

Brown
[CHURCHILL'S TECHNOLOGICAL HANDBOOKS.]

SOAPS AND CANDLES:

EDITED BY

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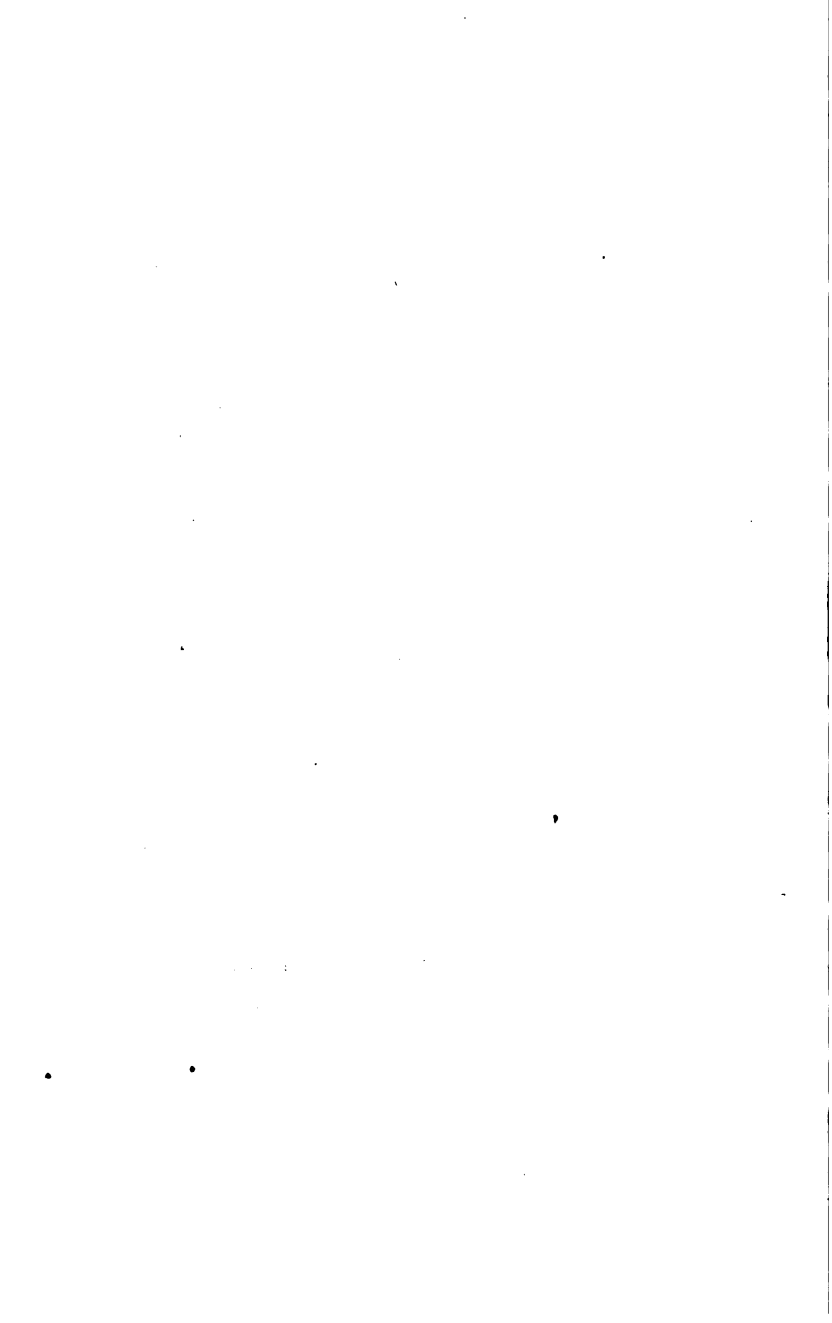
PREFACE.

As in the preceding Handbooks of this series, the articles in COOLEY'S "Cyclopædia" have been added to from various scattered sources, so as to present, in as small a compass as possible, information which, it is hoped, may be found useful to technological students and others interested in the industries described. In order to economize space, it has been assumed that the student has some previous knowledge of theoretical and practical chemistry, and details of many analytical processes, which are described in general treatises on practical chemistry, have been, for that reason, omitted.

The Editor has pleasure in expressing his thanks to Mr. LEOPOLD FIELD, Lambeth, and to Messrs. COOK, East London Soap Works, for much valuable information, and to his colleague, Mr. CHARLES CARTER, for assistance in revising the proofs.

J. C.

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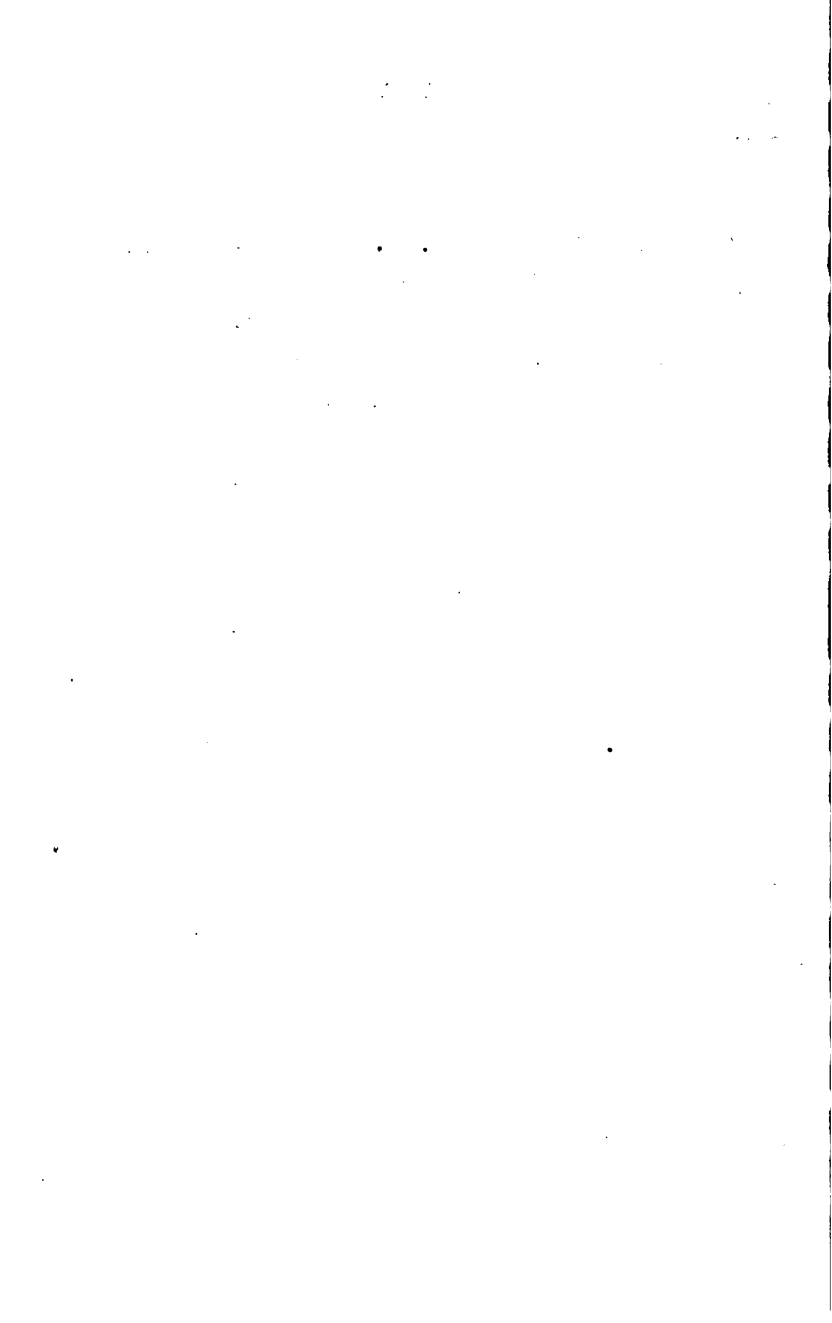
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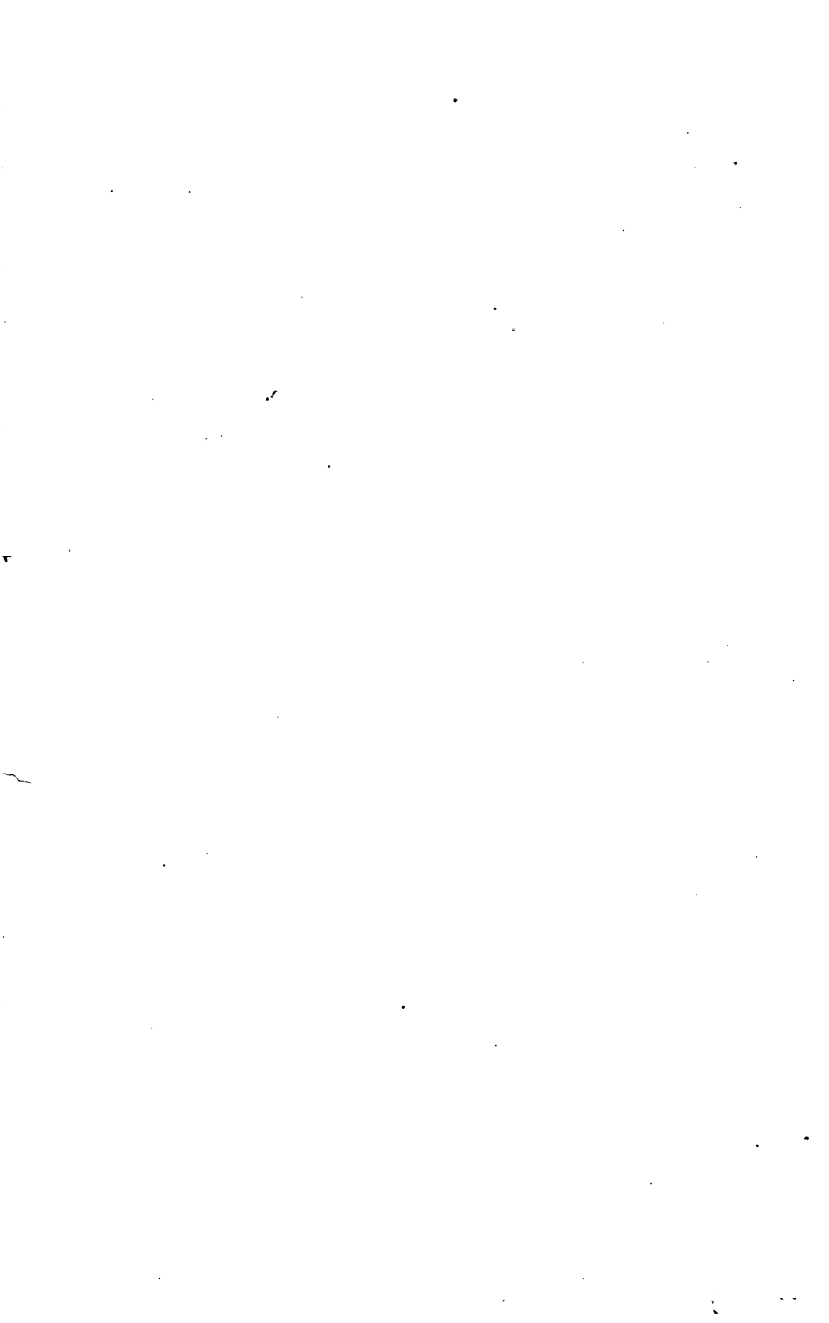
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SOAPS AND CANDLES.

PART I.—SOAPS.

CHAPTER I.

DEFINITION, HISTORY, AND PROPERTIES OF SOAP.

Definition.—Chemically speaking, a *soap* is produced whenever a metallic *base* is combined with a *fatty acid*, such as the acids, of the general formula $C_nH_{2n-2}O_2$, occurring in, or obtainable from, the natural fats or fixed oils, and hence, besides the ordinary commercial soaps, we have the *lead soap*, or lead plaster of pharmacy, and also *manganese*, *copper*, *mercury*, *zinc*, *tin*, *silver*, *aluminium*, and other *metallic* soaps. But, in ordinary language, by *soap* we understand a compound of an *alkali* and a fatty acid—the alkali potash affording, when so combined, *soft soap*, and the alkali soda forming *hard soap*.

According to another definition, soap is a chemical combination of any oily with any saline matter, whereby the oil acquires a solubility in menstrua with which, naturally, it refuses to unite.

MORFIT says: "True soap is a definite chemical compound

of one or more fat acids with a base, and a certain ratio of water of constitution."

KINGZETT* gives this definition: "Soap, considered commercially, is a body which, on treatment with water, liberates alkali."

A similar definition is mentioned in the Reports of the Juries, Exhibition 1851 (p. 607): "Soap is a sort of magazine of alkali, which it gives up in the exact quantity required at any moment when it is rubbed with water."

