DEPARTMENT OF COMMERCE

CIRCULAR

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 62

SPECIFICATIONS FOR AND METHODS OF TESTING SOAPS

ISSUED DECEMBER 16, 1916

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PREFACE

This circular has been prepared by the Bureau of Standards with the cooperation of representatives of other branches of the Government service in the hope that it may lead to more uniform specifications and methods of testing soaps. It is not intended to discuss the manufacturing operations in soap making, but to consider only those matters which are of importance to purchasers and chemists examining purchases of soaps.

S. W. STRATTON,
Director.

SPECIFICATIONS FOR AND METHODS OF TESTING SOAPS

CONTENTS

I. GENERAL COMPOSITION OF SOAP

All metallic salts of the fatty acids are, strictly speaking, soaps; but the fatty-acid salts of the alkali metals are the only ones that are soluble in water, and therefore the only ones commonly used as cleansers. Soaps of some of the other metals are used for particular purposes, such as aluminium soap, which is used for thickening lubricating oils; iron and chromium soaps, which are used in dyeing and color printing; and lead and manganese soaps, which are used as "driers" in paints and varnishes. These, however, are used for purposes so radically different from those which call for the detergent soaps that they will not be considered in this circular.

In addition to alkali salts of fatty acids, all soaps also contain some water and small amounts of impurities and by-products of manufacture. For various purposes, legitimate and otherwise, certain other substances frequently enter into the composition of commercial soaps. Among these substances may be mentioned rosin (colophony), as a partial substitute for fats; sodium carbonate, borax, and other salts, for hardening and rendering soap more detergent; sand, volcanic ash, infusorial earth, pumice stone, and like substances, intended to aid mechanically in the process of cleaning; glycerol, for increasing the emollient properties; sugar, alcohol, and glycerol, for increasing transparency in solid soaps and for preventing clouding and foaming in liquid soaps; colors and perfumes of many varieties; cheapening or filling materials, such as mineral oils and waxes, water glass (sodium silicate), talc, starch, etc. Under special conditions such substances as sulphur, carbolic acid, mercury salts, etc., are added to medicinal soaps; these last, however, are of such a special nature that they will not be considered.

Animal and vegetable fats and oils are mixed esters, or more specifically mixed glycerides of the higher fatty acids, and it is from these materials that soaps in general are made. The difference between a fat and an oil is mainly physical; the term "fat" is generally applied to those glycerides which are solid at ordinary temperatures, and "oil" to those which are liquid under similar conditions. For the sake of simplicity we will use the term "fat" to cover both classes. Each fat as found in nature contains the glycerides of several different fatty acids, the principal ones of which are named, respectively, stearic, palmitic, and oleic acids. These occur in varying quantities in practically all fats, the solid fats containing a large proportion of stearic-acid glyceride (stearin), while the liquid fats contain a large proportion of oleic-acid glyceride (olein). There are a large number of fatty-acid glycerides besides the three mentioned above which occur in certain fats, such as lauric-acid glyceride (laurin) in coconut and palm-kernel oils, myristic-acid glyceride (myristin) in palm-kernel oil, palmitic-acid glyceride (palmitin) in palm oil, and tallow, linoleic-acid glyceride (linolein) in linseed oil, etc.

The numerous animal and vegetable fats, such as tallow, lard, olive oil, palm oil, coconut oil, cottonseed oil, etc., are neutral substances which may be decomposed by the aid of superheated steam or other suitable means into two distinct separate portions—namely, a mixture of "fatty acids" on one hand, and "glycerol" on the other.

Saponification is the term applied to this splitting of an ester by the action of water forming an alcohol and a fatty acid (glycerol is the alcohol in fats). This may be expressed as follows:

Stearin and water give glycerol and stearic acid.

$$C_3H_5(O.C_{18}H_{35}O)_3 + 3H_2O = C_3H_5(OH)_3 + 3C_{18}H_{36}O_2.$$

The stearic acid can be neutralized by alkali, forming soap as follows:

Stearic acid — Sodium hydroxide — Water — Sodium stearate (soap)

$$C_{18}H_{36}O_2 + NaOH = H_2O + C_{18}H_{35}O_2Na.$$

It is not necessary to split the fats into glycerol and fatty acid previous to neutralizing the acid with alkali; in fact, one of the most important and the oldest method in use is to treat the fat itself with alkali, the reaction being represented by the equation—

Stearin — Sodium hydroxide — Glycerol — Sodium stearate

$$C_3H_5(O.C_{18}H_{35}O)_3 + 3NaOH = C_3H_5(OH)_3 + 3C_{18}H_{35}O_2Na.$$

The same types of reactions occur with the other fats, but it must be remembered that mineral and essential oils are of an entirely different nature from the true fats.

