U. S. Gov't
Master
Specification
No. 249

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
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CIRCULAR OF THE BUREAU OF STANDARDS, No. 204

[Issued March 23, 1925]

UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR HYDRATED LIME FOR STRUCTURAL PURPOSES

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 249

This specification was officially promulgated by the Federal Specifications Board on November 5, 1924, for the use of the Departments and Independent Establishments of the Government in the purchase of hydrated lime for structural purposes.

CONTENTS

I. TYPES

Hydrated lime sold under this specification may be of either of two types—M, masons; F, finishing.

II. MATERIAL AND WORKMANSHIP

Hydrated lime sold under this specification 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.

III. GENERAL REQUIREMENTS

1. Chemical Composition.—Hydrated lime shall contain not less than 95 per cent of calcium and magnesium oxides computed on the nonvolatile basis.

It shall contain not more than 5 per cent carbon dioxide (computed on the nonvolatile basis) if the sample is taken at the place of manufacture, nor more than 7 per cent if the sample is taken elsewhere.

2. Fineness.—Hydrated lime shall leave a residue of not more than 0.5 per cent on a No. 30 sieve nor more than 15 per cent on a No. 200 sieve.

3. Soundness.—Hydrated lime when tested by the method herein described shall show no popping or disintegration of the test specimen.

IV. DETAIL REQUIREMENTS

1. Plasticity.—Finishing hydrated lime shall have a plasticity figure greater than 200.

V. METHOD OF INSPECTION AND TESTS

1. Sampling.—One and one-half per cent of the number of packages (but not less than five packages) shall be sampled. Samples shall be taken by means of a sampling tube not less than one-half inch in diameter, inserted longitudinally in the package and extending its entire length. The material so collected shall be mixed and quartered to 15 pounds. This shall be divided into three parts of 5 pounds each, which shall immediately be sealed in air-tight containers. One of these shall be sent to the consignor, one to the consignee, and the third held for retest in case of dispute.

Sampling shall be conducted as expeditiously as possible in order to avoid undue exposure to the air. Samples shall not be taken from broken packages.

2. Chemical Analysis.—(*a*) *Calcium oxide.*—Weigh out 0.5 g of the material and transfer to a platinum or porcelain evaporating dish, mix to a thin slurry with distilled water, add 5 to 10 cc of HCl (sp. gr. 1.20) and digest with gentle heat and agitate until solution is complete. Evaporate this to dryness so far as this may be possible on the water bath. Heat the dish and its contents on a

hot plate for one hour at 120° C. Drench the cooled mass with HCl (sp. gr. 1.20) and allow to stand for a few minutes. Add an equal volume of water and heat on water bath for 10 minutes. Filter. Wash thoroughly with dilute HCl and then twice with cold water. Evaporate the filtrate to dryness. Extract with HCl as before, but allowing only a few minutes time. Filter through a second paper. Ignite both precipitates to constant weight in a platinum crucible. Add 5 cc HF and two drops H_2SO_4. Evaporate to dryness and ignite for two or three minutes. Fuse the residue with a little Na_2CO_3. Dissolve the cooled melt in HCl and add the solution to the filtrate obtained above. Add a few drops of bromine water or nitric acid and boil until all trace of Br or Cl is gone. Then add HCl if necessary to insure the presence of 10 cc of concentrated acid. Add a few drops of methyl red solution, dilute to 200 cc and boil. Neutralize with 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 two or three times with a hot 2 per cent solution of NH_4Cl. Suck dry. Dissolve the precipitate by letting hot dilute HCl run through the paper into the beaker in which the precipitation was made. Wash thoroughly with hot water. Boil to expel any trace of Cl, and reprecipitate with NH_4OH as described above. Filter, wash two or three times with a hot 2 per cent solution of NH_4Cl, and combine this filtrate with the one obtained above. Add a few drops of NH_4OH and boil. 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. Allow to stand 20 minutes or until the supernatant liquid is clear. Filter and wash thoroughly with hot water. Ignite the precipitate in a platinum crucible. Dissolve in hot dilute HCl and make up to 100 cc with water. Add a slight excess of NH_4OH and boil. If any $Al_2(OH)_6$ separates out, filter, wash with 2 per cent NH_4Cl and discard the precipitate. Treat the filtrate with $(NH_4)_2C_2O_4$ and proceed as before. Ignite the precipitate to constant weight in a platinum crucible, and record as calcium oxide.