Dr. C. R. A. WRIGHT† states that "a soap, in the widest sense of the term, implies a compound of a fatty acid with an alkali, or other metallic derivative capable of playing the part of an alkali, glycerides not being classed as soaps, for the reason that glycerin, although capable to a certain extent of playing the part of an alkali, is neither such a metallic derivative nor an alkali itself."

History.—A complete soapery was found in excavating Pompeii, which contained some soap in a good state of preservation. Hence we are at once thrown back to the year A.D. 79 in our search for the first employment of soap. The elder PLINY, who perished at that time, is the first writer who mentions soap in the sense in which we now understand the word. He states‡ that it was made from tallow and ashes, the best materials being *goat's tallow* and *beech-ash*. He was also acquainted with the hard and soft varieties of soap. He ascribes the invention of the compound to the Gauls, but states that it was well prepared in Germany. References to cleansing by writers before this time only show that soap was unknown to them; for instance, HOMER gives us§ an account of the washing expedition of NAUSIKAA,

* "The Alkali Trade," p. 173.

† Cantor Lectures on Toilet Soaps, May 4, 1885.

‡ "Nat. Hist." xxviii. 12, 51 (HOLLAND's translation, ii. 328).

§ "Odyssey," vi. 90-118.

but without any mention of soap. The word occurs twice in the Scriptures,* but the original word in both instances is *borith*, and BECKMANN has shown† that this really means *alkali*.

We have distinct evidence that soap-making flourished in the seventeenth century, but it is only in the most modern times that the manufacture attained that extraordinary development for which this industry is remarkable, and which gave occasion for the following oft-quoted remarks of LIEBIG: "The quantity of soap consumed by a nation would be no inaccurate measure whereby to estimate its wealth and civilization. Of two countries with an equal amount of population, we may declare, with positive certainty, that the wealthiest and most highly civilized is that which consumes the greatest weight of soap."‡

Various circumstances have contributed to the advancement of this manufacture since the commencement of the present century, but two discoveries have specially influenced it—viz., CHEVREUL's discovery of the true nature of fats, and LEBLANC's discovery of a method for the artificial preparation of soda on a large scale. CHEVREUL's researches, although they explain the nature of saponification, have perhaps contributed less to the progress of the soap manufacture than to that of candle-making, but the development of the manufacture of soda has proved a most powerful stimulus to that of soap, by freeing it from dependence on the uncertain and limited supply of barilla and kelp.

Soap was formerly heavily taxed. An excise duty of 1*d.* per lb. was first imposed in 1711 on all soap made in Great Britain, and in 1713 this was raised to 1½*d.* per lb. In

* Jer. ii. 22; Mal. iii. 2.

† "History of Inventions," translated by JOHNSON (BOHN).

‡ "Familiar Letters on Chemistry," letter xi. p. 129.

1782 the duty was again increased, and a distinction was for the first time made between hard and soft soaps, the duty on the former being $2\frac{1}{4}d.$ and on the latter $1\frac{3}{4}d.$ per lb. In 1816 the duty on hard soap was raised to $3d.$ per lb. In 1833 the duty was $1\frac{1}{2}d.$ per lb. on hard soap and $1d.$ per lb. on soft. The duty was repealed in 1853.

Properties.—Only soaps made from alkalies and fatty acids are soluble in water, and consequently such only are valuable for cleansing purposes, and are commercially recognized as soaps. All other soaps are insoluble in water, the most familiar of these insoluble soaps being the lime soap, which separates in curdy particles whenever hard water is used for ordinary washing purposes.

But cold water, however pure, never entirely dissolves soap without decomposition. The neutral salts of which soap consists are resolved, in contact with water, into an alkali which dissolves and an acid salt which is precipitated.* The same decomposition takes place when hot weak solutions of soap are cooled. This behaviour explains why, in using soap even with the purest cold water, a white turbidity (*soap-suds*) is always produced. On this decomposition, probably, the purifying action of soap largely depends. The liberated alkali unites with the greasy dirt, and the insoluble acid salt forms the *lather* which envelopes it, and thus assists its removal.

According to Dr. W. LANT CARPENTER,† considerable light has been thrown upon the manner of removal of dirt by soap by the researches of the late Prof. W. STANLEY JEVONS, F.R.S., upon the "Brownian movement" of small particles. When clay is stirred up with water, and the

* MUSPRATT'S "Dictionary of Chemistry," ii. 875; WATTS' "Dictionary of Chemistry," v. 315; SPON'S "Encyclopædia," v. 1793.

† SPON'S "Encyclopædia," v. 1793.

water is allowed to stand, it clears itself very slowly, and microscopic examination showed that this was due to a kind of molecular movement of the infinitesimally small particles of the clay. To this movement Prof. JEVONS gave the name of *pedesis*, or pedetic action,* and he found that soap and sodium silicate enormously increased this action.† From these observations, and from his own experiments, CARPENTER is of opinion that in the action of these substances in promoting the molecular movement of extremely minute particles is to be sought part of the explanation of the cleansing power of soap.

Soap is readily soluble in alcohol and in *hot* water. A hot concentrated solution of ordinary soap solidifies on cooling to a jelly-like mass. Soap is insoluble in a solution of common salt, and if the latter is added to a hot solution of the former, the soap separates as an oily layer, which solidifies on cooling.

Prof. ROTONDI, of the Royal Industrial Museum of Turin, rejects the theory of BERZELIUS that the usefulness of soaps depends upon the facility with which neutral soaps decompose, on solution, into acid soaps and free alkali, and also that of PERSOZ, who assumes neutral soaps to be soluble in hot water without decomposition, but to be resolved in cold water into acid and basic soaps, the latter dissolving fatty substances by saponification. These hypotheses do not explain why hot soap solutions are more active than cold ones. The following conclusions were arrived at by ROTONDI from experiments upon carefully purified Marseilles soap:—Neutral soaps are decomposed, on solution, into basic and acid soaps; the latter are insoluble in cold, and only slightly soluble in hot water; they are not dia-

* "Quarterly Journal of Science," April 1878, No. lviii.

† Report of the British Association, 1878, p. 435.



lysable, and so are thus separable from the former, which readily dialyse. The neutral soaps, though thus decomposed, lose neither free nor carbonated alkali. Basic soaps are completely soluble in hot and cold water, and are entirely precipitated by sodium chloride without loss of alkali; their solutions dissolve acid soaps on heating, but become turbid on cooling. They emulsify fatty bodies readily, but no saponification of the latter takes place; neutral soaps possess this property to a smaller extent; acid soaps scarcely at all. Carbonic acid produces, in cold solutions of basic soaps, insoluble compounds, which, however, disappear on heating. Hence, waters rich in carbonic acid are not suited for industrial operations with soap. The above explains the greater efficiency of hot soap solutions, and has an important bearing upon the manufacture and industrial uses of soap, inasmuch as different results are often observed in the use of soaps made from the same materials (and containing no free alkali), which are due to their containing *variable amounts of acid and basic soaps*. ROTONDI considers that soaps should consist, as nearly as possible, of neutral compounds, instancing the injury arising in the boiling of silks from the presence of excess of basic soap, and that these points should be borne in mind in the operations of soap-boiling, as well as in soap analysis, when it is desired to ascertain the fitness of a sample for a given purpose.*

The partial decomposition of a neutral soap when treated with cold water is called *hydrolysis*, and has been recently carefully investigated by WRIGHT and THOMPSON,† with very interesting results. Their experiments were made as

* "Chem. Rev." xiv. 228; "J. Soc. Chem. Ind." 1885, p. 601.

† "J. Soc. Chem. Ind." 1885, p. 629.

follows:—The soaps examined were either prepared by themselves, or obtained from manufacturers with information as to the fatty acids present; they were carefully examined, and, in those cases where minute amounts of free alkali were present, corrections were made for these small amounts. Weighed quantities of soap, representing known quantities of *anhydrous* soap, were dissolved in known quantities of distilled water on the water-bath, and, after cooling to near the ordinary temperature, the liquids were treated with pure sodium chloride, so as to throw out of solution all, or nearly all, the soap as curd. The curds thus precipitated, on drying and dissolving in alcohol, were always more or less acid, phenol-phthalein being the indicator, so that one way of determining the amount of hydrolysis was to determine the amount of alcoholic potash or soda solution required to neutralize this acidity. It was found, however, in practice to be far more convenient to determine the alkali contained in an aliquot part of the brine, correcting the amount found for the free alkali (if any) originally contained in the soap. With smaller proportions of water, the addition of moderate quantities of salt sufficed to throw all soap out of solution so perfectly that, at most, only traces of fatty acids could be obtained from the brine by acidulating and shaking with ether; with larger proportions it was found convenient to evaporate the brine until nearly saturated, and filter again from any soap thrown out of solution during the evaporation, using only a fraction of the salt requisite to saturate the water for the salting out, rather than to use more salt in the first instance and titrate without evaporation, greater accuracy in hitting the terminal reaction being thus attained. With cocoa-nut oil soaps the brines were evaporated to dryness, and then treated with just sufficient water to dissolve the salt, and

filtered. In this way, liquids free from more than traces of soap were obtained, the soap originally contained in the brine before evaporation being thus eliminated. The following corrected mean values were obtained in a lengthy series of experiments with various soda soaps, all of which were neutral or only faintly alkaline. The numbers represent the quantities of Na_2O set free by hydrolysis, reckoned per 100 parts of Na_2O combined with fatty acids in the soap, x molecules of water being used for one of anhydrous soap.

| Fatty Acids. | Mean Molecular Weight. | Hydrolysis brought about by x Molecules of Water. | | | | |
|--|------------------------|---|----------|----------|-----------|-----------|
| | | $x=150.$ | $x=250.$ | $x=500.$ | $x=1000.$ | $x=2000.$ |
| Pure stearic acid | 284 | 0.7 | 1.0 | 1.7 | 2.6 | 3.55 |
| Nearly pure palmitic acid | 256 | 1.45 | 1.9 | 2.6 | 3.15 | 3.75 |
| Crude lauric acid (cocoa-nut oil soap) | 195 | 3.75 | 4.5 | 5.4 | 6.45 | 7.1 |
| Pure oleic acid | 282 | 1.85 | 2.6 | 3.8 | 5.2 | 6.65 |
| Crude ricinoleic acid (castor oil soap) | 294 | 1.55 | 2.2 | 3.0 | 3.8 | 4.5 |
| Chiefly stearic, palmitic and oleic acids (palm oil and tallow soap) | 271 | 1.1 | 1.55 | 2.6 | 4.1 | 5.3 |
| Chiefly tallow and resin (primrose) | 280 | 1.5 | 2.2 | 3.1 | 4.2 | 5.3 |
| Cotton seed | 250 | 2.25 | 3.0 | 5.0 | 7.5 | 9.5 |

The above results lead to the following general conclusions :—

1. The amount of hydrolysis brought about by the action of a given quantity of water on a neutral soap is variable with the nature of the fatty acids from which the soap is made, but in all cases increases with the amount of water employed relatively to the soap, but less rapidly.

2. Addition of excess of alkali to a neutral soap causes a diminution in the amount of hydrolysis effected under

given conditions, to such an extent as completely to stop the action with comparatively small proportions of water, when the free alkali only amounts to a fraction (say 20–25 per cent.) of the alkali combined with the fatty acids.

3. Alcohol, even when not absolutely anhydrous (90–95 per cent.), does not decompose ordinary neutral soaps into free alkali and acid salts. If, however, water be added to an alcoholic soap solution, more or less hydrolysis takes place, so that if a gelatinous mass of neutral soap, dissolved in strong spirit containing a little phenol-phthalein, be treated with water, a more or less strongly marked coloration is noticeable as the water diffuses into the mass.

The following observations of LIEBIG on the behaviour of soap with a solution of common salt are of great practical importance to the soap-maker:—"If a piece of common hard soap be placed in a solution of salt at ordinary temperature, it floats upon the surface without even being moistened, and, if the liquid be heated to boiling, it separates without foam into gelatinous flocculæ, which collect on the surface, and, upon cooling, unite into a solid mass, from which the solution flows off like water from fat. If the flocculæ be taken out of the hot fluid, they congeal, on cooling, into an opaque mass, which may be pressed into fine laminæ between the fingers without adhering to them. If the solution is not quite saturated, the soap then takes up a certain quantity of water, and the flocculæ separate through the fluid on boiling. But even when the water contains $\frac{1}{100}$ th part of common salt, ebullition does not produce solution. If the soap is boiled in a dilute and alkaline solution of salt, and suffered to cool, it again collects on the surface of the fluid in a more or less solid state, depending on the greater or less degree of concentration of the solution—that is, on the quantity of water taken up by the soap. By boiling the dilute solution of salt with soap for a considerable time, the

aqueous flocculæ intumesce, and the mixture assumes a foamy appearance; but still they are not dissolved, as the solution separates from them. The flocculæ, however, have become soft and pasty, even after cooling, and their pastiness is due to the quantity of water they have taken up. By continuing the boiling, this character again changes, and in proportion as the water evaporating renders the solution more concentrated, the latter again extracts water from the flocculæ; the liquid, however, continues to foam, but the bubbles are larger. At length a point is reached at which the solution becomes saturated; the larger iridescent bubbles, formed just before, disappear, and the liquid continues to boil without froth; all the soap collects as a translucent mass on the surface; and now the solution and soap cease to attract water from each other. If the plastic soap be now removed and cooled, while the solution is pressed out, it becomes so solid as scarcely to receive an impression from the fingers. In this state it is called *grain-soap*.