Rosin (colophony) is an acid substance, and while its exact nature is not as well understood as that of the fats, it may be tentatively considered to be mainly abietic acid ($C_{20}H_{30}O_2$), which reacts with alkali according to the equation—

$$C_{20}H_{30}O_2 + NaOH = H_2O + C_{20}H_{29}O_2Na.$$

The other substances mentioned above as occurring in commercial soaps do not constitute a part of the true soaps, but are simply admixtures.

Hard soaps are generally soda soaps, while the potash soaps are soft and are more easily soluble than the soda soaps. The character of the fatty materials used also influences the hardness and solubility of the resulting soaps, the hard fats, such as tallow, making harder soaps generally than the more fluid fats; rosin makes soaps softer, and it can therefore be used best in connection with tallow and other hard fats. Sodium carbonate tends to harden rosin soaps, and this substance is frequently added to soaps containing rosin.

The oldest and probably still the best method of soap making is the boiling process, in which the fats are boiled with alkali and the resulting soap is separated from most of the liquid, which contains the glycerol and impurities. The soap maker also uses two other processes known as "cold" and "half boiling." In the cold process the melted fat is mixed with strong alkali and no heat except that of the chemical reaction is required. The half-boiling process is similar to the cold process, except that the ingredients are mixed hot. In both of these processes no separation of soap is effected and the product contains the glycerol and impurities; also the saponification is frequently not completed.

The user of soap should bear in mind that there are many varieties of soaps, and what is satisfactory for one purpose may be unsatisfactory or too expensive for another.

II. VARIETIES OF SOAPS

1. TOILET SOAPS

Toilet soaps should be entirely neutral, since excess alkali is injurious to the skin. Fillers such as sodium carbonate and sodium silicate, having a similar effect, should also be absent. Free-

lathering soap is generally desirable; and since a tallow soap lathers slowly and coconut-oil soap lathers very freely, some coconut oil is frequently added. This oil has a tendency to injure the skin and its odor is also objectionable; hence it is not generally used in large amounts. Some potash is frequently used in toilet soaps to produce freer lathering.

(*a*) *Milled toilet soaps* are prepared by grinding any good soap and compressing into cakes. More delicate perfumes can be used with this class of soaps, since the perfume is mixed in the process of milling, than with ordinary soaps, in which the perfume is added before the soap is dried. (See recommended specification, p. 10.)

(*b*) *Floating soaps* contain entangled air in very fine bubbles, incorporated while the soap is still hot. These air bubbles are so small as to be almost invisible and so numerous that they not only make the soap lighter than water but also largely increase the surface of the soap exposed to water when used, and therefore render it more quickly soluble than the same soap would be without the bubbles. (See recommended specification, p. 12.)

(*c*) *Castile soap*, otherwise known as Marseilles or Venetian soap, is prepared from low-grade olive oils.

(*d*) *Transparent soaps* were originally made by dissolving soap in alcohol, filtering and evaporating the alcohol. The transparency formerly was considered an indication of freedom from impurities, but the same effect can be produced in other ways and the transparency is actually no indication whatever of purity or quality.

(*e*) *Liquid soaps* are water solutions generally of a neutral coconut-oil potash soap, containing glycerol, sugar, or alcohol added to prevent cloudiness and foaming in the container. The glycerol is probably an unobjectionable addition, since it has emollient properties, but sugar can have no beneficial action on the soap itself and may be objected to on account of its tendency to leave the hands sticky. Alcohol is seldom used. (See recommended specification, p. 13.)

(*f*) *Shaving soaps* must possess not only the properties of first-class toilet soaps, but must furnish a very rich lather, which will remain on the face for some time without drying. This lather should soften the beard without injuring the skin. These soaps should have no unpleasant odor and little or no perfume. The fat used in shaving soaps generally contains some coconut oil and

the alkali is generally a mixture of soda and potash. Glycerol is also generally present.

It has not been considered advisable to recommend specifications for castile, transparent, or shaving soaps.

2. SALT-WATER SOAP

Soap for use on ocean-going vessels is soda soap, made entirely of coconut oil, with frequently a small amount of sodium carbonate as a filler. It is less easily precipitated by salt water than soaps made from other fats, and generally contains a large amount of water, frequently over 50 per cent. (See recommended specification, p. 14.)

3. LAUNDRY OR COMMON SOAP

(*a*) *Laundry cake soap* is probably used in larger quantities than any other. Ordinary laundry soap is generally made of soda and tallow, with some rosin; but the tallow is frequently replaced partly or wholly by grease, cottonseed oil, oleic acid, etc. The basis of this soap is the fat, the rosin being used chiefly because it is cheaper, though partly because the soaps containing rosin are more quickly soluble than soaps made entirely from fats. The amount of rosin, when rosin is allowed at all, should not be excessive (not more than one-third the amount of fat used), since large amounts make the soap sticky and too soluble and tend to leave a sticky deposit on the fabric washed with it. The fatty soap is probably the valuable constituent for washing. Since the addition of rosin softens the soap, sodium carbonate is frequently added to rosin soaps to make them harder. In washing, this sodium carbonate aids the cleansing effects of the soap by its own detergent properties and is often a desirable addition where hard waters are used, since it acts as a softening agent. Borax is sometimes used for practically the same purpose as sodium carbonate. Sodium silicate or water glass is also used as a filler, but this is practically an adulterant, though it is claimed to have some detergent and water-softening properties. It should be borne in mind, however, that all these fillers are cheaper than soap, and filled soaps, even when suitable for the purpose intended, should be cheap.

