U. S. Gov't Master Specification No. 250

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR QUICKLIME FOR STRUCTURAL PURPOSES

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 250

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 quicklime for structural purposes.

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I. TYPES

Quicklime sold under this specification may be of either of two types: C, calcium; M, magnesium.

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II. MATERIAL AND WORKMANSHIP

Quicklime sold under this specification shall be well burned from a good quality of limestone, dolomite, or marble.

III. GENERAL REQUIREMENTS

Quicklime shall be reasonably free from ashes, core, overburned lime, or similar deleterious foreign matter.

1. CHEMICAL COMPOSITION

The quicklime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

	Per cent
Calcium and magnesium oxides, minimum	95
Silicon, aluminum and iron oxides, maximum	5
Carbon dioxide, maximum:	
If sample is taken at the kiln	3
If sample is taken elsewhere	10

2. WASTE

When tested in accordance with the method given below, the quicklime shall contain not more than 3 per cent by weight of waste.

IV. DETAIL REQUIREMENTS

Class C (calcium) lime shall contain not less than 75 per cent calcium oxide.

Class M (magnesium) lime shall contain not less than 20 per cent magnesium oxide.

V. METHOD OF INSPECTION, TESTS, ETC.

1. SAMPLING

In sampling bulk lime 10 shovelfuls (not less than 100 pounds) shall be selected by an inspector from different parts of the car. This shall be broken, if necessary, so that it will all pass a 1-inch ring. It shall be mixed and quartered to obtain a final sample of 15 pounds. This shall be divided into three equal parts and each part shall be sealed in an air-tight container.

In sampling lime in barrels 2 per cent of the number of containers (but never less than five barrels) shall be opened and their contents dumped in a pile. This pile shall then be sampled as under "bulk lime."

Sampling shall be done as expeditiously as possible in order to avoid undue exposure of the material to the air.

One of the triplicate samples shall be sent to the consignor, one to the consignee, and the third shall be held for retest in case of dispute. Samples shall not be taken from broken packages.

2. CHEMICAL ANALYSIS

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 agitation until solution is complete. Evaporate this to dryness so far as this may be possible on the water bath.

(*a*) Silica.—Heat the dish and its contents (see above paragraph) on a hot plate for one hour at 200° C. for calcium lime or 120° C. for magnesium lime. 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 to constant weight in a platinum crucible. Add 5 cc of HF and two drops of H_2SO_4. Evaporate to dryness and ignite for two or three minutes. The loss of weight is recorded as silica.

(*b*) Iron and Aluminum Oxides.—Fuse the residue in the platinum crucible (see above paragraph) with a little Na_2CO_3. Dissolve the cooled melt in HCl and add the solution to the filtrate from the silica. Add a few drops of bromine water or HNO_3 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 chlorine, and reprecipitate with NH_4OH as described above. Ignite the precipitate in a platinum crucible and weigh as oxides of iron and aluminum.

(*c*) Calcium Oxide.—To the filtrate from the iron and aluminum (see above paragraph) add a few drops of NH_4OH and boil. Add 35 cc of a saturated solution of ammonium oxalate and continue the boiling until the precipitated calcium oxalate 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 aluminum hydroxide separates out, filter, wash with 2 per cent NH_4Cl, ignite, weigh, and add this weight to the oxides of iron and aluminum determined previously. Precipitate with ammonium oxalate as before, ignite to constant weight in a platinum crucible, and weigh as calcium oxide.

(*d*) 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 few drops of 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.

(*e*) 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 $CaCl_2$, then 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 to the train, and pass a current of CO_2 free air through all of the train excepting the two soda-lime tubes. Close, 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, 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.

(*f*) 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.

(*g*) Calculations.—The weights in grams of the oxides of silicon, iron, aluminum, calcium, and magnesium, as found above, are multiplied by 200 to reduce them to percentages. The weight of the carbon dioxide in grams is multiplied by 20. All of these figures must then be divided by the weight of the nonvolatile matter as found in the preceding paragraph to reduce them to the nonvolatile basis called for in the table of requirements.

3. WASTE

After the sample for chemical analysis has been set aside all of the rest of the sample as received shall be screened on a No. 3 sieve. Weigh the lime retained on the sieve and transfer it to a slaking box.

This lime is then slaked by an experienced operator to produce the maximum quantity of putty, care being taken to avoid "burning" or "drowning" the lime. Cover the box to prevent evaporation and let stand 24 hours. Transfer the putty to a No. 20 sieve, and wash it through the sieve with a stream of water from a hose. This operation must be completed within 30 minutes. The sieve and the residue contained thereon are then dried to constant weight in a current of CO_2 free air at a temperature of 110 to 120° C. The dry weight of this residue is divided by the weight of the lime slaked, multiplied by 100, and the result reported as "per cent waste."

VI. PACKING AND MARKING

1. PACKING

Quicklime may be delivered in bulk if in carload lots, otherwise it shall be delivered in barrels holding 180 or 280 pounds each.

2. MARKING

Each barrel of lump lime shall be legibly marked with a description of the contents, the name of the manufacturer, the place of manufacture, the brand (if any), the net weight of the contents, and some means of identification of the particular contract on which purchase and shipment were made.

Each carload of bulk lime shall carry the above information on a card conspicuously posted inside the car.

Most of the above marking is required by the Federal lime barrel act, Public No. 228, Sixty-fourth Congress.

VII. ADDITIONAL INFORMATION

No details.

VIII. GENERAL SPECIFICATIONS

No details.

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