"The addition of salt, or its solution, to a concentrated alkaline solution of soap in water, precipitates the soap in gelatinous flocculæ, and the mixture behaves precisely as solid soap boiled with a solution of salt. Potassium carbonate and caustic potash act exactly as salt in separating soap from the alkaline fluid.

"The application of these facts to the manufacture of soap is obvious. The fat is kept boiling in an alkaline lye until all pasty matters disappear; but the lye should have only a certain strength, so that the soap may be fairly dissolved in it. Thus, tallow may be boiled for days in a caustic potash solution of specific gravity 1.25 without being saponified. If the lye be stronger, a partial saponification ensues; but, being insoluble in the fluid, the soap floats on the surface as a solid mass. By the gradual addi-

tion of water, with continued boiling, the mass at a certain point becomes thick and clammy, and with more water an emulsion is formed. On continued heating, this becomes perfectly clear and transparent if a sufficient quantity of alkali be present. In this state it may be drawn out into long threads, which, on cooling, either remain transparent, or are milky and gelatinous. As long as the hot mass suffered to drop from a spatula exhibits a milkiness or opalescence, the boiling is continued, or more alkali added. When excess of alkali is present, the milkiness arises either from imperfect saponification, or want of water; the former is known by dissolving a little in pure water, which becomes perfectly clear when the whole is saponified. If the lye contain lime, the mixture is also turbid, but the addition of an alkaline carbonate causes the turbidity arising from this cause to disappear instantly.

“In order to separate the soap from water, free alkali, and glycerin, a large quantity of salt is gradually added to the boiling mass, waiting, after each addition, till the portion added is completely dissolved. The first addition increases the consistency of the mass, while each successive portion renders it more fluid, till it loses its adhesive character, and drops from the spatula in short thick lumps. As soon as the congelation is complete—that is, when gelatinous floculæ separate from a clear watery liquid—the fire is extinguished, the soap allowed to collect on the surface, and cooled either on the liquid or ladled out, and allowed to get solid.

“The same results are also produced, although in a less energetic manner, by potassium chloride, alkaline carbonates, sodium sulphate, potassium acetate, and ammonium chloride. Of these, sodium sulphate and potassium chloride have but a very slight action. Concentrated caustic lyes also separate soap from its solution in the

same manner as common salt. In weak caustic lye, on the contrary, soap is perfectly soluble. On this account, soap-boilers, especially at the commencement of the operation, except in the case of cocoa-nut oil, always use weak lyes, as the stronger would prevent the necessary amount of contact amongst the ingredients, and very much retard the process of saponification. Thus, by means of caustic or saline solutions, not only all foreign matters, but also the glycerin, may be completely separated from soap."

T. N. WHITELOW,* starting with the well-known fact that soaps, when boiled with solutions of common salt, retain amounts of water inversely as the quantity of sodium chloride in solution, has described certain experiments instituted by him with the view of defining this action. Tallow and palm-nut oil soaps were selected as affording types of the manner in which solutions of soap behave with salt, the greater number of oils used in soap-making resembling tallow, while cocoa-nut oil is more like palm-nut oil in the manner in which its soap solution behaves. Six grms. of fatty acids from tallow and palm-nut oils respectively were saponified in a flask of about 250 c.c., separated with excess of caustic soda from solution, and, after cooling till the soap curds had solidified, the caustic soda liquor was allowed to drain off.

The soaps so obtained were dissolved in 100 c.c. of distilled water, and a weighed quantity of pure NaCl added, enough to obtain distinct separation of the soap in small curds. Then water was added in small quantities from a burette, and the soap solution brought to the boiling point and well agitated after each addition, a cork provided with about 14 inches of glass tubing preventing any loss of water during the momentary boiling.

* "Journ. Soc. Chem. Ind." 1886, p. 90.

With a certain strength of solution, the soap grains were distinct and separate, without any tendency to settle out fluid soap. With a further addition of water, the grains became softer, and a thin layer of fluid soap could be observed settling beneath them. With the addition of still more water, the grains became entirely fluid, and the fluid soap occupied more and more of the total fluid volume as more water was added, until a point was reached when the fluid became clear and bright, and the soap completely dissolved.

The points of distinct separation in grains, and the points of complete solubility in boiling solutions, were found to be as follow :—

| | Separation in Distinct Grains. NaCl per Cent. | Completely Soluble. NaCl per Cent. |
|----------------------------|---|--|
| Tallow soap | 6.5 | 3.0 |
| Palm-nut oil soap. | 18.0 | 13.0 |

On cooling these solutions, the tallow soap remained *completely soluble*, and, when cold, formed a firm jelly, while the palm-nut oil soap separated as it cooled into a thin layer on the surface of the salt solution. It was found that the tallow soap is nearly as soluble in the *cold* as in the *hot* solution of salt, but that while palm-nut oil soap is soluble in *boiling* water containing 13 per cent. of NaCl, it is insoluble in cold solution of 3 per cent.

A difference was observed in the composition of the soap according as it separated in *distinct grains* from the saline solution, or as *slightly liquefied grains* floating on a thin layer of liquid soap above the solution. In the first case we have an ordinary soft *curd soap*, and in the second case, if we consider the supernatant saline solution removed, we have soap grains *washed by a solution of soap*. Both these methods—viz., *purifying with salt* and *purifying with soap*

—are adopted on the large scale to obtain pure soap of a definite composition.

WHITELAW obtained the following results from the analysis of the two soaps thus prepared:—

| | Soap settled from | |
|--|-------------------|---------------------|
| | Salt. | Soap. |
| | Palm Oil—Curd. | Tallow—Fitted Soap. |
| Water | 31.38 | 31.4 |
| Soda (Na_2O) | 7.13 | 7.0 |
| Fatty anhydrides | 59.82 | 60.3 |
| Sodium chloride | 1.67 | 1.3 |
| | 100.00 | 100.0 |

Action of More Concentrated Solutions of Common Salt upon Soaps.—A soap from a good quality of olive oil, after boiling for thirty minutes with an 8 per cent. solution of salt, retained 31.6 per cent. of water; with a 17 per cent. solution, it retained 25.7 per cent.; and with a saturated salt solution, it retained 19.1 per cent.

The following are analyses of various soaps after thirty minutes' treatment with a hot saturated solution of salt:—

| Soap. | Water. Per Cent. | Fatty Anhydrides. Per Cent. | Soda. Per Cent. | Sodium Chloride. Per Cent. |
|------------------|---------------------|-----------------------------------|--------------------|----------------------------------|
| Olive oil . . . | 19.1 | 67.9 | 7.8 | 5.2 |
| Tallow . . . | 16.94 | 64.49 | 7.64 | 10.93 |
| Palm-nut . . . | 18.8 | 66.4 | 9.9 | 4.9 |
| Cotton oil . . . | 17.2 | 62.4 | 6.4 | 14.0 |
| Castor oil . . . | 48.3 | 31.3 | 3.7 | 16.7 |

Prolonged boiling does not reduce the quantity of water retained by the soap after thirty minutes' treatment. This is shown by the following analysis of a soap after sixty minutes' treatment:—

Olive Oil Soap—treated Sixty Minutes.

| | |
|----------------------------|--------|
| Water | 19.41 |
| Fatty anhydrides | 67.38 |
| Soda | 7.75 |
| Sodium chloride | 5.46 |
| | <hr/> |
| | 100.00 |

There is thus a limit to the action of sodium chloride in withdrawing water from soaps.

Soap which contains a larger amount of water than curd soap is called *watered* when water or weak lye is added and mixed with the curd in the pan itself, or when the curd is treated subsequently with water whilst still in contact with the brine. When, however, the water is added and crutched into the curd *after* its removal from the pan, the soap is termed *liquored* or *filled*. The term *filled* is also applied to soap which has been mixed either with the soluble alkaline carbonates, sulphates, or silicates, or with such insoluble materials as barium sulphate, chalk, clay, china clay, fuller's earth, pumice stone, sand, steatite, starch, talc, &c.

The following are the characters of soap given in the British Pharmacopœia (1885):—

HARD SOAP (*Sapo durus*: white Castile soap), made from soda and olive oil.—Colour, yellowish-white. Dry, inodorous. Horny, and pulverizable, when kept in dry warm air. Easily moulded when heated. Soluble in rectified spirit. Soluble also in hot water, the solution being neutral or only faintly alkaline to test-paper. It does not impart a greasy stain to paper. Incinerated, it yields an ash which does not deliquesce.

CURD SOAP (*Sapo animalis*).—Made from soda, and a purified animal fat consisting principally of stearin. Colour, white, or with a very light-greyish tint. Otherwise, its characters are the same as those of *sapo durus*.

SOFT SOAP (*Sapo mollis*).—Made from potash and olive

oil. Colour, yellowish-green. Inodorous. Of a gelatinous consistence. Soluble in rectified spirit. Does not impart an oily stain to paper. Incinerated, it yields an ash which is very deliquescent.

The United States Pharmacopœia (1883) gives the following characters of soap prepared from soda and olive oil :—A white or whitish solid ; hard, yet easily cut when fresh ; having a slight, peculiar odour, free from rancidity ; a disagreeable, alkaline taste, and an alkaline reaction. Readily soluble in water and in alcohol. When cut into thin slices, and dried to a constant weight at a temperature of 110° C. (230° F.), it should not lose more than 34 per cent. of its weight (absence of an undue amount of water). A 4 per cent. alcoholic solution should not gelatinize on cooling (absence of animal fats). 100 parts of the soap when dissolved in alcohol should not give more than 3 parts of insoluble residue (limit of sodium carbonate, &c.), and at least 2 parts of this residue should be soluble in water (limit of silica, and other accidental impurities).

CHAPTER II.

M A T E R I A L S.

THE materials which are essential to the soap manufacturer may be classified under two heads:—1. Fatty matters and rosin; 2. Alkalies.

1. FATTY MATTERS.

The fatty substances employed have been fully described in the fourth volume of this series of Handbooks,* but the tabular statement given on pp. 18–22 will be convenient here.

Recovered Grease.—Obtained from the washings of woollen works. These are decomposed with sulphuric acid, steam is admitted to hasten separation, the fatty acids are filtered through hempen cloth, and the fatty mass then subjected to hydraulic pressure. It is of a brownish colour, and requires to be used with judgment, as it is liable to contain varying quantities of unsaponifiable oils. Also, extracted by carbon disulphide from the residue of stearin factories, from sawdust which has served for filtering oil, from refuse waggon grease, from oily rags, &c.

Recovered grease is never used alone in soap-making, but mixed with palm oil or tallow, and chiefly for rosin soaps.

Rosin.—Syn. COLOPHONY.—This is an important ingredient in the production of soap. It is the residue left

* "Oils and Varnishes."