Some laundry soaps are free from rosin and fillers and while they are slower in dissolving, where soft water is used, they are probably the best. Laundry soaps differ from toilet soaps in many particulars, for example, generally they need not be

entirely neutral, since a somewhat alkaline soap is more effective, especially with hard water; nor are they required to produce so rich a lather. (See recommended specifications for two types of laundry soap, pp. 15 and 17.)

(*b*) *Chip soaps* are hard soaps, generally made from tallow and soda, and should be of high purity and very dry. (See recommended specification, p. 18.)

4. CLEANSING POWDERS

The terms "soap powder" and "scouring powder" are used very loosely. A soap powder should be entirely soluble in water and should consist of powdered soap and sodium carbonate; the major portion often is the latter substance.

A scouring powder should consist of a mixture of soap powder and an insoluble abrasive, but very frequently a soluble powder is spoken of as scouring powder, and one containing abrasive as soap powder. This is a misuse of the respective terms and should be avoided.

5. SCOURING CAKES

Scouring cakes consist largely of abrasive material, such as sand, powdered pumice, volcanic ash, etc., with a binder of soap, and frequently considerable sodium carbonate.

Owing to the great variation in the uses to which these materials are applied, no general recommended specifications for cleaning powders and scouring cakes have as yet been prepared.

III. SPECIFICATIONS RECOMMENDED

Large consumers, such as the various branches of the Government service, municipalities, etc., have for many years purchased various kinds of soap under definite specifications, but an examination of such existing specifications shows a great variety of requirements for the same kind of soap. It would be of distinct advantage to both manufacturers and consumers if one specification for any particular type of soap could be generally used by a large number of consumers. Such a specification should secure a soap suitable for the intended use, but as far as possible admit material of regular commercial makes. It should allow the greatest freedom in the selection of stock that is consistent with quality, so that the manufacturer can take advantage of both varying market conditions and advances in technology of fats and oils and

thus prevent, as far as possible, excessive prices due to temporary scarcity in any specific raw material. Methods of sampling and testing should be clearly defined and be made a part of the specification.

In preparing the recommended specifications conferences were held first with representatives of many branches of the Government service, for the needs of these departments were considered as fairly representing those of the general public. After much discussion and correspondence with these representatives a tentative set of specifications was submitted to a large number of manufacturers for criticism and suggestion. Due consideration of the criticisms of manufacturers has been given in preparing the specifications now recommended.

It should be remarked that the percentage of water to be allowed in soap is the most fruitful source of disagreement between purchaser and manufacturer. Analyses of samples from a large number of deliveries of laundry and white floating soaps indicate that as delivered to the Government departments in Washington the water content is generally about 20 per cent. However, it is frequently somewhat above this figure, but seldom more than 28 per cent. Producers claim that as manufactured soap frequently contains from 30 to 33 per cent of water, and from a consideration of the weight of cakes tested and their probable weight as manufactured this claim seems to be sustained. A large amount of water causes the soap to be used up very rapidly. It is believed fair to all parties to make payment on a basis of material containing a definite amount of water, to allow water somewhat above the limit specified with or without penalty, but to reject when this is in excess of a maximum limit.

The following proposed specifications are presented in a form which can be used by purchasers in contracting for various classes of soap. In each case the methods of sampling and testing would be those defined in Section IV of this circular.

1. MILLED TOILET SOAP

The soap desired under this specification is a high-grade milled cake soap at least as good in every respect as one made exclusively from soda and a mixture of about 87 per cent best-grade tallow and 13 per cent pure coconut oil; as free as possible from water; uncolored; either unscented or perfumed in a manner indicated in the contract for the same; 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 water.

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.)

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.1 per cent.

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall not exceed 0.3 per cent. Not more than one-half of the alkali as alkaline salts shall be as sodium silicate. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

Sulphate, calculated as sodium sulphate (Na_2SO_4), shall not exceed 0.1 per cent.

Chloride, calculated as sodium chloride (NaCl), shall not exceed 0.3 per cent.

Matter insoluble in water shall not exceed 0.1 per cent.

Unsaponified saponifiable matter shall not exceed 0.1 per cent.

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

Titer and acid number of the mixed fatty acids prepared from the soap must be respectively not less than 37° C and not less than 203 nor more than 212.

