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S. W. STRATTON, Director.

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UNITED STATES GOVERNMENT SPECIFICATION FOR CHIP SOAP.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION No. 31.

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 soap desired under this specification is a soap in chip form made from soda and fats, without rosin, as free as possible from water and all substances other than true soap, of a light uniform color, free from disagreeable odor, and suitable for high grade laundry work with soft water, when the presence of alkaline salts is objectionable. Bidder shall state size and weight of package.

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

Matter volatile at 105° C. shall not exceed 15 per cent. Deliveries which yield more than 15 per cent of volatile matter will be rejected without further test.

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The sum of free alkali, total matter insoluble in alcohol, and sodium chloride shall not exceed 3 per cent.

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.5 per cent.

Matter insoluble in water shall not exceed 0.2 per cent.

Titer of the mixed fatty acids prepared from the soap must be not less than 39° C.

Color and odor must be as specified.

The percentage of matter volatile at 105° C. will be computed on the basis of the soap as received, but all other constituents will be calculated on the basis of material containing 10 per cent of volatile matter.

The material will be purchased by net weight, provided the matter volatile at 105° C. does not exceed 10 per cent. With deliveries containing more than 10 per cent but not exceeding 15 per cent of matter volatile at 105° C. settlement will be made on the basis of 10 per cent of matter volatile at 105° C.; that is, nine-tenths of a pound of nonvolatile matter will be considered 1 pound of soap.

Examples.—1. Yield 6 per cent of matter volatile at 105° C.; pay for net weight.

2. Yield 12 per cent of matter volatile at 105° C.; percentage of net weight to be paid for is calculated as follows:

$$(100-12) \times 10/9 = 97.8 \text{ per cent.}$$

2. SAMPLING.

(*a*) When Packed in Cans or Cartons.—One can or carton 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 can or carton 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 cans or cartons 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 cans or cartons tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped can or carton, record its weight and the date of weighing on the wrapper, place

the wrapped cans or cartons 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.

(*b*) When in Bulk.—A grab sample of not less than one-half pound shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain not less than 100 pounds each. In case of smaller containers a grab sample of not less than one-half pound shall be taken at random from each lot of containers totaling not to exceed 10,000 pounds. The total sample shall in all cases consist of not less than three grab portions 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. The inspector should rapidly mix the sample, place in an air-tight container, which shall be filled, seal, mark, accurately weigh, record its weight and date of weighing on the package, 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.—Rapidly disintegrate and mix the sample, if desired quarter down to about 1 pound, and weigh out all portions for analysis at once. Unused portions of the sample used for analysis shall be preserved in an air-tight container in a cool place.

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 or 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. (Time can be saved by having a layer of about 3 mm of ignited sand and a small stirring rod weighed with the dish and dissolving the sample in absolute alcohol, evaporating to dryness, breaking up the sample with the rod, adding more alcohol, again evaporating, and completing the drying in the oven as above.) 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 10 g sample with 200 c c 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, 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.

(2) *Free Alkali or Free Acid.*—Titrate the filtrate from 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 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.

(*d*) Chloride.—Dissolve 5 g of the sample in 300 c c of water, boiling if necessary to effect solution of all soluble matter. Add an excess of neutral chlorine-free magnesium nitrate solution (about 25 c c of a 20 per cent $Mg(NO_3)_2.6H_2O$ solution). Without cooling or filtering titrate with standard silver nitrate solution, using potassium chromate as indicator. Calculate the chloride as sodium chloride.

(*e*) Titer Test.—(1) *Preparation of Total Fatty Matter.*—Dissolve about 50 g of soap in 500 c c of hot water, add 100 c c of 30 per cent sulphuric acid, heat until the fatty matter collects in a clear layer, draw off the acid layer and wash the fatty matter free from sulphuric acid with hot water. Decant the fatty matter into a dry beaker, filter, using a hot-water funnel, or placing both funnel and receiving beaker in a water-jacketed oven, and dry for 20 minutes at the temperature of boiling water.

(2) *Determination.*—Cool the fatty acids, prepared as described, to about 50° C. and transfer about 25 c c to a tube of clear glass about 1 mm thick, 25 mm in diameter, and 100 mm long. Place the tube in a salt-mouth bottle about 70 mm in diameter

and 15 mm high (a 16-ounce salt-mouth bottle), fitted with a cork which is perforated so as to hold the tube rigidly when in position. Suspend a thermometer graduated to 0.1° C., so that it can be used as a stirrer and stir the mass slowly, first in one direction and then in the other, being careful not to touch the side of the tube, until the mercury remains stationary for 30 seconds. Then allow the thermometer to hang quietly, with the bulb in the center of the mass, and observe the rise of the mercury. The highest point to which it rises is recorded as "titer."

(*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 H_2SO_4 (specific gravity = 1.53) to this. A fugitive violet color indicates the presence of rosin.

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*) STANDARD SILVER NITRATE SOLUTION.—0.10 N, or about 17 g of silver nitrate dissolved in water and diluted to 1 liter. Standardized against chemically pure sodium chloride.

(*d*) POTASSIUM CHROMATE SOLUTION.—A 10 per cent solution of potassium chromate (K_2CrO_4) in water.

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

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