A. Animal Fats.

| NAME. | SOURCE. | PROPERTIES, &c. |
|-----------------------|--|---|
| Bone grease . | Fresh or refuse bones bruised, boiled in water, and the fat skimmed off when cold. | Colour—light yellow to brownish. |
| Egg oil . | Yolk of eggs—the residue in the preparation of albumen for the use of calico-printers. | Colour—deep yellow. Semi-fluid at common temperatures. Begins to solidify at 46° to 50° F. The impure kinds are used for "Kazan" soap. |
| Glue fat . | A bone grease obtained in the process of making glue. | Resembles bone grease. |
| Horse grease . | Fat of horses | Odour—peculiar. Use—chiefly confined to palm and rosin soap. Sp. gr. .919 to .920 at 60° F. |
| "Kitchen stuff". | Kitchen waste | Various, being a mixture of different fats. |
| Lard | Fat of the pig, melted, and strained through flannel or hair-sieves. | White. Sp. gr. at 100° F., .9037 to .90483. M. P. 108° to 114° F. Fixed acids, 95.62 to 95.93 per cent. (Bell). By saponification, yields 9 parts of glycerin and 94.65 per cent. of mixed fatty acids. |
| Tallow | By "rendering" the fat of oxen, sheep, and other ruminants. Inferior kinds for soap-making are <i>town tallow</i> , <i>melted stuff</i> , and <i>rough stuff</i> . | Olein, 62 per cent.; solid portion, 38 per cent. (BRACONNOT). |
| Tallow oil, or olein. | By pressure from tallow | M. P. 115° to 121° F. Contains stearin, palmitin, and olein, the first predominating. |
| | | Thick. Consists chiefly of olein. Well adapted for the finer kinds of soap. |

B. Fish Oils.

| | | |
|-----------------------|---|--|
| Cod-liver . . . | As implied by name . . . | Pale: sp. gr. .923 to .9238. Light brown: sp. gr. .924 to .9245. Dark: sp. gr. .929 to .9315. Colour — brownish. Smell — unpleasant. Dries slowly. |
| Seal . . . | Blubber of hooded seal (<i>Phoca cristata</i>), harp seal (<i>Phoca greenlandica</i>), and other species of seal. | |
| Sperm . . . | The fluid portion of the head-matter of the sperm whale (<i>Physeter macrocephalus</i>). | Sp. gr. .875. |
| Spermaceti . . . | The solid portion of the head-matter of the sperm whale. | White, sealy, or crystalline, brittle. Sp. gr. .943. M. P. 110° to 113° F. |
| Whale, or train, oil. | Blubber of common or Greenland whale (<i>Balaena mysticetus</i>). | Colour — brownish. Sp. gr. .923. |

C. Vegetable Oils.

1. Drying.

| | | |
|------------------------|--|---|
| Castor . . . | By pressure from seeds of <i>Ricinus communis</i> . | Sp. gr. .9611 to .9612. |
| Cotton-seed . . . | From the "lint" or "wool" of <i>Gossypium barbadense</i> by heat and pressure. | <i>Crude oil</i> : sp. gr. .928 to .930 (GILMOUR). <i>Refined oil</i> : sp. gr. .920 to .923. Congealing point, 45° to 32° F. Now much used in soap-making. |
| Dilo, or Tamannu . . . | Seeds of <i>Calophyllum inophyllum</i> , a tropical tree. | Colour — greenish. Sp. gr. about .940. |
| Hemp-seed . . . | Seeds of <i>Cannabis sativa</i> . . . | Colour — greenish-yellow, turning brown with age. Sp. gr. .925 to .931. Thickens at 5° F. Freely soluble in boiling alcohol. |

C. VEGETABLE OILS—(continued).

1. *Drying*—(continued).

| NAME. | SOURCE. | PROPERTIES, &c. |
|------------------------|--|---|
| Linseed . . . | By pressure from seeds of <i>Linum usitatissimum</i> . | Sp. gr. .930 to .935. |
| Poppy-seed . . . | By pressure from seeds of opium poppy (<i>Papaver somniferum</i>), yellow-horn poppy (<i>Glaucium luteum</i>), spiny poppy (<i>Argemone mexicana</i>). | Colour—pale. Taste—sweet. Sp. gr. .913 to .924. Does not freeze till cooled to 0° F. |
| Sunflower-seed *. | From the seeds of <i>Helianthus annuus</i> and <i>perennis</i> . The seeds yield from 15 to 28 per cent. of oil. | Clear, tasteless, and of a pale-yellow colour. It dries slowly. Thickens and becomes turbid at 60° F. (15.5° C.), and solidifies at 4° F. (—16° C.). Sp. gr. .926. Saponifies readily in the cold to a white, odourless soap, giving a free and permanent lather. |
| 2. <i>Non-drying</i> . | | |
| Almond, sweet . . . | By pressure from sweet or bitter almonds. | Odourless. Colour—yellow. Sp. gr. .915 to .920. Solidifies at —25° C. Chiefly consists of olein. |
| Beech . . . | By cold or hot expression of the decorticated nuts of the beech (<i>Fagus sylvatica</i>). | Colour—yellow. Taste—slightly acid. Sp. gr. .9225. |

* "Colonial and Indian Exhibition Reports, 1887," "Oils and Fats," p. 273. LEOPOLD FIELD says, "The perfect soap has yet to be made, and it will probably be made from sunflower oil."

| | | |
|---|--|---|
| Cacao butter | From seeds or nlbs of <i>Theobroma cacao</i> , by heat and pressure. | Colour—white to yellowish. Fuses about 86° F. (30° C.). Sp. gr. .945 to .952. Used for high-class toilet soaps. Does not readily become rancid. |
| Cocoa-nut oil, or butter (sometimes also called <i>Copra oil</i>). | Separated by hydraulic pressure from the dried kernels of the common cocoa-nut (<i>Cocos nucifera</i>). | Consistence of butter. Fuses at 73° to 80° F. Contains olein, and a solid fat used for candle-making. |
| Colza, or rape | Seeds of <i>Brassica campestris</i> , var. <i>oleifera</i> . | When fresh, has a sweet taste and agreeable odour, but easily turns rancid. |
| Illipa oil | By pressure, or by boiling with water, from seeds of <i>Bassia latifolia</i> or <i>longifolia</i> . | Colour—yellow, or brownish yellow. Sp. gr. .912 to .920. Congeals at 21° F. |
| Galam, or Shea, butter. | From <i>Bassia parkii</i> , a native of Africa. | Colour—light-greenish yellow. Smell—aromatic. M. P. 72 to 73½° F., and therefore solid at moderate temperatures. Prone to rancidity. |
| Ground-nut or arachis. | Seeds of ground-nut (<i>Arachis hypogaea</i>). | Similar in appearance to palm oil. Colour—dirty yellow. Solid at 97° F. On saponification, yields only stearic and oleic acids. |
| Olive | Only the inferior kinds are used in soap-making—e.g.: (a) "Gorgon"—obtained by fermenting and boiling in water the pressed cake, or marc, after extraction of the finer kinds, and skimming off the oil. (b) "Oil of the infernal regions"—skimmed off the waste water in the reservoirs. (c) "Droppings" or "foots"—the drippings of casks, cisterns, and utensils used in the storage of olive oil. | Sp. gr. .916. Sp. gr. .914 to .918. |

C. VEGETABLE OILS—(continued).
2. Non-drying—(continued).

| NAME. | SOURCE. | PROPERTIES, &c. |
|---------------------------|---|--|
| Palm oil, or butter | From the fruit of several species of palm, chiefly <i>Elaeis guineensis</i> . | Colour — orange-yellow tint. Taste — sweetish. Odour—like violets or orris root. M. P. 76° to 95° F. Sp. gr. about .968. "Lagos" oil is generally the most neutral, and "Brass" oil the hardest. It is bleached by exposure to sunlight, chlorine, chromic acid, and sulphuric acid. It is readily bleached by exposure to the air, and in this way the characteristic agreeable odour of the oil can be obtained in soaps, without the drawback of any yellow tinge being imparted to the lather. Mr. LEOPOLD FIELD expresses the opinion that the delicacy and persistency of the scents of French toilet soaps is in some measure due to the fact that French toilet-soap makers employ bleached palm oil much more than their English brethren.* |
| Palm-nut, or palm kernel. | From the nuts or kernels of the fruit. | Colour—primrose-yellow. Odour—resembles coconut oil. |

* "Colonial and Indian Exhibition Report, 1887," p. 266.

in the retorts after the distillation of common turpentine. Chemically, it is a mixture of a large quantity of *abietic anhydride* or *pinic acid* ($C_{44}H_{68}O_4$) with a little *sylvic* ($C_{20}H_{30}O_2$) and *colophonic* or *pimaric acid* ($C_{20}H_{30}O_2$), a mixture which, from the nature of the components, possesses the properties of a weak acid. In making rosin soap, the alkali becomes merely saturated with these resinous acids; there is no basic constituent, like the glycerin of the fats, to displace.

When an aqueous solution of a rosin soap is treated with common salt, there is no separation of the soap, as in the case of soaps made from fats. Rosin soap also differs from ordinary soap in the circumstance that its hot concentrated solution does not gelatinize on cooling.

2. ALKALIES.

The alkalies used in soap-making are soda and potash, which are commercially obtainable either as carbonates or in the caustic state.

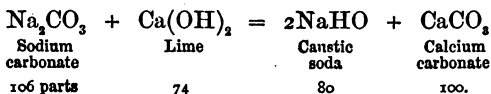
Caustic-soda Lyes.—Formerly the soap-maker purchased the carbonates, and causticized them himself, but the obvious advantages derivable from obtaining in the first instance *caustic* alkalies have led to the frequent abandonment of the causticizing process in the soap factory itself, especially in the smaller factories. By this change a saving is effected in space, plant, time, and labour, and the strengths of the lyes* are more under control. It is only necessary to dissolve the caustic soda or potash in a given quantity of water to produce lyes ready for use of any required strength.

The following is an outline of the process for preparing caustic lyes from soda-ash:—Five parts of freshly burnt

* This word is often pronounced *lees*, and is variously spelt, but the orthography adopted is probably the most satisfactory.

lime are laid evenly over the bottom of the vat, and water is poured on till it begins to slake. Over this layer is then immediately spread a layer of six parts of the soda-ash. Then a second layer of slaked lime is placed above these, followed by another quantity of soda-ash, and so on. After standing two hours, the tank is *stanch*ed by gradually filling up with water or weak lye. In about fifteen hours the plug at the bottom may be loosened, and the *first runnings* drawn off. The tank is afterwards again filled up with water, which is allowed to stand a sufficient time, and then drawn off as *second runnings*. After this the contents are turned over into another vat, covered with water, and, after a little time, again run down. The runnings from this operation are very weak, and are usually employed instead of water for filling up the first vats.

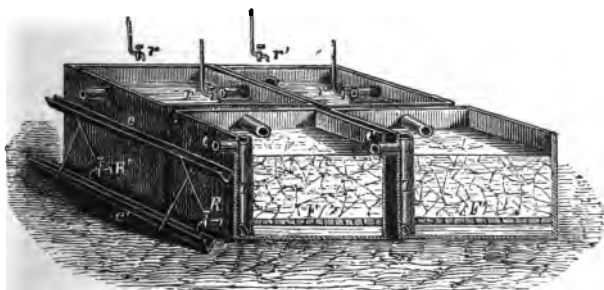
The reaction which occurs when soda-ash is causticized may be represented by the equation—



Caustic-soda lyes may also be prepared from *black ash*. The composition of this is given in the Appendix. It will be seen that it contains a large number of salts, but that the chief ingredients are *sodium carbonate* and *calcium monosulphide*, these two together amounting to from 50 to 75 per cent. of the whole. By lixiviation, the soluble carbonate is washed out with the smallest possible quantity of water, leaving as an insoluble residue the monosulphide, with the excess of lime and calcium carbonate. Many methods have been proposed for extracting the soda as thoroughly as possible, all, however, on the principle of treating the fresh ash with strong lye, the partially exhausted ash with weak lye, and the nearly exhausted ash with water. The follow-

ing is a description of the plan devised by the late JAMES SHANKS, of St. Helens, Lancashire. It is based on the fact that a solution becomes more dense the more saline matter it has in solution, and that a column of weak lye of a certain height balances a shorter column of stronger lye. The tanks are arranged as shown in Fig. 1, and through them water is made to flow, acting upon the black ash in its passage, and thus becoming more and more saturated and dense in each consecutive vessel of the series, the saturated lye running off from the last tank. The tanks are 2.6 metres long by 2 metres in depth. *F* is the perforated

FIG. 1.

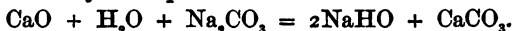


sheet-iron bottom. The tanks are connected with each other at the top and bottom by the tube *T t*. Water may be run in by the pipes *r, r', r'', r'''*, and the lye emptied into the channel *c'* by the taps *R, R'*. As a rule, four lixiviations are sufficient. The working is as follows:—Black ash having been thrown in until the tanks are nearly filled, water is run in. After a time fresh water is run in from the same tap, and the liquor is driven up the pipe *T* through *t* into the second tank. By means of a plug at the upper end of the pipe the flow can be regulated. The operation is continued by bringing fresh water upon the exhausted ash and

saturated liquor upon fresh black ash. The average time for working off a vat is about forty-eight hours.

The process does not thoroughly wash out the sodium carbonate, about 3 per cent. being still left in the waste.

The saturated lye, diluted, if necessary, to 22° Tw., may be causticized with milk of lime, about 15 cwt. of unslaked lime being required for every ton of caustic soda to be obtained. The lye and the hydrate of lime are agitated thoroughly together by means of a stirrer, and in about half an hour the decomposition is finished. The reaction is represented by the equation—



After the calcium carbonate has settled, the clear caustic soda lye is run off.