Odor and character of cake 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 deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[1]

The material will be purchased by weight. In calculating the weight of soap to be paid for, nine-tenths of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap, provided the volatile matter does not exceed 10 per cent. On deliveries

[1] When it is desired to copy or reprint separately this specification complete, this paragraph should read: "The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1(*a*), 2(*a*), and 3(*a*), (*b*), (*d*), (*e*), (*f*), (*g*), (*h*), (*i*), (*k*), (*l*), (*m*), and (*o*).

containing more than 10 per cent, but not more than 15 per cent volatile matter, a deduction of 2 per cent from the weight of soap to be paid for shall be made for each 1 per cent of volatile matter in excess of 10 per cent. (See example *c*.)

Examples:

(*a*) Yield 5 per cent matter volatile at 105° C $(100-5)\times\frac{10}{9}=105.5$. Therefore pay for 105½ per cent of delivered weight.

(*b*) Yield 10 per cent matter volatile at 105° C $(100-10)\times\frac{10}{9}=100$. Therefore pay for 100 per cent of delivered weight.

(*c*) Yield 12 per cent matter volatile at 105° C $\left[(100-12)\times\frac{10}{9}\right]-[(12-10)\times 2]=93.7$. Therefore pay for 93.7 per cent of delivered weight.

2. WHITE FLOATING SOAP

The soap desired under this specification is a high-grade cake soap at least as good in every respect as one made from soda and a mixture of high-grade tallow with 25 per cent to 30 per cent of coconut oil; containing not more than 20 per cent water; of good light color; thoroughly saponified; so prepared as to float on water; in cakes of a size and shape specified in the contract.

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

Matter volatile at 105° C shall not exceed 34 per cent. (Deliveries which yield more than 34 per cent of volatile matter will be rejected without further test.)

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.1 per cent.

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall not exceed 0.5 per cent. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

Chloride, calculated as sodium chloride (NaCl), shall not exceed 1 per cent.

Matter insoluble in water shall not exceed 0.1 per cent.

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

Titer and *acid number* of the mixed fatty acids prepared from the soap must be respectively not less than 35° C and not less than 212.

Odor and *character of cake* 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 28 per cent of volatile matter.

The deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[2]

The material will be purchased by weight. In calculating the weight of soap to be paid for, four-fifths of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap, provided the volatile matter does not exceed 28 per cent. On deliveries containing more than 28 per cent but not more than 34 per cent volatile, a deduction of 2 per cent from the weight of soap to be paid for shall be made for each 1 per cent of volatile in excess of 28 per cent. (See example *c*.)

Examples:

(*a*) Yield 28 per cent matter volatile at 105° C $(100-28)\times\frac{5}{4}=90$.

Therefore, pay for 90 per cent of delivered weight.

(*b*) Yield 20 per cent matter volatile at 105° C $(100-20)\times\frac{5}{4}=100$.

Therefore, pay for 100 per cent of delivered weight.

(*c*) Yield 30 per cent matter volatile at 105° C $\left[(100-30)\times\frac{5}{4}\right]-[(30-28)\times 2]=83.5$. Therefore, pay for 83.5 per cent of delivered weight.

3. LIQUID SOAP

The soap desired under this specification is a clear solution of pure vegetable oil-potash (or potash and soda) soap, with or without alcohol or glycerol, suitably perfumed and free from all foreign matter. It should quickly form a satisfactory lather when applied to the hands and have no injurious effect and leave no objectionable odor on the skin.

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[2] When it is desired to copy or reprint separately this specification complete, this paragraph should read:

"The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1 (*a*), 2 (*a*), and 3 (*a*), (*b*), (*d*), (*g*), (*i*), (*k*), (*l*), (*m*), and (*o*).

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

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

Total soap shall be not less than 20 per cent.

Free alkali, calculated as potassium hydroxide (KOH), shall not exceed 0.05 per cent.

Alkali as alkaline salts, calculated as potassium carbonate (K_2CO_3), shall not exceed 0.3 per cent. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

Sulphates and *sugar* shall not be present.

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

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

The deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[3]

The material will be purchased by weight or by volume delivered in accordance with the contract agreement.

4. SALT-WATER SOAP

The soap desired under this specification is a well-made soap, manufactured, without removing glycerol, exclusively from pure coconut oil and the necessary alkalies; entirely soluble in both sea water and fresh water; free from "fillers" and "makeweights" of any kind; and suitable to make a good lather.

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.)

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

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[3] When it is desired to copy or reprint separately this specification complete, this paragraph should read: "The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1(*b*), 2(*b*), and 3(*c*), (*d*), (*e*), (*f*), (*n*), (*o*), and (*q*).

