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Master
Specification
No. 244

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR MILLED TOILET SOAP

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 244

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 milled toilet soap.

CONTENTS

I. GRADE

Milled toilet soap shall be of one grade only, as hereinafter described.

II. MATERIAL AND WORKMANSHIP

Milled toilet soap shall be made of high-grade materials and shall be entirely satisfactory for the purpose intended.

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III. GENERAL REQUIREMENTS

The soap desired under this specification is a high grade, milled cake soap, as free as possible from water, either colored or uncolored, and unscented or perfumed in a manner indicated in the contract, thoroughly saponified, well compressed in firm, smooth cakes of a size and shape specified in the contract. It should lather freely when used with cold, soft water. Bidder shall state weight and number of cakes in each box.

IV. DETAIL REQUIREMENTS

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

1. 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.
2. The sum of free alkali, total matter insoluble in alcohol, and sodium chloride shall not exceed 1.5 per cent.
3. Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.1 per cent.
4. Matter insoluble in water shall not exceed 0.2 per cent.
5. Unsaponified saponifiable matter shall not exceed 0.3 per cent.
6. Rosin, sugar, and foreign matter shall not be present.
7. The color, odor, and character of the soap must be as specified by the purchaser.
8. 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 15 per cent of matter volatile at 105° C.

V. METHOD OF INSPECTION AND TESTING

1. SAMPLING

(*a*) No samples shall be submitted with bids.

(*b*) 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.

2. METHODS OF TESTING

(*a*) Preparation of Sample.—In case of samples that can be easily disintegrated and mixed, run the entire sample through a suitable chopper. When the sample is large each cake may be quartered, and one-quarter of each cake run through the chopper. With samples that can not be handled as above, 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.

When a determination shows nonconformity with the 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 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 the 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 200 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, 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 the above, using phenolphthalein as indicator, with standard acid or alkali

solution and calculate the alkalinity to sodium hydroxide or acidity to oleic acid.

(*d*) 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 the matter insoluble in water. The nature of this may be determined by further examination.

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

(*f*) Unsaponified Saponifiable Matter.—Weigh 5 g of the soap in a beaker and dissolve in about 100 cc of 50 per cent alcohol on the steam bath. If the sample has been found to contain free fatty acid, add just enough aqueous alkali to neutralize this. Evaporate off the bulk of the alcohol; take up with about 200 cc of hot water, and transfer to a separatory funnel of about 500 cc capacity, designated as No. 1. When cool, rinse out the beaker with about 50 cc of ether and add it to the soap solution. Shake thoroughly for one minute. By the addition of small amounts of alcohol (5 cc portions and the total not to exceed 25 cc) a clear and rapid separation of the aqueous and ethereal layers is effected. After each addition of alcohol the separatory funnel is not shaken, but merely given a whirling movement. Draw off the aqueous portion into another separatory funnel, designated as No. 2. Wash the ether solution with 10 cc portions of water until this water is no longer alkaline to phenolphthalein. Add all of these washings to funnel No. 2 and extract this solution with 20 cc portions of ether until the ether is absolutely colorless (three or four extractions should be sufficient). Combine these ether extracts in a third separatory funnel (No. 3) and wash with 10 cc portions of water until the water is no longer alkaline to phenolphthalein. Now add the ether in funnel No. 3 to that in funnel No. 1, a small amount of ether being used to rinse out funnel No. 3. Wash the ether solution with 20 cc of 10 per cent hydrochloric-acid solution and then successively with 20 cc portions of water until the water is no longer acid to methyl orange. Filter the ether solution through a dry filter paper into a weighed beaker or flask. Evaporate or distil off the ether on the steam bath, dry as under the determination of matter volatile at 105° C., and

weigh the residue. Then heat with alcohol and, when cool, neutralize with standard alkali, using phenolphthalein. Deduct any appreciable amount of fatty acid found by this titration from the weight of the residue. This residue consists of the unsaponifiable matter and any neutral fat that may have been present in the soap. Thoroughly saponify the residue with alcoholic alkali and repeat the above procedure. The residue obtained is unsaponifiable matter only, and its weight deducted from that of the residue before saponification gives the weight of unsaponified saponifiable matter.

(*g*) *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). A fugitive violet color indicates the presence of rosin.

(*h*) *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 two minutes with an equal volume of boiling Fehling's solution. The formation of red cuprous oxide indicates the presence of sugar.

3. REAGENTS

(*a*) Standard Sodium Hydroxide Solution.—0.25 N, or about 10 g, of sodium hydroxide dissolved in water and diluted to 1 liter. Standardize against Bureau of Standards benzoic acid.

(*b*) Standard Sulphuric Acid Solution.—0.5 N, or about 25.8 g, of concentrated sulphuric acid (specific gravity=1.84) diluted to 1 liter. Standardize against standard sodium hydroxide solution V, 3, (*a*).

(*c*) Standard Silver Nitrate Solution.—0.10 N, or about 17 g of silver nitrate dissolved in water and diluted to 1 liter. Standardize against pure sodium chloride.

(*d*) Potassium Chromate Solution.—Dissolve 10 g of chloride-free potassium chromate (K_2CrO_4) in 100 cc of water.

(*e*) Sulphuric Acid (sp. gr.=1.53).—Mix 62.5 cc of strong sulphuric acid (specific gravity=1.84) with 61.5 cc of water.

(*f*) Fehling's Solution.—(1) *Copper sulphate solution.*—Dissolve 34.64 g of copper sulphate ($CuSO_4.5H_2O$) in water and dilute to 500 cc.

(2) *Alkaline tartrate solution.*—Dissolve 173 g of Rochelle salts ($NaKC_4H_4O_6.4H_2O$) and 50 g of sodium hydroxide in water and

dilute to 500 cc. Mix equal volumes of (1) and (2) immediately before use.

VI. PACKING AND MARKING

No details.

VII. ADDITIONAL INFORMATION

Basis of Purchase.—The material will be purchased by net weight.

VIII. GENERAL SPECIFICATIONS

No details.

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