Steam Lyes.—In preparing these, 100 lb. of soda require 50 lb., and 100 lb. of potash 80 lb., of lime. The proportion of water is 12 parts to 1 of potash, and rather less for soda. The previously slaked lime and the alkali are introduced into the vat and boiled by a current of steam for several hours, until a portion taken out and left to repose shows that the whole of the alkali has been causticized by not effervescing on addition of hydrochloric acid, or by remaining clear on addition of lime-water. The mixture is then allowed to repose till the calcium carbonate deposits, and the supernatant liquor is drawn off into the alkali tank. The residue is then stirred up with fresh water, and the weak liquor thus obtained may be used to dilute stronger lyes, or instead of water in another boil.

By this method the decomposition of the sodium carbonate is very thorough, and, as lime is less soluble in hot than in cold water, the resulting lyes contain rather less lime than if made in the cold.

Caustic-potash Lyes may, when desired, be prepared in the same way as the caustic-soda lyes.

To preserve lyes it has been suggested* to throw upon the surface of the yet warm lyes a sufficient quantity of paraffin to cover, when melted, the whole surface, and thus form a layer which will completely shut out carbonic acid. No special vessels are necessary, and the paraffin can be used repeatedly for the same purpose.

Testing Soda-ash.—In England the equivalent of sodium is taken as 24, in commercial analyses, instead of 23, and that of sodium carbonate, 108, instead of 106. The comparison between English degrees and the French, or *DEGROIZILLES'*, degrees is shown in the Appendix.

Soda-ash is sold according to the percentage of available soda, calculated as sodium carbonate, which it contains—at so much *per degree*, or *per unit*. This percentage is arrived at by neutralizing with standard sulphuric acid a solution of a known weight of the ash in hot water—sodium hydrate, aluminate, and silicate all testing, in this way, as carbonate. It is unnecessary here to enter into the details of the process, as these, together with the tests for the impurities, such as chlorides, sulphides, sulphates, &c., will be found fully described in general works on practical chemistry.

Sodium Silicate.—*SOLUBLE GLASS* ($\text{Na}_2\text{SiO}_3 \cdot 8\text{OH}_2$).—The use of sodium silicate in the manufacture of soap is due to Mr. SHERIDAN, who took out a patent for the invention about 1835. His process for the preparation of this silicate is essentially as follows:†—A mixture of 1 part of sand with 3 parts of soda is heated to fusion in a reverberatory furnace. The product of this operation is then drawn out into water, and dissolved therein by the aid of heat. If, instead of sand, flint or quartz is the silicious material, it is

* CRISTIANI, "Technical Treatise on Soap and Candles," p. 254.

† MUSPRATT, "Dictionary of Chemistry," ii. 885; WATT, "Art of Soap-making," p. 30.

first calcined, and then powdered by wet grinding with horizontal stones. The impalpable powder obtained is thinned out with water, and then boiled for about eight hours with caustic-soda lye of 30° Tw. (sp. gr. 1.15). When the mass becomes homogeneous, the operation is finished. It is called, technically, *detergent mixture*, and is ready for mixture with soap paste.

GOSSAGE'S METHOD.*—Mix 9 parts of soda-ash of 50 per cent. with 11 parts of clear sand or powdered quartz, and fuse the mixture in a reverberatory furnace provided with a tap-hole through which the finished product may be run off. The product is received in metallic moulds, or in moulds formed of damp sand. The charge for a furnace having a bed of 60 square feet area is about a ton of the mixed sand and alkali, and each charge requires about four or five hours to be properly fused and combined. It is always desirable to use such proportions of alkali and sand for the production of the silicate that the latter may be almost wholly soluble in water. When the alkali is deficient, however, such is not the case, and to obtain perfect solution it is then necessary to use as the solvent a solution of caustic soda or potash. To effect its solution, the silicate is first ground to powder, and then heated in water, steam being introduced into the water to keep up the temperature. When nearly all is dissolved, the undissolved matters are allowed to subside, and the solution, transferred to a cast-iron evaporating pan, is concentrated by the application of heat till it has a specific gravity of about 1.45, when it becomes viscous on cooling, and is then in a condition to be added to the soap.

The solution of sodium silicate which is usually supplied to soap-makers is composed of silicic acid and soda in various

* MUSPRATT, ii. 885.

proportions, and is of two kinds, the *neutral* and the *caustic*. The *neutral silicate* has a specific gravity of about 1.37–1.45, and contains—

| | | | | | |
|----------------------|---|---|---|---|--------------------|
| Water | . | . | . | . | about 65 per cent. |
| Silicic acid | . | . | . | . | " 26 " |
| Soda and impurities. | . | . | . | . | " 9 " |

The *caustic silicate* has a specific gravity of about 1.7, and contains—

| | | | | | |
|----------------------|---|---|---|---|--------------------|
| Water | . | . | . | . | about 43 per cent. |
| Silicic acid | . | . | . | . | " 33 " |
| Soda and impurities. | . | . | . | . | " 24 " |

Potassium Silicate.—For admixture with soft soaps the soluble glass is formed by the fusion of a mixture of equal parts of sand and dry potassium carbonate (or pearl-ash) in the same way as in the preparation of sodium silicate. No compound of definite composition is known.*

Soap-makers, however, generally obtain a viscous mass of the alkaline silicate, which they reduce with hot water to any strength they require.†

DUNN'S PROCESS.—By means of the apparatus represented on p. 65, Fig. 10, either silica itself or an alkaline silicate is made to unite with soap under steam pressure. The crushed flint or quartz is introduced into the boiler with caustic-soda or potash lye in the proportion of 1 cwt. of the former to 100 gallons of the latter at 21° B. (32° Tw.—sp. gr. 1.16). The whole is then heated to about 310° F. and kept under a pressure of 50 to 70 lb. to the square inch for three or four hours. The alkaline silicate so obtained is then discharged, and cooled down. It is then ready for mixing with the soap paste in the boiler or pan, before the latter has become cold.

* FRANKLAND and JAPP, "Inorganic Chemistry," p. 466.

† WATT, "Art of Soap-making," p. 31.

WAY'S PROCESS.—The following is the description of this method as given in the specification of the patent :—

“I put into a suitable pan, heated by steam or in any convenient manner, a quantity of caustic alkaline lye (potash, or soda, or both, as the case may be) of about 18° Tw., so that the silica solution when made shall have a gravity as nearly 36° as possible, and, having raised this lye to the boiling point, I add by degrees the rock or clay” (found in Surrey, and containing sometimes as much as 70 per cent. of silica), “either in small pieces or ground to powder, until the alkali has taken up as much silica as it will dissolve. The heat is now withdrawn, and the undissolved earthy matter is allowed to settle. The clear liquor is run off, and a fresh quantity of water is added to the sediment to wash out further portions of soluble matter. The liquors so obtained are solutions of alkaline silicates. The quantity of rock or clay required will vary with the percentage of soluble silica which it contains. I find it necessary for every 31 parts of actual soda, or 53 parts of carbonate of soda rendered caustic, to employ as much of the rock or clay as contains 78 parts of soluble silica.

“I produce similar alkaline silicates from the rock or clay by gently heating it in a furnace with alkalies or alkaline carbonates. In this case, combination of the materials and production of the alkaline silicates takes place at a temperature much below that which is necessary when other forms of silicious matter are used, and, though preferring the method formerly mentioned for the treatment of the rock or clay, the one last described may be employed. The alkaline silicate is dissolved out from the furnaced materials by water or alkaline lye.

“I prefer, in either case, to saturate the alkali as fully as possible with silica; but this is not absolutely necessary. The silicates so produced are more suitable for the soap-

maker, for the following, amongst other, reasons:—
 (1) They are more economically produced; (2) The caustic property of the alkali contained in them is more perfectly neutralized; (3) They contain no iron, alumina, or other matter injurious to the soap; (4) The soap produced by them is therefore of superior quality, as well as cheaper.

“The alkaline silicate produced by either of these processes may be employed in any of the modes now used by soap-makers in incorporating the silicates of the alkalis with soap.”

Sodium Aluminate ($\text{Na}_2\text{Al}_2\text{O}_4^*$).—BONAMY, of St. Germain, near Paris,† seems to have first suggested the use of this material in the fabrication of soap.

The two chief substances from which sodium aluminate is prepared are *bauxite* and *cryolite*. *Bauxite* ($(\text{AlFe})_2\text{O}_3\text{H}_4$) is an aluminate of iron. This is calcined with soda-ash, and the resulting sodium aluminate is separated from the iron oxide by lixiviation. The dry commercial salt has the following composition:—

| | | | | | | | | |
|----------------------|---|---|---|---|---|---|---|----------|
| Soda | . | . | . | . | . | . | . | 43 parts |
| Alumina | : | : | : | : | : | : | : | 48 „ |
| Water and impurities | . | . | . | . | . | . | . | 9 „ |
| | | | | | | | | <hr/> |
| | | | | | | | | 100 „ |

Cryolite ($6\text{NaF}.\text{Al}_2\text{F}_6$) is a double fluoride of sodium and aluminium. From this the soap-maker may prepare his own aluminate, either by boiling the finely powdered mineral with lime, when insoluble calcium fluoride is formed and the alumina is dissolved in the excess of soda, or by calcining the mixture of cryolite and lime in a reverberatory furnace and afterwards lixiviating.

* ROSCOE and SCHORLEMMER, “Treatise on Chemistry,” vol. ii. pt. i. p. 445.

† “Polytech. Centralblatt,” 1865, s. 1452.

Natrona refined saponifier is the name under which the Pennsylvania Salt Manufacturing Company of Natrona, U.S.A., send out, in boxes, a white dry powder prepared from cryolite, and having, according to DUSSAUCE,* the following composition :—

| | |
|-------------------|----------|
| Soda | 44 parts |
| Alumina | 24 „ |
| Water | 32 „ |
| | <hr/> |
| | 100 „ |

Water.—The character of the water employed in soap-works is not a matter of indifference. Hard waters should be avoided, or softened before use, as the salts of lime and magnesia form *insoluble* soaps in the pan, and not only waste the fatty matters, but interfere with the appearance of the finished product. Suspended impurities may be removed by subsidence or filtration.

PRELIMINARY TREATMENT OF RAW FATTY MATERIALS.

Many soap-makers *render*, or clarify, their own fatty materials, insuring in this way greater uniformity in the purity of their goods.

I. Rendering Animal Fats.

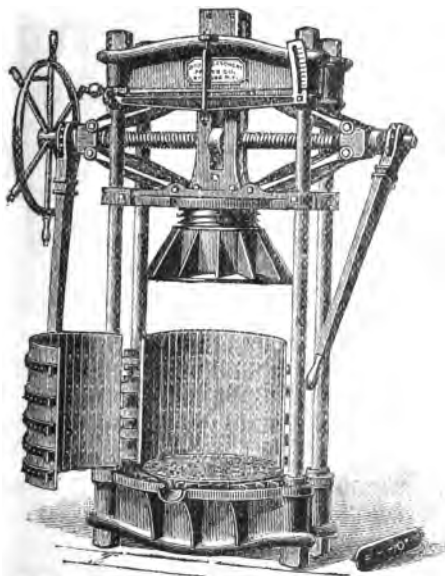
1°. DRYING AND MINCING.—The rough fats are hung up to dry in a well-aired room, and are then minced, either, as in large establishments, by steam-driven machinery or, as in small works, by a lever-knife fixed upon a table.

2°. BOILING.—On a small scale this is done in an *open* boiler or copper. It is essential that the fire should only come in contact with the bottom of the vessel, to avoid the risk of burning and darkening the fat. A quantity of pre-

* “General Treatise on the Manufacture of Soap,” p. 750.

viously rendered fat is first put into the boiler, and afterwards the minced tallow to be operated upon; when the first is melted, the entire contents are stirred together till the whole of the fat is extracted. When this is accomplished, the melted fat is removed, and, after passing it through a sieve, such as a wicker, or wire basket, or brass-wire sieve, and allowing it to rest for some time to deposit further im-

FIG. 2.



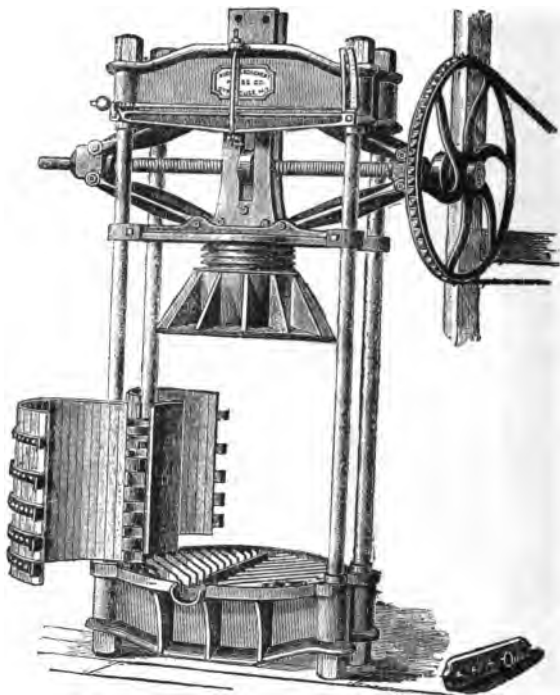
purities, it is finally, before solidification commences, distributed into store casks, or, if required for immediate use, conveyed direct to the soap pan.

