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S. W. STRATTON, Director.

CIRCULAR OF THE BUREAU OF STANDARDS.

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UNITED STATES GOVERNMENT SPECIFICATION FOR  
HAND GRIT SOAP.<sup>1</sup>

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION No. 35.

This Specification was officially adopted by the Federal Specifications Board on June 20, 1922, for the use of the Departments and Independent Establishments of the Government in the purchase of materials covered by it.

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1. GENERAL.

The material desired under this specification is a high-grade cake soap containing about one-third its weight of clean, finely divided insoluble siliceous matter, as free as possible from water, either uncolored or colored in a manner indicated in the contract, either unscented or perfumed in a manner indicated in the contract for the same, thoroughly saponified, and well compressed in firm, smooth cakes.

Bidder shall state size, weight, and number of cakes in each box.

Failure to meet any of the following requirements will be cause for rejection:

Matter volatile at 105° C. shall not exceed 25 per cent. Deliveries which yield more than 25 per cent of volatile matter will be rejected without further test.

<sup>1</sup> For Specification for Grit Cake Soap see B. S. Circular No. 130; For Specification for Scouring Compounds see B. S. Circular No. 131.  
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Alkali as alkaline salts (total alkalinity of matter insoluble in alcohol), calculated as sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), shall not exceed 1 per cent.

Free alkali, calculated as sodium hydroxide ( $\text{NaOH}$ ), shall not exceed 0.1 per cent.

Insoluble siliceous material shall be not less than 25 nor more than 40 per cent. This siliceous material must be so finely ground that not less than 98 per cent of it will pass through a No. 100 screen and not less than 90 per cent through a No. 200 screen.

Rosin, sugar, and foreign matter shall not be present.

Anhydrous soda soap shall be within 1 per cent of the difference between 100 and the sum of matter volatile at  $105^\circ\text{C}$ ., insoluble siliceous material, and alkali as alkaline salts.

The percentage of matter volatile at  $105^\circ\text{C}$ . will be computed on the basis of the soap as received, but all other constituents will be calculated to the basis of material containing 25 per cent of matter volatile at  $105^\circ\text{C}$ .

Material will be purchased by net weight.

## 2. SAMPLING.

One cake shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain not less than 50 pounds each. In the case of smaller containers a cake shall be taken at random from each lot of containers totaling not to exceed 5,000 pounds. The total sample shall in all cases consist of not less than three cakes taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 20 pounds. Wrap the individual cakes tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped cake, record its weight and the date of weighing on the wrapper, place the wrapped cakes in an air-tight container, which should be nearly filled, seal, mark, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling and when he so requests shall be furnished with a duplicate sample.

## 3. LABORATORY EXAMINATION.

(a) PREPARATION OF SAMPLE.—Select a cake of average weight, quarter by cutting at right angles in the center, and shave equally from all freshly cut surfaces sufficient soap for analysis. Mix

and weigh out all portions for analysis promptly. Preserve the remainder in an air-tight container in a cool place. (With material that can not be quartered as above, break a cake of average weight and shave the sample from the freshly broken surfaces.)

When a determination shows nonconformity with specification, a duplicate shall be run.

(b) **MATTER VOLATILE AT 105° C.**—Weigh 5 g of the sample in a porcelain or glass dish, about 6 to 7 cm in diameter and 4 cm deep, dry to constant weight in a vacuum oven or an inert atmosphere at a temperature not exceeding 105° C. Report loss in weight as matter volatile at 105° C.