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall be not less than 2 per cent nor more than 3 per cent. Not more than one-quarter of the alkali as alkaline salts shall be as sodium silicate. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

Chloride, calculated as sodium chloride (NaCl), shall not be less than 2.5 per cent and not more than 3.5 per cent.

Mineral matter, including insoluble in water, sodium sulphate, and other nonvolatile matter not otherwise specified, shall not exceed 0.5 per cent.

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

Acid number of the mixed fatty acids prepared from the soap must be not less than 256.

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 55 per cent of volatile matter.

The deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[4]

The material will be purchased by weight. In calculating the weight of soap to be paid for, one-half of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap.

5. SPECIAL-GRADE LAUNDRY SOAP

The soap desired under this specification is a high-grade, well-made, uniformly mixed laundry soap, made from soda and fats of high melting point; free from, or with only a relatively small proportion of, rosin; low in alkaline salts; free from objectionable odor, makeweights, or fillers of any kind; and containing not more than 20 per cent of water; in cakes of size and shape specified in contract; and suitable for use with soft water for general cleaning and laundry purposes, where the presence of sodium carbonate or sodium silicate may be objectionable.

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[4] When it is desired to copy or reprint separately this specification complete, this paragraph should read:

"The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1(*a*), 2(*a*), and 3(*a*), (*b*), (*d*), (*e*), (*f*), (*g*), (*i*), (*l*), (*m*), and (*o*).

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

Matter volatile at 105° C shall not exceed 34 per cent. (Deliveries which yield more than 34 per cent volatile matter will be rejected without further test.)

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

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall not exceed 1 per cent. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

The sum of the *chloride* and *sulphate*, calculated as sodium chloride (NaCl) and sodium sulphate (Na_2SO_4), shall not exceed 1 per cent.

Matter insoluble in water shall not exceed 0.1 per cent.

Rosin shall not exceed 15 per cent.

Titer of the mixed fatty and rosin acids prepared from soap must be not less than 35° C.

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 28 per cent of volatile matter.

The deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[5]

The material will be purchased by weight. In calculating the weight of soap to be paid for, four-fifths of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap, provided the volatile matter does not exceed 28 per cent. On deliveries containing more than 28 per cent but not more than 34 per cent volatile, a deduction of 2 per cent from the weight of soap to be paid for shall be made for each 1 per cent of volatile matter in excess of 28 per cent. (See example *c*.)

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[5] When it is desired to copy or reprint separately this specification complete this paragraph should read:

"The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification; 1(*a*), 2(*a*), and 3(*a*), (*b*), (*d*), (*e*), (*f*), (*g*), (*j*), (*k*), (*m*), (*o*), and (*p*).

Examples:

(*a*) Yield 28 per cent matter volatile at 105° C (100–28) $\times\frac{5}{4}=90$. Therefore pay for 90 per cent of delivered weight.

(*b*) Yield 20 per cent matter volatile at 105° C (100–20) $\times\frac{5}{4}=100$. Therefore pay for 100 per cent of delivered weight.

(*c*) Yield 30 per cent matter volatile at 105° C $\left[(100-30)\times\frac{5}{4}\right]-[(30-28)\times 2]=83.5$. Therefore pay for 83.5 per cent of delivered weight.

6. ORDINARY-GRADE LAUNDRY SOAP

The soap desired under this specification is a well-made, uniformly mixed laundry or common soap, made from soda and fats of high melting point; with no excessive proportion of rosin and moderate amount of alkaline salts; free from objectionable odor or make-weights; containing not more than 20 per cent of water; in cakes of a size and shape specified in contract; and suitable for use with moderately hard water for general cleaning and laundry purposes where the presence of rosin is not objectionable, and where the presence of sodium carbonate is either not objectionable or is actually advantageous.

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

Matter volatile at 105° C shall not exceed 34 per cent. (Deliveries which yield more than 34 per cent volatile matter will be rejected without further test.)

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

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall be not less than 2 per cent nor more than 6 per cent. Not more than one-half of the alkali as alkaline salts shall be in the form of sodium silicate. (The term "alkaline salts" as used here includes carbonates, borates, and silicates.)

Chloride, calculated as sodium chloride (NaCl), shall not exceed 1 per cent.

Sulphate, calculated as sodium sulphate (Na_2SO_4) shall not exceed 1 per cent.

Matter insoluble in water shall not exceed 0.5 per cent.

Rosin shall not exceed 25 per cent.

Titer of the mixed fatty and rosin acids prepared from the soap must be not less than 33° C.

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 28 per cent of volatile matter.

The deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[6]

The material will be purchased by weight. In calculating the weight of soap to be paid for, four-fifths of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap, provided the volatile does not exceed 28 per cent. On deliveries containing more than 28 per cent but not more than 34 per cent volatile, a deduction of 2 per cent from the weight of soap to be paid for shall be made for each 1 per cent of volatile in excess of 28 per cent. (See example *c*.)