The solid residue, called *greaves* or *cracklings*, is subjected to heat and pressure, and a further quantity of fat is obtained.

A convenient form of press for this purpose, made by the Boomer and Boschert Press Company, Syracuse, N.Y., is shown in Figs. 2 and 3.

The hoop is composed of a cast-iron section bolted to the base of the press, to which are hinged two doors completing

FIG. 3.



the circle. These doors are composed of wrought-iron bands to which are riveted the perpendicular staves, with a space of about $\frac{1}{8}$ th inch between each. The ends of the bands are turned outwards and a steel clamp slipped over them, locking them securely together. The base has ribs cast on

the upper surface, over which, inside the hoop, is placed a plate perforated with holes. After being pressed, the clamp is removed and the doors swung open (see Fig. 3), leaving the cracklings free for removal, and avoiding the heavy labour connected with the ordinary form of hoop. A patent pressure indicator is attached, and shows the amount of pressure being put upon the scrap. A cast follower, attached by a heavy screw to the platen, may be raised or lowered, to suit the amount of material in the hoop, and obviates the necessity of wood-blocking.

It is desirable to render separately the various kinds of crude fats, so as to secure uniformity in the quality. The fats also should be tolerably fresh, otherwise the rendered products are apt to be rancid and discoloured.

As a *maximum* product which is seldom attained,* beef suet yields 95 per cent. of tallow and 2 per cent. of refuse, mutton suet 91 per cent. and 4.5 per cent. of refuse.

The chief objections to this method are:—(1°) The difficulty of keeping the heat uniform throughout; (2°) The cellular tissue is not thoroughly broken up, and becomes so hard that the subsequent action of the press fails to squeeze out the whole of the retained fat; (3°) The extremely objectionable odours evolved.

Other means have, therefore, been devised, such as the use of steam instead of the open fire; the more effectual breaking up of the fatty cells by mechanical power, or by D'ARCET's dilute sulphuric acid treatment; the employment of a hood fitted with a pipe to convey the vapours through the furnaces; and the use of steam-tight cylinders in place of the open boiler.

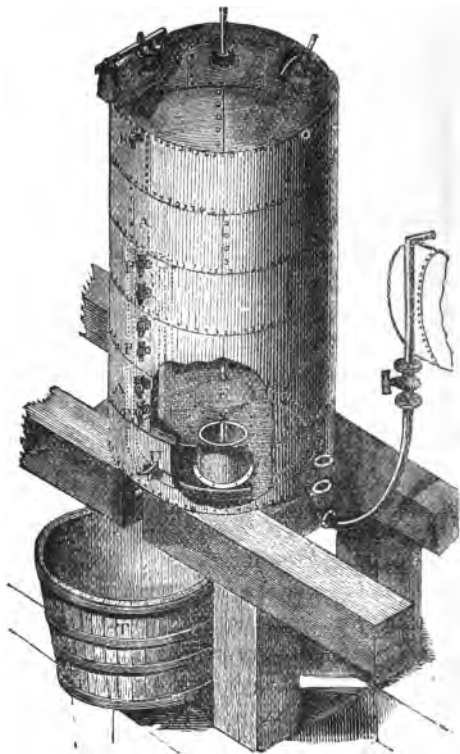
In D'ARCET's method the crude fat is boiled by steam with about one-fourth its bulk of water, acidulated with

* RICHARDSON and WATTS, "Technology," vol. i. pt. ii. p. 423.

2-3 per cent. of sulphuric acid, in an open, or loosely covered lead-lined vessel. The fats so rendered are whiter than those purified by the older method.

A very effective steam-tight cylinder is shown in Fig. 4,

FIG. 4.



which is highly spoken of by MORFIT. Its capacity is from 1200 to 1500 gallons, and it is made of strong iron, or boiler-plates riveted together. The height of the cylinder is two and a half times greater than the diameter. The

method of working the apparatus is as follows :*—The false bottom being arranged in its place and the discharging hole closed up, the cylinder is filled through the man-hole with the rough tallow, or lard material, to within $2\frac{1}{2}$ feet of the top. This done, the man-plate, *κ*, is securely fitted into the hole, *η*, and steam let on from an ordinary boiler through the foot-valve into the perforated pipe, *c*, within the tank. The weight on the valve is set at the required pressure, and, during the steaming, the state of the contents is frequently tested by opening the test-tap, *r*. If the quantity of condensed steam is too great, it will be indicated by the ejection of fatty matters. In such case the regulating cock, *x*, must be opened, and the condensed steam drawn off into the receiving tub, *t*, until the fatty matter ceases to run from the tap, *r*. After ten to fifteen hours' continued ebullition, the steam is shut off, and the excess of uncondensed steam in the cylinder allowed to escape through *r* and the safety valve. After sufficient repose, the fatty matter separates entirely from water and foreign admixture, and forms the uppermost stratum. It is drawn off through the cocks, *p p*, in the side of the tank, into coolers of ordinary construction. After the fat has been thus removed from the cylinder, the cover, *f*, is raised by means of the rod, *g*, from the discharging hole, *ε*, and the residual matters at the bottom fall into the tub, *t*. If, on inspection, any fatty matters are found in this tub, they should be returned to the tank with the next charge.

The pressure of steam may be from 50 to 60 or 70 lb. per square inch, but a pressure beyond this is apt to injure the tallow, and cause a proneness to decomposition.

It is stated that the steam-cylinder process extracts about 12 per cent. more tallow, or 6 per cent. more lard, than any other method.

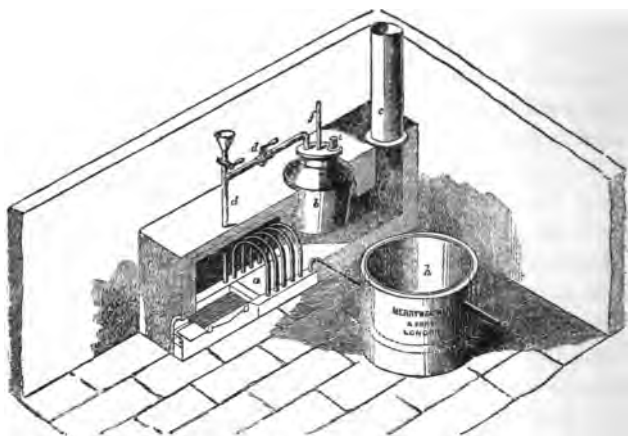
* MUSPRATT'S "Chemistry" 1. 216.



An illustration is given in Fig. 5 of Merryweather & Sons' patent superheating apparatus for rendering fat by steam.

The following advantages are said to be obtained by this apparatus over the method of melting by fire-heat :—(1) The

FIG. 5.



a is the superheater, formed of wrought-iron lap-welded tubes, set in a brick oven with ordinary furnace and bars, as shown.

b is the steam boiler, the water in which is kept to its proper level by means of a self-regulating feed.

c is the chimney.

d d are the pipes and cocks connecting the boiler with the superheater.

g is the pipe which connects the superheater with

h, the fat-pan, which is set in brickwork, to prevent loss of heat.

In cases where it is essential to destroy the obnoxious fumes arising from the melting process, a patent cover is provided for the fat-pan, *h*.

copper is not injured by local heat, and will last for many years ; (2) There is little risk of burning the tallow or fat during the heating ; (3) The pan costs 50 per cent. less than those ordinarily used ; (4) The heat can be instantly

checked, thus preventing the danger of boiling over; (5) No risk of accidents from fire; (6) Economy of fuel.

Glue fat often contains 2 or 3 per cent. of lime. The presence of lime can be easily ascertained by stirring up a little of the melted fat with a solution of ammonium oxalate or of oxalic acid. If no lime is present, the liquid under the fat remains clear. Lime causes the fats, when saponified, to become spongy, in which state its separation can only be with difficulty accomplished by means of common salt. It is therefore desirable, before using such fat for soap-making, to treat it with dilute sulphuric acid.

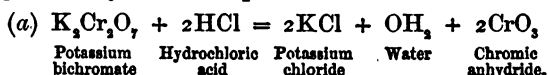
II. Bleaching.

There are various ways of decolorizing the fatty matters used in soaperies. The BICHROMATE METHOD OF WATT, which is very generally followed for palm oil, is as follows:—

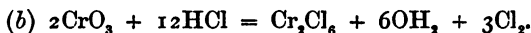
1°. The oil or fat is melted in a copper, and the dregs are allowed to subside.

2°. The oil, now at about the temperature of 120° to 130° F., is run off from the dregs into another vessel, and treated with a mixture of potassium bichromate and hydrochloric acid. For 1 ton of fat the following quantities are used:—25 lb. of the bichromate, dissolved in boiling water, are first poured into the melted oil, with constant stirring, and, after thorough admixture, 60 lb. of hydrochloric acid are added, and the mixture constantly stirred till it acquires a uniform greenish tint, or till the fat is sufficiently decolorized. A little more of the bleaching materials may be added if necessary. After this the whole is well washed with hot water and allowed to settle. In about twelve hours the *green liquor*, as it is called, containing chromic chloride and hydrochloric acid, may be drawn off, and the bleached oil removed. The reaction which takes place may,

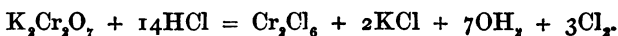
for the sake of simplicity, be represented in two stages, though actually both take place at once—



The chromic anhydride thus liberated is at once attacked by more hydrochloric acid, with the production of chromic chloride and free chlorine—



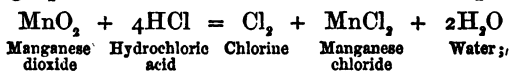
This result may be represented by a single equation, as follows:—



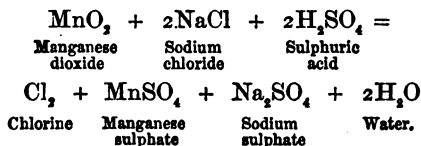
The theoretical quantities are as nearly as possible equal proportions of the acid and bichromate, but, in practice, to insure completeness, a large excess of the acid is used—two or three times the weight of the bichromate.

NITRIC ACID was at one time employed for bleaching palm oil. The objections to its use are that it bleaches imperfectly, and to a great extent destroys the peculiar and characteristic violet odour.

CHLORINE, generated from manganese dioxide and hydrochloric acid, or from manganese dioxide, sodium chloride, and sulphuric acid, has been much used for bleaching this oil. The ingredients are mixed with the melted fat. The following equations show the reactions which take place:—



or



The efficacy of chlorine as a bleaching agent is con-

sidered to depend upon its affinity for hydrogen. *Dry* chlorine gas will not bleach, but, if water be added, the action at once commences. The chlorine takes hydrogen from the water, and liberates oxygen in the *nascent* state, which, in this condition, readily unites with vegetable colouring matters to form colourless compounds. The immediate bleaching agent is, therefore, oxygen.

Another way of employing chlorine is in the form of chloride of lime, or *bleaching powder* (calcic chloro-hypochlorite, $\text{Ca}(\text{OCl})\text{Cl}$, or, according to another view, a mixture of calcium chloride, CaCl_2 , and calcium hypochlorite, CaCl_2O_2). The gently heated oil is stirred for some time with about 1 per cent. of good chloride of lime previously made into a milky liquor by trituration with water; about $1\frac{1}{2}$ per cent. of sulphuric acid diluted with twenty times its weight of water is then added, and the agitation renewed and maintained for at least two hours; it is, lastly, well washed with steam or hot water.

The objection to the use of chlorine, or of chlorides, is that, although the colouring matters are readily destroyed in the way mentioned, the chlorine acts injuriously upon the fat, probably on account of its affinity for its hydrogen, and is very apt to produce a brownish tint.

DUNN's method is effective and simple, and applicable to palm and other oils. The fat or oil is heated to 180° – 200° F., and then air is forced through the melted materials, by means of a blowing apparatus, in numerous small streams. The vat in which the operation is conducted is furnished with a hood, communicating with a chimney, to convey away the unpleasant vapours given off. When the fat is sufficiently bleached, it is washed with steam or hot water.