(c) **TOTAL MATTER INSOLUBLE IN ALCOHOL, FREE ALKALI, OR FREE ACID.**—(1) *Matter Insoluble in Alcohol.*—Digest hot a 5 g sample with 100 cc of freshly-boiled neutral ethyl alcohol (94 per cent or higher). Filter through a counterpoised filter paper neutral to phenolphthalein, or a weighed Gooch crucible with suction into a dry weighed beaker, protecting the solution during the operation from carbon dioxide and other acid fumes. Wash the residue on the paper or in the crucible with hot neutral alcohol until free from soap. Dry the filter paper or crucible and residue at 100 to 105° C. for three hours, cool, and weigh the total matter insoluble in alcohol. (Since the percentage of the matter insoluble in alcohol is not required under this specification, time may be saved by omitting the drying and weighing and proceeding directly with the moist residue to the determination of matter insoluble in water (3).)

(2) *Free Alkali or Free Acid.*—Titrate the filtrate from the above, using phenolphthalein as indicator, with standard acid or alkali solution and calculate the alkalinity to sodium hydroxide (or potassium hydroxide) or acidity to oleic acid.

(3) *Matter Insoluble in Water.*—Proceed as in the determination of matter insoluble in alcohol. After filtering and thoroughly washing the residue extract it with water at 60° C. and wash the filter thoroughly. (When the matter insoluble in water is all inorganic, boiling water may be used for the extraction and washing.) Dry the filter and residue at 100 to 105° C. for three hours, cool, and weigh matter insoluble in water. The nature of this may be determined by further examination. The insoluble matter should be siliceous.

(4) *Alkali as Alkaline Salts (Total Alkalinity of Matter Insoluble in Alcohol).*—Titrate the filtrate from the determination of matter



insoluble in water with standard acid, using methyl orange as indicator. Calculate alkalinity to sodium carbonate ( $\text{Na}_2\text{CO}_3$ ).

(d) SCREEN TEST.—Transfer a weighed sample of the insoluble siliceous material to a No. 100 screen and carefully brush through. Weigh the amount passing through and calculate percentage. After weighing transfer to a No. 200 screen and treat in the same manner, weigh the amount passing through, and calculate percentage.

(e) TOTAL ANHYDROUS SOAP.—Evaporate the alcoholic solution obtained after filtering off and washing the matter insoluble in alcohol—(c), (1) and (2)—to dryness, dry at  $105^\circ\text{C}$ . to constant weight. Report the result as total anhydrous soap.

(f) ROSIN.—A qualitative test for rosin may be made as follows: After decomposing a solution of the soap and separating the fatty acids heat a small quantity of the latter with acetic anhydride, cool, place a few drops on a spot plate, and add a drop of  $\text{H}_2\text{SO}_4$  (specific gravity = 1.53) to this. A fugitive violet color indicates the presence of rosin.

(g) SUGAR.—A qualitative test for sugar may be made as follows: Add a decided excess of hydrochloric acid to a solution of the soap, heat on a steam bath for 15 minutes, cool, filter from fatty acids, and test a portion of the filtrate which has been neutralized with sodium hydroxide solution by boiling for 2 minutes with an equal volume of boiling Fehling solution. The formation of red cuprous oxide indicates the presence of sugar.

#### 4. REAGENTS.

(a) STANDARD SODIUM HYDROXIDE SOLUTION.—0.25 N, or about 10 g sodium hydroxide dissolved in water and diluted to 1 liter. Standardized against Bureau of Standards benzoic acid.

(b) STANDARD SULPHURIC ACID SOLUTION.—0.5 N, or about 25.8 g strong sulphuric acid (specific gravity = 1.84) diluted to 1 liter. Standardized against standard sodium hydroxide solution (a).

(c) SULPHURIC ACID (SPECIFIC GRAVITY = 1.53).—Mix 62.5 cc of sulphuric acid (specific gravity = 1.84) with 61.5 cc of water.

(d) FEHLING SOLUTION.—(1) *Copper Sulphate Solution*.—Dissolve 34.639 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water and dilute to 500 cc.

(2) *Alkaline Tartrate Solution*.—Dissolve 173 g. of Rochelle salts ( $\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) and 50 g of sodium hydroxide in water and dilute to 500 cc. Mix equal volumes of (1) and (2) immediately before use.