Examples:

(*a*) Yield 28 per cent matter volatile at 105° C $(100-28)\times\frac{5}{4}=90$.

Therefore pay for 90 per cent of delivered weight.

(*b*) Yield 20 per cent matter volatile at 105° C $(100-20)\times\frac{5}{4}=100$.

Therefore pay for 100 per cent of delivered weight.

(*c*) Yield 30 per cent matter volatile at 105° C $\left[(100-30)\times\frac{5}{4}\right]-[(30-28)\times 2]=83.5$. Therefore pay for 83.5 per cent of delivered weight.

7. CHIP SOAP

The soap desired under this specification is a soap in chip form, made from soda and fats of high melting point, 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

[6] When it is desired to copy or reprint separately this specification complete, this paragraph should read: "The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1 (*a*), 2 (*a*), and 3 (*a*), (*b*), (*d*), (*e*), (*f*), (*g*), (*j*), (*k*), (*m*), (*o*), and (*p*).

suitable for high-grade laundry work with soft water when the presence of alkaline salts is objectionable.

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.)

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

Alkali as alkaline salts, calculated as sodium carbonate (Na_2CO_3), shall not exceed 0.5 per cent.

Chloride, calculated as sodium chloride (NaCl), shall not exceed 0.5 per cent.

Matter insoluble in water shall not exceed 0.1 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 deliveries made under this specification will, in general, be sampled and examined by the methods given on pages 20 to 25, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications.[7]

The material will be purchased by weight. In calculating the weight of soap to be paid for, nine-tenths of a pound of matter *not* volatile at 105° C shall be considered 1 pound of soap, provided the volatile matter does not exceed 10 per cent. On deliveries containing more than 10 per cent but not more than 15 per cent volatile matter a deduction of 2 per cent from the weight of soap to be paid for shall be made for each 1 per cent of volatile matter in excess of 10 per cent. (See example *c*.)

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[7] When it is desired to copy or reprint separately this specification complete, this paragraph should read: "The deliveries made under this specification will, in general, be sampled and examined by the following methods, but the testing laboratory reserves the right to use any other tests which may be deemed advisable to ascertain whether or not a particular sample complies with the specifications."

The following paragraphs from pages 20 to 25 should then be also copied or printed as a part of the specification: 1 (*c*), 2 (*c*), and 3 (*a*), (*b*), (*d*), (*g*), (*j*), (*k*), (*m*), and (*o*).

Examples:

(*a*) Yield 5 per cent matter volatile at 105° C $(100-5)\times\frac{10}{9}=105.5$. Therefore pay for 105½ per cent of delivered weight.

(*b*) Yield 10 per cent matter volatile at 105° C $(100-10)\times\frac{10}{9}=100$. Therefore pay for 100 per cent of delivered weight.

(*c*) Yield 12 per cent matter volatile at 105 °C $\left[(100-12)\times\frac{10}{9}\right]-[(12-10)\times 2]=93.7$. Therefore pay for 93.7 per cent of delivered weight.

IV. METHODS OF SAMPLING AND ANALYSIS

1. SAMPLING DELIVERIES

(*a*) *Cake Soap.*—One cake shall be taken at random from each 1000 cakes to constitute the test sample, except when a sample so drawn shall weigh less than 1 pound and be less than three cakes, in which case additional cakes, to make a sample of not less than 1 pound and not less than three cakes shall be taken. The sample cakes shall be at once placed in an air-tight container, properly labeled, and sent to the laboratory for test.

(*b*) *Liquid Soap.*—After thorough mixing of the contents, 1 quart shall be drawn from one container taken at random from each delivery of 100 gallons or less of the material. If a larger volume of the material is delivered at one time, 1 quart shall be taken to constitute a sample for each 100 gallons. Each sample shall be at once placed in a quart glass bottle which has previously been thoroughly cleaned and dried, securely stoppered with a new cork, properly labeled, and sent to the laboratory for test. If desired the entire contents of one small container selected at random from each delivery may be sent as the sample.

(*c*) *Chip Soap.*—Not less than 1 pound taken at random from each 1000 pounds or less of any shipment shall constitute a sample. The sample shall be at once placed in a clean, dry, air-tight container, which must be completely filled with the sample, properly labeled, and sent to the laboratory for test.

2. PREPARATION OF LABORATORY SAMPLE

(*a*) *Cake Soap.*—Weigh each cake, set aside in an air-tight container one or two cakes of about medium weight, with record of weights as received, for making check analysis if necessary.

Select another cake of about medium weight, cut in two in a diagonal direction, and cut from these fresh surfaces, in thin layers, a sufficient quantity for all determinations, taking care to cut entirely across so as to get a fair proportion of the outer and inner parts. Preserve this sample in a tightly stoppered bottle and weigh the different portions required as soon as possible. Instead of preparing the sample by cutting, one or more cakes may be run three times, rapidly, through a meat chopper.