Tallow.—Commercial tallows very often require further purification, especially before use in candle-making. The

following has been recommended as a good process for bleaching tallow :*—1°. About 50 lb. of caustic-soda lye are placed in a clean boiler, and the steam turned on. Salt is then added to the lye till it shows a density of 25° to 28° B. 2°. 300 lb. of tallow are now placed in the boiler and heated to boiling. It is allowed to boil up about 1 or 2 inches only, and then left for from three to five hours to clarify. 3°. At the end of this time the upper saponified layer is ladled off; the purer tallow is removed and passed through a hair sieve into a clean vessel, until the lower saponified layer is reached. The residue in the boiler, consisting of saponified fat and lye, together with the upper layer, may be used in the preparation of curd soap. 4°. The boiler having been thoroughly cleansed, about 30 to 35 lb. of water with $\frac{3}{4}$ lb. to 1 lb. of alum are placed therein and heated to boiling. To this solution the fat is added, and the whole is boiled for about fifteen minutes, till the filth has disappeared from the fat. Transferred after this to another vessel, it is left to itself again for from three to five hours. 5°. The fat obtained from this operation is again placed in the boiler and heated to the temperature of 170° to 200° C. In this last treatment the fat becomes snow-white and fit for use. The steam must be turned off as soon as the slightest disagreeable odour is emitted, whether the temperature be 150° or 170° C., otherwise the fat will again turn dark.

Freshly rendered, sweet fat is most readily bleached, and may be heated quite high. Still, the fat used should not be too fresh, or there will be risk of saponifying the whole of the 300 lb. without leaving any to bleach.

Tallow which has been treated in this way, when used in toilet soaps, gives them a white colour and agreeable odour.

* "Oil Trade Review," Oct. 1884.

Such tallow is also well adapted for candle-making, as it becomes exceedingly hard.

III. Bone-boiling.

This is an operation sometimes performed on the soapery premises.

The bones are first sorted out, and those which are unsuitable for the manufacture of articles of bone are crushed by suitable machinery. The bones are placed in boxes, or cradles, made of iron bars, or of perforated iron, and lowered in this way into the boilers. In these vessels the bones are boiled in water heated by steam. The steam is injected as long as any appreciable quantity of fat gathers on the surface of the water. This is then skimmed off, and, without further purification, is available for soap-making. The extraction of fat in this way is always imperfect, about 6 per cent. of the fat remaining behind in the bones. On an average, from 2 to 4 per cent. of grease is obtained, or from select fatty bones about 6 per cent.

SELTSAM's method.*—By this means bones of all kinds may be extracted, yielding double, or even triple, the quantity of fat obtained by the above process, and of superior quality. The apparatus consists of a strong wrought-iron cylinder of about 370 cubic feet capacity. The cylinder is filled with bones through a man-hole at the top, except a space, 8 inches in depth, at the bottom, separated from the bone-chamber by a perforated false bottom. In this space there is placed a coil of pipe, through which steam can be passed. Petroleum spirit is let into the bottom of the chamber, till it stands about 18 inches high. This spirit, boiled and vaporized by the steam coil, gradually rises amongst the bones, expelling the air, and, after about an

* English patent 2976—1881; "Jour. Soc. Chem. Ind." 1882, p. 112.

hour, there is perceptible at the man-hole a smell of petroleum. The man-hole is then closed, and the remaining air and aqueous vapour pass through a pipe at the top of the cylinder through the condenser. In a little while, petroleum spirit alone runs out, and then the cock is shut, and pressure allowed to accumulate in the bone-chamber to the extent of about 22 lb. above atmospheric pressure. Steam is then shut off, and the whole left to cool slowly. After a sufficient interval, or next morning, steam is again let into the lower coil, till a pressure of $7\frac{1}{2}$ lb. is attained. By this means most of the petroleum spirit is again vaporized among the bones, and all the fat is collected below the diaphragm. The fat is next drawn off and introduced into the still. Steam is again let into the cylinder, the cock into the condenser opened, and the petroleum spirit forced out into the latter. The condensed petroleum spirit and water are received into a covered tank. The water is allowed to siphon off through a pipe at the bottom of the tank, and the spirit, as it collects on the surface of the water, passes through a pipe at the side to the petroleum-spirit tank. When no more petroleum spirit comes through the condenser, the steam is shut off, and the bones emptied through a man-hole just above the false bottom. The grease, in the meanwhile, is heated in the still, the spirit distilled off, and run back into the tank. The grease is then run out, and will be found to be sweet.

After this treatment the bones are almost chemically free from fat, and may be crushed and calcined for charcoal. The smaller fragments, instead of being calcined, may be used for the production of glue, and the mineral matter remaining may be sold or made into superphosphate.

CHAPTER III.

HYDROMETERS AND LYE-TESTING.

THEORY OF THE HYDROMETER.—It is one of the laws of hydrostatics that a body immersed in a fluid is buoyed, or pressed upwards by a force exactly equal to the weight of the bulk of the fluid which it displaces. Hence, if the body float, the weight of the volume of liquid which would fill the space occupied by the portion immersed is exactly equal to the entire weight of the body itself. Upon this simple fact the whole theory of hydrometry rests.

Hydrometers may be constructed of glass, silver, copper, brass, or German silver. The great economy of glass, its perfect cleanliness, resistance to corrosion, incapability of fraudulent change of form or weight, and facility of manufacture, are qualities possessed to the same extent by no other known substance. The chief objection to the glass hydrometer is its fragility, and this often renders metallic instruments preferable. Metallic hydrometers should be gilded with gold or platinum, so as to be rendered incapable of corrosion by liquids generally.

A hydrometer, whether of glass or metal, is simply a hollow bulb, carrying a graduated stem above, and having, below, a counterpoise, or ballast, to preserve it in stable equilibrium when in a vertical position.*

* For details as to the mode of construction, see "Reports from

In order that a hydrometer may be convenient and useful, it is not necessary that it should show *specific gravities*, and it is, perhaps, not desirable that it should do so, for it is easier to remember, for instance, that a solution has the density of 20° Baumé, than that its specific gravity is 1.1515. Hence we find that in France, though BRISSON brought forward an instrument reading specific gravities, and succeeded in causing a violent opposition to BAUMÉ's hydrometer, the latter came into general use, not merely in France, but also in our own and in other countries of Europe. But no doubt the difficulty of construction, and consequent high price, of an instrument based on rigid scientific principles also contributed to BRISSON's hydrometer being superseded by one which, though constructed on arbitrary rules, is simple, easy of construction and use, and economical.

The forms of hydrometer chiefly used by soap-makers are BAUMÉ's on the Continent and TWADDELL's in England.

BAUMÉ'S HYDROMETER.—For fluids *lighter* than water, BAUMÉ invented a spirit hydrometer (*pèse-esprit*, Fr.; *Brantweinmesser*, Ger.), and for fluids *heavier* than water his hydrometer for acids and saline and saccharine solutions (*pèse-acide*, *pèse-sel*, or *pèse-sirop*, Fr.; *Säuremesser*, *Salzspindel*, or *Zuckermesser*, Ger.). These instruments are entirely distinct, and form no part of a common system, being constructed on different bases. The degrees of one are not equal to those of the other, and the zero point of the *pèse-esprit*, determined by a solution containing 10 per cent. of common salt, corresponds in the *pèse-acide* to the density of pure water.

The *pèse-esprit* is constructed by immersion in a 10 per

the Secretary of the Treasury of Scientific Investigations in relation to Sugar and Hydrometers," by Prof. R. S. McCULLOCH (Washington, 1848).

cent. solution of common salt to obtain the zero point. Then it is floated in water to determine another point, which BAUMÉ called 10° . The interval is graduated equally, and the scale is extended by laying off repeatedly, with a pair of dividers, corresponding intervals on the stem.

The zero point of the *pèse-acide* is given by the surface of the distilled water in which it floats. Immersion in a 15 per cent. solution of common salt fixes the point which is to be marked 15° upon the scale. Hence, 1° B. = 1 per cent. of salt. Degrees beyond 15° are determined by the same process of extension employed for the *pèse-esprit*.

Results of Different Observers, obtained by Experimental Comparison of BAUMÉ'S Hydrometer with Specific Gravities at 54.5° F.

(*Pèse-acide*, or Hydrometer for Liquids heavier than Water.)

| Degrees B. | FRANCŒUR. | DELEZENNES. | GILPIN. |
|------------|-----------|-------------|---------|
| 0 | 1.0000 | 1.0000 | 1.000 |
| 3 | 1.0201 | 1.0209 | 1.020 |
| 6 | 1.0411 | 1.0448 | 1.040 |
| 9 | 1.0630 | 1.0687 | 1.064 |
| 12 | 1.0857 | 1.0937 | 1.089 |
| 15 | 1.1095 | 1.1200 | 1.114 |
| 18 | 1.1343 | 1.1475 | 1.140 |
| 21 | 1.1603 | 1.1764 | 1.170 |
| 24 | 1.1875 | 1.2068 | 1.200 |
| 27 | 1.2160 | 1.2389 | 1.230 |
| 30 | 1.2459 | 1.2727 | 1.261 |
| 33 | 1.2773 | 1.3083 | 1.295 |
| 36 | 1.3103 | 1.3333 | 1.333 |
| 39 | 1.3451 | 1.3861 | 1.373 |
| 42 | 1.3818 | 1.4285 | 1.414 |
| 45 | 1.4206 | 1.4735 | 1.455 |
| 48 | 1.4615 | 1.5217 | 1.500 |
| 51 | 1.4951 | 1.5730 | 1.547 |
| 54 | 1.5510 | 1.6279 | 1.594 |
| 57 | 1.6000 | 1.6868 | 1.659 |
| 60 | 1.6522 | 1.7501 | 1.717 |
| 63 | 1.7070 | 1.8184 | 1.779 |
| 66 | 1.7674 | 1.8922 | 1.848 |
| 69 | 1.8313 | 1.9721 | 1.920 |
| 70 | 1.8537 | 2.0003 | |

The differences in these results are generally accounted for by differences in the accuracy of the hydrometers used. The following is the table generally used for converting the reading of BAUMÉ's degrees into specific gravities :—

Comparison of the Degrees of BAUMÉ's Hydrometer with the Real Specific Gravities at 54.5° F. (FRANCEUR).

(For Liquids heavier than Water.)

| Degrees. | Specific Gravity. | Degrees. | Specific Gravity. | Degrees. | Specific Gravity. | Degrees. | Specific Gravity. |
|----------|-------------------|----------|-------------------|----------|-------------------|----------|-------------------|
| 0 | 1.0000 | 20 | 1.1515 | 39 | 1.3451 | 58 | 1.6170 |
| 1 | 1.0066 | 21 | 1.1603 | 40 | 1.3571 | 59 | 1.6344 |
| 2 | 1.0133 | 22 | 1.1692 | 41 | 1.3694 | 60 | 1.6522 |
| 3 | 1.0201 | 23 | 1.1783 | 42 | 1.3818 | 61 | 1.6705 |
| 4 | 1.0270 | 24 | 1.1875 | 43 | 1.3945 | 62 | 1.6889 |
| 5 | 1.0340 | 25 | 1.1968 | 44 | 1.4074 | 63 | 1.7070 |
| 6 | 1.0411 | 26 | 1.2063 | 45 | 1.4206 | 64 | 1.7273 |
| 7 | 1.0483 | 27 | 1.2160 | 46 | 1.4339 | 65 | 1.7471 |
| 8 | 1.0556 | 28 | 1.2258 | 47 | 1.4476 | 66 | 1.7674 |
| 9 | 1.0630 | 29 | 1.2358 | 48 | 1.4615 | 67 | 1.7882 |
| 10 | 1.0704 | 30 | 1.2459 | 49 | 1.4758 | 68 | 1.8095 |
| 11 | 1.0780 | 31 | 1.2562 | 50 | 1.4902 | 69 | 1.8313 |
| 12 | 1.0857 | 32 | 1.2667 | 51 | 1.4951 | 70 | 1.8537 |
| 13 | 1.0935 | 33 | 1.2773 | 52 | 1.5000 | 71 | 1.8765 |
| 14 | 1.1014 | 34 | 1.2881 | 53 | 1.5353 | 72 | 1.9000 |
| 15 | 1.1095 | 35 | 1.2992 | 54 | 1.5510 | 73 | 1.9241 |
| 16 | 1.1176 | 36 | 1.3103 | 55 | 1.5671 | 74 | 1.9487 |
| 17 | 1.1259 | 37 | 1.3217 | 56 | 1.5833 | 75 | 1.9740 |
| 18 | 1.1343 | 38 | 1.3333 | 57 | 1.6000 | 76 | 2.0000 |
| 19 | 1.1428 | | | | | | |

TWADDELL'S HYDROMETER.—This hydrometer is a good deal used in this country by soap-makers. The instrument is so graduated that the real specific gravity can be deduced easily from the hydrometer degree by multiplying the latter by 5 and adding 1000—the sum is the specific gravity, water being 1000. Thus 10° Tw. $\times 5 + 1000 =$ sp. gr. 1050, or 1.05; 15° Tw. $\times 5 + 1000 =$ sp. gr. 1075, or 1.075—or, in other words, 1° Tw. is equal to five degrees of gravity.

