

³ As the liquid in which the magnesium is precipitated contains all the material added in the analysis, careless addition of reagents may give so strong a solution of ammonium and sodium salts that the precipitation of the magnesium phosphate will not take place promptly. However, the precipitation will be complete in time. To get rid of the ammonium salts, the filtrate should be evaporated to dryness with 3 ml of concentrated HNO_3 for each gram of NH_4Cl , which will decompose and remove ammonium salts. Then any remaining magnesium may be precipitated.