(*b*) *Liquid Soap.*—No preparation of the sample 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 noting whether the material is clear, free from objectionable odor, and whether it forms a satisfactory lather.

(*c*) *Chip Soaps.*—Rapidly mix the sample and weigh out all the portions for analysis, preserving the remainder in an air-tight container.

3. METHODS OF ANALYSIS

The following methods are to be used in examining soap purchased under any of the specifications recommended in this circular:

(*a*) *Matter Volatile at 105° C.*—Place 2 grams of the sample in a weighed porcelain dish 3 inches in diameter, spreading the sample as evenly as possible over the bottom of the dish; dry for one hour at 60° C, add 50 cc of absolute alcohol, evaporate, dry for one hour at 60° C, and then for two hours in an oven at 105 ° C, cool in a desiccator, and weigh. Calculate percentage loss.

(*b*) *Free Alkali or Acid (Method for All Except Liquid Soaps).*—Dissolve 5 grams of the sample in about 200 cc of freshly boiled, neutral, 95 per cent alcohol, heating to complete the solution. Filter through a weighed Gooch crucible (if the sample is known to contain less than the specified amount of matter insoluble in water, a filter paper may be used), wash with hot, freshly boiled neutral alcohol. Unite filtrate and washings, cool, add phenolphthalein, and titrate with standard acid or alkali. Calculate the percentage of free alkali as NaOH or free acid as oleic acid.

(*c*) *Free Alkali or Acid (Method for Liquid Soaps).*—Dissolve 10 grams of the sample in about 200 cc of freshly boiled, neutral absolute alcohol. Filter and wash the precipitate with freshly boiled, neutral alcohol. Unite the filtrate and washings, add phenolphthalein, and titrate with standard acid or alkali.

Calculate the percentage of free alkali as KOH or free acid as oleic acid.

(*d*) *Alkali as Alkaline Salts.*—Extract with hot water the matter insoluble in alcohol which is separated during the determination of free alkali or acid, add methyl orange to the water solution, and titrate with standard acid (HCl). Calculate the percentage of alkaline salts as Na_2CO_3 (or as K_2CO_3 in the case of liquid soaps). (It is understood that the "alkali as alkaline salts" as thus determined includes all alkali in the form of carbonate, borate, and silicate.)

(*e*) *Silicate.*—Add an excess of hydrochloric acid to the solution remaining after titration of alkaline salts, evaporate to dryness, take up with HCl, and determine silica in the regular way. Calculate the equivalent Na_2CO_3 assuming the silica to be in the form $Na_2Si_4O_9$.

(*f*) *Sulphate.*—Determine the sulphate in the filtrate from the silica by precipitation with barium chloride and weighing as barium sulphate in the ordinary manner; calculate as sodium sulphate (Na_2SO_4), except for liquid soaps, which are calculated as K_2SO_4.

(*g*) *Matter Insoluble in Water.*—Dry the Gooch crucible used in the determination of alkali as alkaline salts for two hours in an oven heated to 105° C, weigh, and calculate the percentage of total insoluble matter.

(*h*) *Unsaponified Saponifiable Matter.*—Dissolve 5 grams of soap in about 100 cc of 50 per cent alcohol; if any free fatty acids are present, add just enough standard alkali to neutralize them and wash into a separate funnel with 50 per cent alcohol. Extract with 100 cc ether. Wash the ether with water, evaporate, and weigh unsaponified matter. Add 25 cc alcoholic potash or soda, boil under a reflux condenser for one hour, evaporate to dryness, dissolve in water, transfer with ether and water to a separatory funnel, wash the ether free from alkali, transfer to a weighed dish, evaporate the ether, dry, and weigh. This weight gives the unsaponifiable matter, which is subtracted from the unsaponified matter. To the remainder add the weight of free fatty acid previously determined; the sum is the total unsaponified saponifiable matter.

(*i*) *Preparation of Total Fatty Acids (Method for Milled-Toilet, White-Floating, or Salt-Water Soaps).*—Dissolve about 50 grams of the soap in 300 cc of hot water, transfer to a separatory funnel, add 150 cc of approximately 2N H_2SO_4, cool somewhat, add 120 cc of

ether, shake, draw off the acid layer, and wash the ether layer free from acid with a strong salt (NaCl) solution. Then draw off the aqueous layer as completely as possible, transfer the ether layer to a flask (it is not necessary to transfer quantitatively), add 20 to 30 grams of anhydrous sodium sulphate (Na_2SO_4), stopper the flask, shake, and let stand at a temperature below 25° C until the ethereal liquid becomes perfectly clear, showing that all water has been taken up by the sodium sulphate. Filter through a dry paper into another Erlenmeyer flask, and completely evaporate off the ether by passing through the flask a current of dry air and heating the flask to a temperature not above 50° C.