(*b*) *Magnesium oxide.*—Acidify the filtrate from the calcium determination (see above paragraph) with HCl, evaporate to 150 cc and boil. Add 10 cc of a saturated solution of $NaNH_4HPO_4$ and continue the boiling for several minutes. Cool to room temperature. Add NH_4OH drop by drop with constant stirring, then add a moderate excess of NH_4OH and continue stirring for several minutes. Let stand in a cool atmosphere for 12 to 48 hours and filter. Dissolve the precipitate in hot dilute HCl, dilute to 100 cc add 1 cc of saturated solution of $NaNH_4HPO_4$, and reprecipitate with NH_4OH as before. Filter and wash with dilute NH_4OH (2½ per cent) containing a little NH_4NO_3. Ignite the precipitate and weigh as $Mg_2P_2O_7$. Multiply this weight by 0.3621 to find the weight of magnesium oxide.

(*c*) *Carbon dioxide.*—Weigh out a 5 g sample and transfer it to a small Erlenmeyer flask. Put this flask in a carbon dioxide train composed as follows: After the flask comes an upwardly inclined condenser, then U-tubes containing $CaCl_2$, anhydrous $CuSO_4$, and $CaCl_2$, than a U-tube filled with porous soda-lime, and finally a U-tube containing half soda-lime and half $CaCl_2$, arranged so that the $CaCl_2$ comes last. Pour some hot water on the sample, connect the flask in the train and pass a current of CO_2 free air through all of the train excepting the soda-lime tubes. Weigh and attach the soda-lime tubes. Through a separatory funnel, let some 1:1 HCl into the flask slowly. When any apparent action has ceased, heat the flask gradually to boiling, and continue boiling until no more gas is evolved. Cool gradually. The current of CO_2 free air is continued throughout the experiment. Detach the soda-lime tubes, close them, and let them stand in the balance case, weighing them at 30-minute intervals until two successive weighings agree within 0.5 mg. The gain in weight of the soda-lime tubes is recorded as the weight of the carbon dioxide.

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

(*e*) *Calculation.*—The weights in grams of the oxides of calcium and magnesium are added together and multiplied by 200 to reduce to per cent. The weight of the carbon dioxide is multiplied by 20. Each of these figures is then divided by the weight of the nonvolatile matter as found in the preceding paragraph, to reduce it to the nonvolatile basis called for in the requirements.

3. FINENESS.—Place 100 g of the sample in a No. 30 sieve which is nested above a No. 200 sieve. Wash the material through the sieve by means of a stream of water from a faucet. Wash until the water comes through clear, which point must be reached within 30 minutes. Dry the residue to constant weight in an atmosphere free from CO_2 and at a temperature of 100 to 120° C. The weights of the residues are calculated to percentages of the sample. The residue on the No. 200 sieve must be corrected by the addition of any material previously removed by the No. 30 sieve.

The sieves used shall meet the requirements given in the Bureau of Standards screen scale, as published in Letter Circular 74, April 15, 1924.

A small piece of rubber tubing attached to a water faucet is to be used for the washing. The velocity of the water may be increased by pinching the tube, but should not be sufficient to cause any splashing of the sample over the sides of the sieve.

Care must be taken not to let water accumulate on the No. 200 sieve, else the openings will become clogged and the operation can not be completed in 30 minutes.

4. SOUNDNESS.—To 20 g of the sample, 100 g of standard Ottawa sand is added, mixed thoroughly, and enough water added to the mixture to make a good plastic mortar of rather dry consistency. This is spread on a clean glass plate to form a layer about one-fourth inch thick by 4 inches square. The pat is of even thickness throughout, and not tapering at the edges. If the mortar is too dry to work well, more water may be added. The pat is placed in a closet at a temperature of 65 to 75° F. for 24 hours. Free circulation of air is provided for in the closet, but without direct draft striking the pat. The pat is then soaked in water until a film of water will remain unabsorbed on its surface. If the pat has cracked, the consistency of the mortar was too wet, and a new pat must be made.

Mix 20 g of the sample with enough water to form a thick cream. Spread this in a thin layer on the surface of the pat. Let stand 15 minutes to permit air bubbles to form. Trowel to an even surface, making this skim coat as thin as possible without allowing the sand to show through. Replace the pat in the closet for 24 hours.

Suspend the pat in a vessel partially filled with cold water in such a way that the water can boil without touching the pat. Gradually bring the water to boiling and keep boiling gently for five hours, the pat being surrounded with steam during this time. Permit the water to cool for at least 12 hours. Remove and examine the pat.

