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RECOMMENDED SPECIFICATION FOR QUICKLIME FOR USE IN CAUSTICIZING.

This is the third 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 R. C. Wells, physical chemist, U. S. Geological Survey, has been unanimously approved by the above conference. It has the approval, with reservation, of the National Lime Association, and has been approved by the Technical Association of the Pulp and Paper Industry, representing the largest users of this kind of lime.

ABSTRACT.

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

The standard of quality for lime for this purpose is set at 85 per cent available lime. Any shipment containing less than 70 per cent available lime, or more than 3 per cent magnesia should be rejected as uneconomical to use.

Complete directions for sampling, testing, and retesting are included.

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1. GENERAL.

(a) DEFINITION OF QUICKLIME.—Quicklime is a 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) USE OF LIME IN CAUSTICIZING.—Lime is added to solutions containing alkali carbonates for the purpose of forming the corresponding hydroxides, or caustic alkalis. These remain in solution, while calcium carbonate is precipitated. The solution and precipitate are separated; the solution is concentrated by evaporation to the extent desired for the object in view, and the calcium carbonate sludge is either converted into lime or discarded.

(c) QUALITY.—The impurities ordinarily found in lime, as well as underburned and overburned lime, retard the slaking process. They also increase the volume of the sludge, which then either requires more time in washing or carries off more inclosed caustic alkali. Magnesium compounds do not settle either quickly or well in the solution of caustic. The presence of any impurities thus introduces delays which reduce the efficiency of the equipment.

(d) PACKING.—Quicklime is shipped either in bulk in carloads lots, or in barrels each holding 180 or 280 pounds net.

2. REQUIREMENTS.

(a) COMPOSITION.—The standard of composition for quicklime for use in causticizing shall be a content of 85 per cent of available lime.¹ Lime containing more than 3 per cent magaesia or less

¹ It is recommended that a bonus or a penalty of 1½ per cent of the contract price be added to or deducted from the payment for each 1 per cent of available lime above or below the standard 85 per cent. It may be desirable in some cases to make other constituents than lime subject to bonus or penalty provisions.

than 70 per cent of available lime shall be rejected as uneconomical to use. All percentages enumerated herein are based on the sample as received at the laboratory.

(b) 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.

(c) 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 resample the material. The 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, and shall be crushed to pass a 1-inch ring, 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.

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 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 = $2A + 5B$.

Magnesia.—The magnesia determination is made after removing silica, iron oxide, alumina, and lime. Ignite 1 g, or if the magnesium content is high, 0.5 g, of the sample strongly in a platinum crucible for 15 minutes. When cool, moisten the residue and dissolve it in 1 : 1 hydrochloric acid, evaporate to dryness on the steam bath, take up in 5 cc of hydrochloric acid, add 50 cc of water, and filter off the silica. To the filtrate add 5 cc of hydrochloric acid, then make it slightly alkaline with ammonia, heat to boiling, and filter off the oxides of iron and aluminum. Acidify the filtrate slightly, heat to boiling, add ammonium oxalate slowly to excess, then ammonia slowly until in slight excess. After the precipitate has settled for one to two hours filter and wash with an ammoniacal ammonium oxalate solution. Reserve the filtrate. Ignite the precipitate. When cool moisten it, dissolve in hydrochloric acid, and reprecipitate. Filter off the calcium oxalate, washing with ammoniacal ammonium oxalate solution. Discard the precipitate,

combine the filtrate with the first one, slightly acidify with hydrochloric acid, and add an excess of microcosmic salt. Then slowly add ammonia until a crystalline precipitate forms or the solution is alkaline. Finally add one-fifth of the volume of ammonia water in excess. After the precipitate has fully formed, preferably by standing overnight, filter and wash with dilute ammonia water (5 per cent by volume). Dissolve the precipitate in a slight excess of hydrochloric acid, add one or two drops of phosphate solution, and reprecipitate by adding ammonia while stirring, finally adding 2 or 3 cc of ammonia in excess. Filter, wash with 5 per cent ammonia, and ignite to constant weight. Weight of magnesium pyrophosphate $\times 0.3621$ = weight of magnesium oxide.

For rapid work the first precipitate of magnesium phosphate may be ignited, or it may be washed slightly with dilute ammonia, then with alcohol until free from ammonia, dried on the paper at 80° C. in a small beaker, dissolved in an excess of tenth-normal sulphuric acid, and titrated back with tenth-normal sodium hydroxide, using methyl orange as indicator (cc acid - cc alkali) $\times 0.00201$ = grams magnesium oxide.

WASHINGTON, April 11, 1923.

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