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UNITED STATES GOVERNMENT SPECIFICATION FOR AUTOMOBILE SOAP.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION No. 30.

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.

CONTENTS.

1. GENERAL.

The soap desired under this specification is a pure vegetable oil paste soap containing no free alkali or acid, relatively free from matter insoluble in alcohol, homogeneous, free from adulterants of any kind, and without objectionable odor. Bidder to state size and weight of unit.

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

Matter volatile at 105° C. shall not exceed 55 per cent. Deliveries which yield more than 55 per cent of volatile matter will be rejected without further test.

The sum of free alkali and total matter insoluble in alcohol shall not exceed 1 per cent.

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.2 per cent.

Free acid, calculated as oleic, shall not exceed 0.2 per cent.

Matter insoluble in water shall not exceed 0.2 per cent.

Unsaponified matter shall not exceed 4 per cent.

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Rosin shall not be present.

Odor must be as specified in contract.

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 50 per cent of volatile matter.

The material will be purchased by net weight.

2. SAMPLING.

(*a*) When Packed in Cans or Cartons of 5 Pounds or Less.—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 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, seal, mark, and send to laboratory for test.

(*b*) When Packed in Bulk.—Take at random a trier sample of not less than one-half pound from not less than 1 per cent of the vendors' shipping containers, provided such containers do not contain less than 50 pounds each. In case of smaller containers a trier sample 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 3 half-pound portions taken at random from separate containers. With very large lots where the sample drawn as above will amount to more than 10 pounds the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 10 pounds. The inspector shall promptly place the combined sample in a clean, dry, air and water tight container, which shall be filled, 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.—Mix thoroughly by kneading and quarter down to about 1 pound. Weigh out all portions for analysis promptly and preserve the remainder in an air-tight container in a cool place.

When a determination shows nonconformity with specifications, 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. (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 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 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.

(*d*) UNSAPONIFIED MATTER.—In a beaker on the steam bath dissolve 5 g of the soap in about 100 cc of 50 per cent alcohol. 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 aqeuous and ether layers is effected. After adding each alcohol portion 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 distill 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.

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

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