5. STANDARD CONSISTENCY.—Make the lime into a stiff putty with water and let soak overnight. Mold in a Vicat rubber ring on a glass plate.

The needle used is a modified form of Vicat needle, 12.5 mm diameter and weighing 30 g. It may be conveniently made from a suitable piece of aluminum tubing, the lower end being closed without shoulders or curvature, and the tube loaded with shot to the specified weight. It is mounted in the Vicat needle stand.

The initial reading is taken with the bottom of the needle in contact with the surface of the sample. The final reading is taken 30 seconds after the needle is released. A penetration of 20 mm shall be considered as standard.

If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, and retested. If the penetration is more than standard, the sample must be discarded and a new one prepared.

6. PLASTICITY—Plasticity is measured by means of the instrument shown in Figure 1 as follows: 300 g of the sample are mixed

with enough water to form a thick putty. After aging for not less than 16 nor more than 24 hours, this putty is adjusted to standard consistency as defined in section V, 6, with a permissible variation of ±5 mm.

A Vicat rubber ring is placed on the porcelain base plate of the instrument, filled with the paste, and struck off level. Remove the ring by raising it vertically without distorting the paste. The base plate and paste are placed in the instrument and the carriage turned up by hand until the surface of the paste is in contact with the disk and the distance between the bottom of the disk and the top of the base plate is 1¼ inches.

The carriage is then thrown into gear and the motor started. It is essential that the motor shall be started exactly 120 seconds after the first portion of the paste was put in the mold. Record the scale reading every minute until the test is completed.

The test shall be considered complete when (*a*) the scale reading reaches 100, (*b*) any reading is less than the one before, or (*c*) the scale reading remains constant for three consecutive readings (two minutes) and the specimen has visibly ruptured.

The plasticity figure is calculated from the formula

$$P=\sqrt{F^2+(10T)^2}$$

in which P is the plasticity figure, F is the scale reading at the end of the experiment and T is the time in minutes from when the first portion of the paste was put in the mold to the end of the experiment.

VI. PACKING AND MARKING

1. Packing.—Hydrated lime is shipped in cloth bags holding 100 pounds each, or paper bags holding 50 pounds each.

2. Marking.—Each bag shall have legibly marked thereon the net weight of its contents, the name of the manufacturer, the place of manufacture, the brand (if any) and some means of identifying the particular contract on which the purchase and shipment were made.

VII. ADDITIONAL INFORMATION

There are two kinds of hydrated lime, known to the trade as "masons" and "finishing." Masons hydrate is intended to be used for any structural purpose except the finishing coat of plaster. Finishing hydrate is intended for use in the finishing coat of plaster, but may be used for any structural purpose.

VIII. GENERAL SPECIFICATIONS

No details.

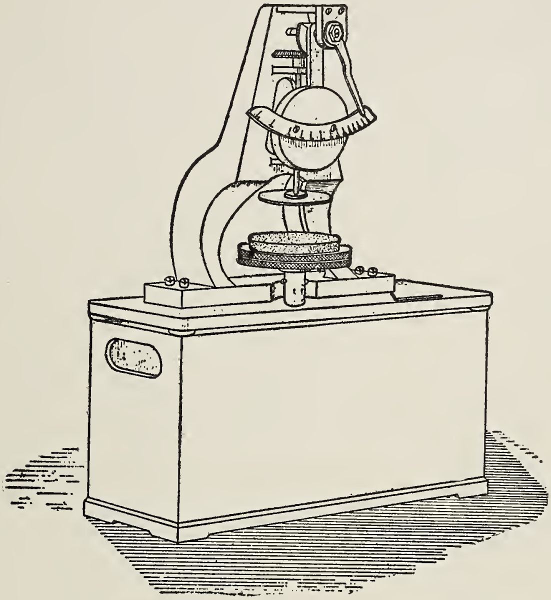

FIG. 1.—*Emley plasticimeter*

Constants of the machine: Absorption of porcelain base plate, 20 to 25 per cent. Dimension of base plate, 1 inch in thickness by 4 inches in diameter. Dimensions of disk, one-thirty-second inch in thickness by 3 inches in diameter. Speed of vertical shaft, one revolution in 6 minutes, 40 seconds. Upward movement of base plate, one-thirteenth inch per revolution. Torque on disk when bob reading is 100, 14,400 gcm.

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