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UNITED STATES GOVERNMENT SPECIFICATION FOR LIQUID SOAP.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION No. 27.

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 clear solution of pure vegetable oil potash (or potash and soda) soap with or without glycerol or alcohol, suitably perfumed, and free from all foreign matter. It should quickly form a satisfactory lather and have no injurious effect and leave no objectionable odor on the skin. Bidder shall state number of gallons to the container.

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

The material must be a clear solution, free from objectionable odor, other than from coconut oil, and must quickly form a satisfactory lather.

Total anhydrous soap shall be not less than the equivalent of 15 per cent potash soap.

Total matter insoluble in alcohol shall not exceed 0.5 per cent. Free alkali calculated as potassium hydroxide (KOH) shall not exceed 0.05 per cent.

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Chloride calculated as potassium chloride (KCl) shall not exceed 0.3 per cent.

More than traces of sulphates and sugar shall not be present.

All constituents shall be calculated on the basis of the original sample.

The material will be purchased by volume in accordance with the contract agreement. A gallon of soap shall mean 231 cubic inches at 15.5° C.

2. SAMPLING.

A sample of not less than one-half pint shall be taken at random from not less than I per cent of the vendors' shipping containers, provided such containers contain not less than IO gallons each. In case of smaller containers a sample of not less than one-half pint shall be taken at random from each lot of containers totaling not to exceed I,000 gallons. The total sample shall in all cases consist of not less than three portions of one-half pint each taken at random from separate containers. Before drawing the sample from the container selected the contents of the container shall be thoroughly agitated. The inspector shall thoroughly mix the samples drawn, place in clean, dry cans or bottles, which shall be completely filled and securely stoppered with clean corks or caps, seal, mark, and send to the laboratory for test. 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.—No preparation of the sample, other than thorough mixing, is necessary unless it is received during very cold weather, when it should be allowed to stand at least one hour after it has warmed up to room temperature (20° to 30° C.) before it is noted whether it forms a satisfactory lather.

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

(b) Total Anhydrous Soap.—Dissolve 10 g of the sample in 100 cc of water in a 250 cc Erlenmeyer flask. When solution is complete, add dilute sulphuric acid in slight excess, insert a small funnel in the neck of the flask, and heat the flask at a temperature not exceeding 60° C. until the fatty acids separate as a clear layer. Transfer to a separatory funnel, draw off the acid layer into a second separatory funnel, and shake the acid aqueous liquid with two 20 cc portions of ethyl ether. Dissolve the fatty acids in the ether used for washing the aqueous liquid and shake with 10 cc portions of water until they are no longer acid to

methyl orange. Unite the water portions used for washing and shake with 20 cc of ether, wash this ether until the wash water is neutral to methyl orange. Unite the ether solutions (if necessary, filter, washing the paper with ether) in a suitable weighed vessel, add 100 cc of neutral alcohol free from carbon dioxide, add phenolphthalein and titrate to exact neurtality with standard sodium hydroxide solution. Evaporate off the alcohol, dry to constant weight as in the determination of matter volatile at 105° C., and calculate the percentage of soda soap. This soap naturally includes any mineral oil and neutral fat, which, if determined separately, must be deducted from the result to obtain the true soap. Calculate the combined sodium oxide (Na,O) and deduct from the weight of soda soap to give the anhydrides. If the original soap was potash soap, proper calculation must be made to reduce to potassium oxide (K2O) or the titration made directly with standard potassium hydroxide solution. In case the soap shows an excess of free acid, proper corrections must be made in calculating the combined alkali in the original soap. (A blank test should be made on the sodium or potassium hydroxide solution for neutral salts and the proper corrections made if neces-

- (c) Total Matter Insoluble in Alcohol. Free Alkali, or Free Acid.—(1) Matter Insoluble in Alcohol.—Digest hot a 10 g sample with 200 cc of freshly boiled neutral ethyl alcohol (94 per cent or higher). Filter through a counterpoised filter paper neutral to phenol-phthalein, 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 potassium hydroxide) or acidity to oleic acid.
- (d) Chloride.—Dissolve 10 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 (NO₃6H₂O solution). Without cooling or filtering titrate with standard silver nitrate solution, using potassium chromate as indicator. Calculate the chloride as potassium chloride.

- (e) Sulphate.—A qualitative test may be made as follows: Proceed as in the determination of alcohol insoluble until the insoluble matter has been thoroughly washed in a Gooch crucible or on a filter paper with hot alcohol. Dissolve this insoluble matter in hot water, acidify with hydrochloric acid, and evaporate to dryness. Take up with a small amount of hydrochloric acid and water, filter, and test for sulphate.
- (f) Sugar.—A qualitative test 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 solution. The formation of red cuprous oxide indicates the presence of sugar.

4. REAGENTS.

- (a) STANDARD SODIUM HYDROXIDE SOLUTION.—0.25N, 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.—0.5 N, or about 25.8 g strong sulphuric acid (specific gravity = 1.84) diluted with water to 1 liter. Standardized against standard sodium hydroxide solution (a).
- (c) STANDARD ALCOHOLIC POTASSIUM HYDROXIDE SOLUTION.—0.25 N or about 14 g of potassium hydroxide dissolved in neutral ethyl alcohol (94 per cent or higher) and diluted to 1 liter with alcohol. Standardized against Bureau of Standards benzoic acid.
- (d) STANDARD ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Same as (a) excepting that (94 per cent or higher) ethyl alcohol is used instead of water. Standardized against benzoic acid.
- (e) 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.
- (f) Potassium Chromate Solution.—A 10 per cent solution of potassium chromate in water.
- (g) Fehling Solution.—(1) Copper Sulphate Solution.—Dissolve $34.639~\rm g$ of copper sulphate (CuSO₄.5H₂O) 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.



