

1928 1928

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS
George K. Burgess, Director

CIRCULAR OF THE BUREAU OF STANDARDS No. 373

RECOMMENDED SPECIFICATIONS FOR QUICKLIME
FOR USE IN THE DISTILLATION OF AMMONIA
FROM AMMONIA LIQUORS OBTAINED IN
COKE AND GAS MANUFACTURE

DECEMBER 26, 1928



PRICE 5 CENTS

Sold only by the Superintendent of Documents, U. S. Government Printing Office
Washington, D. C.

UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON

1929

RECOMMENDED SPECIFICATION FOR QUICKLIME FOR USE IN THE DISTILLATION OF AMMONIA FROM AMMONIA LIQUORS OBTAINED IN COKE AND GAS MANUFACTURE

[This is the thirteenth 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; the Bureau of Chemistry and Soils and 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 approved unanimously by the above conference and by the National Lime Association.]

ABSTRACT

Quicklime is used in the distillation of ammonia in two ways: (1) In the concentration of crude ammonia liquors, and (2) in the distillation of concentrated ammonia liquors to produce ammonical products. For these purposes the quicklime should slake to form a product of which not less than 95 per cent will pass a No. 50 sieve, and not more than 10 per cent will be retained on a No. 200 sieve. The available lime content should be 90 per cent or more, based on the nonvolatile portion of the material.

CONTENTS

	Page
I. General.....	2
1. Definition of quicklime.....	2
2. Use of lime in distillation of ammonia.....	2
3. Quality.....	2
4. Packing.....	2
II. Requirements.....	2
1. Size.....	2
2. Fineness.....	2
3. Composition.....	2
III. Sampling and testing.....	3
1. Sampling.....	3
2. Testing.....	3
(a) Available lime.....	3
(b) Fineness.....	4
(c) Nonvolatile matter.....	4
IV. Retesting.....	4

I. GENERAL

1. DEFINITION OF QUICKLIME

Quicklime is the product resulting from the calcination of limestone.

2. USE OF LIME IN DISTILLATION OF AMMONIA

Quicklime is used in the distillation of ammonia liquors in two ways: (1) In the concentration of crude ammonia liquors at gas plants or by-product coke-oven plants for shipment to chemical factories, and (2) in the distillation of concentrated ammonia liquors to produce aqua ammonia, anhydrous ammonia or ammonium salts.

3. QUALITY

The impurities ordinarily found in lime, together with underburned and overburned lime, retard the slaking process. The presence of any inert material will thus introduce delays which reduce the efficiency of the equipment. High calcium quicklime is employed except in a limited number of cases wherein a magnesium lime may be desirable.

4. PACKING

Quicklime is shipped in bulk in carload lots, in wooden barrels or metal drums holding 180 or 280 pounds each, or in waterproof bags holding 180 pounds.

II. REQUIREMENTS

1. SIZE

The maximum allowable size of lumps and the percentage of fines shall be agreed upon between the lime producer and the ammonia manufacturer.

2. FINENESS

At least 95 per cent by weight of the thoroughly slaked lime shall pass a standard No. 50 sieve and not more than 10 per cent shall be retained on a standard No. 200 sieve.

3. COMPOSITION

Quicklime shall contain not less than 90 per cent available calcium oxide, CaO, based upon 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 taken 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.

When sampling quicklime, 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

(a) AVAILABLE LIME.—Place 1.4 g of the carefully prepared and finely ground (passing a No. 100 sieve) quicklime 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 1 or 2 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 1-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 slightly 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 sample as received is $2A+5B$. Calculate this result to the nonvolatile basis using the data from (c).

(b) FINENESS.—Determine fineness of quicklime as follows: Thoroughly slake the lime by adding 100 g of the laboratory sample to 500 ml of water. Stir the mixture slowly until the violent evolution of heat has ceased. Stir six times during the next 24 hours. Slow stirring for two minutes at each of the six times is recommended.

Strain the resulting milk of lime through a standard No. 50 sieve nested above a standard No. 200 sieve. Wash the residues on the No. 50 sieve thoroughly until the water passing into the No. 200 sieve is clear. The No. 50 sieve may then be removed and the No. 200 sieve washed in a similar manner.¹ Washing may be considered complete when the water passing through the sieve is clear, but washing should not be carried on for over 30 minutes.

Dry the sieves containing the residues to constant weight in an oven at a temperature of 110 to 120° C. The residues on each sieve may then be removed and weighed, thus obtaining the per cent retained on each sieve. Add the weight remaining on the No. 50 sieve to that remaining on the No. 200 sieve to obtain the total residue on the No. 200 sieve.

(c) NONVOLATILE MATTER.—Weigh out a 1 g sample and ignite to constant weight at approximately 1,200° C. 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 days after the receipt of the shipment at the destination. If the consignor desires a retest, he shall notify the consignee within five 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 1, 1928.

¹ A convenient length of rubber tubing attached to the water tap provides a suitable method for washing the sieves. The discharge end of the tubing may be pinched to control the velocity of the stream of water, but care must be observed that the velocity is not so great that any of the sample is splashed over the sides of the sieves.