(*j*) *Preparation of Fatty and Rosin Acids* (*Method for Ordinary or Special Grade Laundry or Chip Soaps*).—Dissolve about 50 grams of the soap in 500 cc of hot water, add 100 cc 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.

(*k*) *Titer Test.*—Cool the fatty acids, prepared as described, to about 50° C and transfer about 25 cc 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 150 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 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 the "titer."

(*l*) *Acid Number of Fatty Acids.*—Place from 2 to 5 grams of the fatty acids, prepared as described, in a 200-cc Erlenmeyer flask, add 50 cc of neutral alcohol free from carbon dioxide, warm to dissolve the fatty matter, add phenolphthalein, titrate with standard alcoholic alkali, and calculate the acid number (expressed as milligrams KOH per 1 gram total fatty matter).

(*m*) *Total Alkali* (*Method for all Except Liquid Soaps*).—Dissolve 5 grams of the soap in 100 to 150 cc of hot water, add 40 cc N/2 H_2SO_4, heat on a water bath until the fatty acids have collected in a clear layer, cool by placing the beaker containing the

mixture in cold water, break the cake of fatty acids, and filter the aqueous liquid, leaving as much of the fatty acids as possible in the beaker; thoroughly wash the beaker, fatty acids, and filter paper with water. Unite the filtrate and washings, add methyl orange, and titrate the excess acid with N/5 NaOH. Calculate the total alkali as Na_2O.

(*n*) *Total Alkali* (*Method for Liquid Soaps*).—Warm together all of the acid aqueous liquids from the determination of total soap to drive off dissolved ether, add methyl orange and titrate the excess acid with N/5 NaOH. Calculate the alkali as per cent K_2O in original sample.

(*o*) *Chloride.*—Add neutral potassium chromate (K_2CrO_4) solution to the neutral solution from the titration of total alkali, titrate with N/10 $AgNO_3$. Calculate the chloride as sodium chloride (NaCl) (or as KCl in the case of liquid soaps).

(*p*) *Rosin.*—A qualitative test for rosin may be made as follows: After decomposing a solution of the soap and separating the fatty acids a small quantity of the latter is heated with acetic anhydride. When cool a few drops are placed on a spot plate and when a drop of H_2SO_4 (sp. gr. = 1.53) is added to this a fugitive violet color indicates the presence of rosin. For a quantitative determination proceed as follows: Dissolve 5 grams of the sample in 100 to 200 cc of hot water, add 40 cc N/2 H_2SO_4, heat until the fatty acids, etc., collect in a clear layer, cool in ice water, remove the layer of fatty acids, etc. Wash the fatty acids with ice water, transfer to a 200 cc Erlenmeyer flask, dry for one hour at 105° C, cool, dissolve in 20 cc of absolute alcohol, add 10 cc of a solution of one volume of strong sulphuric acid and four volumes of absolute alcohol, and boil for 4 minutes under a reflux condenser. Add to the liquid about five times its volume of 7 to 10 per cent solution of NaCl and extract with ether. Shake out the aqueous portion two or three times with ether. Unite the ether solutions and wash with the sodium chloride solution until the washings are neutral. Titrate the rosin acids with standard alcoholic sodium hydroxide solution (1 cc normal alkali = 0.346 g. rosin). Calculate percentage of rosin.

(*q*) *Total Soap* (*Method for Liquid Soaps*).—Dissolve 10 grams of the soap in 100 cc water, add 40 cc N/2 H_2SO_4, heat until a clear layer of fatty acids separates, transfer to a separatory funnel, draw off the acid layer, and shake the acid aqueous liquid with two 20-cc portions of 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 ether, wash this ether free from mineral acids, unite the ether solutions in a weighed 400-cc beaker, evaporate most of the ether at a temperature below 50° C, dissolve the residue in neutral alcohol free from carbon dioxide, add phenolphthalein and titrate to exact neutrality with standard alcoholic potash. Evaporate the alcohol, dry to constant weight at 105° C, and weigh the soap.

Instead of using standard alcoholic potash for this determination, standard alcoholic soda may be used; but in this case the weight of Na_2O added should be calculated from the volume of standard NaOH used in titration and one-half of this weight added to the determined weight of soap before calculating the percentage.

If unsaponified or unsaponifiable matter is suspected, the determination may be corrected by dissolving the dried soap in 50 per cent alcohol, shaking out with ether, evaporating the ether and weighing the unsaponified and unsaponifiable matter. This weight is to be deducted from the weight of anhydrous soap before calculating percentages.

(*r*) *Sugar—Qualitative Test.*—Add a decided excess of hydrochloric acid to a solution of the soap, heat on steam bath for fifteen minutes, cool, filter from fatty acids, and test a portion of the filtrate for sugar by boiling for two minutes with an equal volume of Fehling solution. The formation of red cuprous oxide indicates sugar.

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