In hydrometric determinations the *temperature* of the sample must be carefully attended to, as fluids expand as the temperature is increased. Hydrometer tables used in

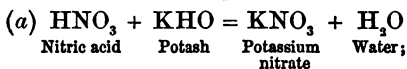
England are generally adjusted to the standard temperature of 60° F., but, when tables giving the correction for variation of temperature are not accessible, the fluids to be examined must be brought, by cooling or heating, to this temperature.

Unless lyes are made from pure alkalies, the indications of the hydrometer do not accurately give their strength. This can then be only correctly determined by the process of *alkalimetry*, in the way described in textbooks on practical chemistry.

CHAPTER IV.

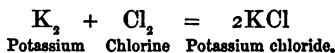
SAPONIFICATION.

WHEN a solution of an alkali, such as soda or potash, is gradually added in the cold to an acid, such as nitric acid, the intensity of the acidity of the latter gradually diminishes till at length a point is reached when the mixture ceases to affect either blue or red litmus-paper. We say that the acid has been *neutralized* by the alkali added—it has ceased to be *free* acid, having entered into combination with the soda or potash to produce sodium or potassium nitrate. Such a compound we call a *salt*. The reaction which takes place is represented by the equation—

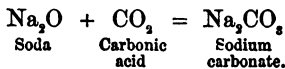


but there are several other ways in which salts may be formed—*e.g.* :

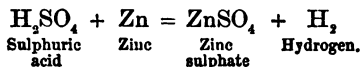
(b) Union of *elements*—



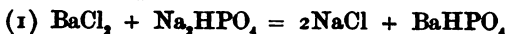
(c) Union of *acid* and *basic oxide*—



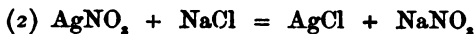
(d) Action of *acid* on a *metal*—



(e) *Double decomposition* between two salts—

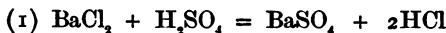


| | | | |
|--------------------|-----------------------------|--------------------|------------------------------|
| Barium chloride | Acid sodium phosphate | Sodium chloride | Acid barium phosphate. |
|--------------------|-----------------------------|--------------------|------------------------------|

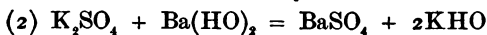


| | | | |
|-------------------|--------------------|--------------------|--------------------|
| Silver nitrate | Sodium chloride | Silver chloride | Sodium nitrate. |
|-------------------|--------------------|--------------------|--------------------|

(f) *Displacement* of the acid or base in a salt by another acid or base—

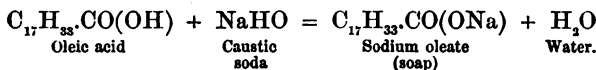


| | | | |
|--------------------|-------------------|--------------------|-----------------------|
| Barium chloride | Sulphuric acid | Barium sulphate | Hydrochloric acid. |
|--------------------|-------------------|--------------------|-----------------------|



| | | | |
|-----------------------|-------------------|--------------------|-----------------------|
| Potassium sulphate | Barium hydrate | Barium sulphate | Potassium hydrate. |
|-----------------------|-------------------|--------------------|-----------------------|

The simplest example of soap-making, which, however, is not strictly *saponification*, is afforded by the union under the influence of heat of a *free* fatty acid, such as oleic acid, with an alkali. This may be thus represented:—



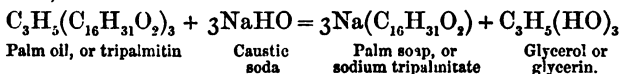
| | | | |
|------------|-----------------|-------------------------|--------|
| Oleic acid | Caustic soda | Sodium oleate (soap) | Water. |
|------------|-----------------|-------------------------|--------|

It will be at once seen that the fatty acid and the soda have united so as to produce a *salt*, just as the nitric acid and potash in example (a) above.

The oils and fats, however, used by the soap-maker are not *acids*, and the explanation of their saponification is therefore not quite so simple. CHEVREUL, by his researches, extending from 1813 to 1823,* demonstrated the true nature of the animal and vegetable fixed oils and fats, and to him we are indebted for the right understanding of what takes place when a fatty body is saponified. He showed that fats are compound bodies, formed from an organic *base*, glycerin (*glycerol*), and various fatty *acids*, thus constituting true *salts*. Thus, mutton and beef fat are chiefly

* "Recherches chimiques sur les Corps gras" (Paris, 1823).

glyceryl + stearic acid; palm oil is chiefly *glyceryl + palmitic acid*; olive oil, *glyceryl + oleic acid*. These compounds are neutral salts, *etheral salts* or *glycerides*. The glyceride of stearic acid is also called *stearin*; that of palmitic acid, *palmitin*; and that of oleic acid, *olein*. Olein is liquid, and the other two glycerides are solid, at ordinary temperatures. Stearin has the highest melting point. Hence, the softest fats are those which contain most olein, and the hardest those which contain most stearin. Mutton and beef tallows and lard are rich in stearin. Palm oil is rich in palmitin. Sperm and cod-liver oil contain a large proportion of olein. The fatty acids in these glycerides have less affinity for the glyceryl than they have for alkalies. Hence, when a fat is heated with an alkali, and *saponified*, the basic constituent, or *glyceryl* (as the radical C_3H_5 is termed), is displaced by the alkali, which unites with the fatty acid, or acids, previously combined with the glyceryl, and a new salt (*soap*) is formed, as in the last example of the formation of a salt (*f*, 2) given above, thus :—

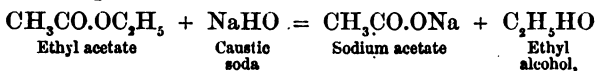


Actually what takes place in saponification is not so simple, because each of the various oils and fats contains *several* glycerides, either mixed or in chemical combination.* Thus it follows that the potassium or sodium salts resulting from saponification must also contain several fatty acids. Ordinary hard soap, for instance, is a mixture of sodium stearate, palmitate, and oleate.

The production of soap by the combination of oleic acid with an alkali, or of resinous acids with an alkali, is not strictly *saponification*, which term is, scientifically, confined

* "Churchill's Technological Handbooks" — "Oils and Varnishes," p. 12; BELL's "Chemistry of Foods," pt. ii. p. 44.

to the decomposition of *etheral* salts, such as the ordinary fats, by an alkali. Hence, the term saponification is extended to include the decomposition of any *etheral* salt by an alkali. For instance, when *ethyl acetate* (an *etheral* salt) is decomposed into acetic acid and alcohol, saponification takes place, thus—



although sodium acetate is never called a soap.

The production of soap is not, like its decomposition by an acid, a *momentary* process, but there are a number of stages in the operation, each occupying a considerable length of time, from the first mixing of the fat with the alkali, when a milky liquid is produced, to the point when the union between the alkali and the fatty acids is complete. It has been said that acid salts are first produced, and that these hold the remainder of the fat in a state of solution or division until it also is able to combine with the alkali, and transform the *acid* into *neutral* salts ready for use as soap. This reaction may be easily observed if the fat is boiled with one-half the requisite quantity of alkali; the whole of the oil is at length dissolved, but the solution becomes turbid on cooling, and, when diluted with water, and boiled, unsaponified fat separates, which had been retained in the fluid only by the stearate, palmitate, &c., of the alkali formed.*

DECHAN and MABEN,† however, are of opinion that, as the fatty acids are monobasic, the formation of acid salts cannot take place, but that *basic* oleates, stearates, and palmitates are first formed, and that, as saponification pro-

* MUSPRATT'S "Chemistry," ii. 875; RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 638.

† "Pharm. Journ." June 13, 1885.

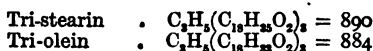
ceeds, more of the alkali enters into combination, till, finally, if the operation is properly conducted, a neutral compound results.

The following tables include the chief fatty acids derived from natural fats :—

Acetic, or $C_nH_{2n+1}.CO(OH)$ Series.

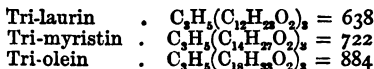
| Name of Acid. | Formula. | Equivalent. | Melting Point, C. | Source. |
|------------------------|-----------------------|-------------|-------------------|--|
| Butyric . | $C_3H_7.CO(OH)$ | 88 | Below -20° | Butter. |
| Caproic . | $C_5H_{11}.CO(OH)$ | 116 | - 2 | |
| Enanthic . | $C_7H_{13}.CO(OH)$ | 130 | - 10.5 | |
| Caprylic . | $C_7H_{15}.CO(OH)$ | 144 | 14 | |
| Capric, or ruric . | $C_9H_{19}.CO(OH)$ | 172 | 30 | Butter, cocoa-nut oil. |
| Cocinic . | $C_{10}H_{21}.CO(OH)$ | 186 | 35 | Cocoa-nut oil, spermaceti, Chaulmoogra oil. |
| Lauric . | $C_{11}H_{23}.CO(OH)$ | 200 | 40.5 | Cocoa-nut oil, spermaceti, laurel butter or bay-fat. |
| Myristic . | $C_{13}H_{27}.CO(OH)$ | 228 | 53.8 | Muscat fat, Dika bread, cocoa-nut oil, spermaceti. |
| Palmitic . | $C_{15}H_{31}.CO(OH)$ | 256 | 62 | Palm oil. To some extent in most animal fats. |
| Stearic . | $C_{17}H_{35}.CO(OH)$ | 284 | 69.2 | Tallow, suet, lard, and in most fats. |
| Arachidic . | $C_{19}H_{39}.CO(OH)$ | 312 | 75 | Earth-nut oil. |
| Behenic, or benic . | $C_{21}H_{43}.CO(OH)$ | 340 | 76 | Ben oil. |
| Cerotic . | $C_{26}H_{53}.CO(OH)$ | 410 | 78 | Bees'-wax. |
| Melissic . | $C_{29}H_{59}.CO(OH)$ | 452 | 88 | " |

The proportion of tallow equivalent to $3\text{Na}_2\text{O}$ is similarly found to be 887—



$$1774 \div 2 = 887$$

and that of cocoa-nut oil, 748, thus:—



$$2244 \div 3 = 748$$

Calculating thus, the following proportions are obtained:*

| 100 lb. of | Require of | |
|----------------------------------|---|--|
| | Soda (100 % Na_2O). | Potash (100 % K_2O). |
| Tallow | 10.50 lb. | 15.92 lb. |
| Palm oil | 11.00 | 16.67 |
| Cocoa-nut oil | 12.43 | 18.86 |
| Oleic acid (tri-olein) | 10.52 | 15.95 |

As the percentage of available alkali at command is never 100, it is requisite to make a correction for the percentage available. If that were 60 per cent., then the amount of alkali to be employed for every 100 lb. of fat would be the above quantities increased in the proportion of 60 to 100; or if it contained 20 per cent. of available alkali, then the proportion would be five times the above; and so on.

The quantity of alkali necessary to saponify any fat may also be found experimentally by KOETTSTORFER's saponification method,† which, after the standard solutions have

* CRISTIANI, "Treatise on Soap and Candles," p. 154.

† For details of the process, see "Analyst," 1879, p. 106; or "Churchill's Technological Handbooks"—"Oils and Varnishes," p. 246.

been prepared, is simple, accurate, and rapid. The following are the figures obtained in this way by KOETTSTORFER, STODDART, ARCHBUTT, MOORE, HÜBL, ALLEN, and others:*

| Nature of Oil. | Percentage of † KHO for Saponification, or lb. KHO for 100 lb. Fat. | Saponification Equi- valent, or No. of Grammes of Oil or Fat saponified by One Equivalent in Grammes of any Alkali. |
|--------------------------------------|--|--|
| A. OLEINS— | | |
| Lard oil | 19.10 to 19.60 | } 285 to 296 |
| Olive oil | 19.10 to 19.60 | |
| Olive oil | 18.93 to 19.26 | |
| Almond oil (sweet) . | 19.47 to 19.61 | |
| Arachis oil | 19.13 to 19.66 | |
| Tea oil | 19.55 | |
| Sesamé oil | 19.00 to 19.24 | |
| Cotton-seed oil . . | 19.10 to 19.66 | |
| B. RAPE OIL CLASS— | | |
| Colza and rape oils . | 17.08 to 17.90 | } 313 to 330 |
| Rape oil | 17.02 to 17.64 | |
| Mustard-seed oil . . | 17.40 to 17.50 | |
| Cabbage-seed oil . . | 17.52 | |
| C. VEGETABLE DRYING OILS— | | |
| Linseed oil | 18.74 to 19.52 | } 286 to 300 |
| Poppy-seed oil . . . | 19.28 to 19.46 | |
| Hemp-seed oil . . . | 19.31 | |
| Walnut oil | 19.60 | |
| Niger-seed oil . . . | 18.90 to 19.10 | |
| D. MARINE OLEINS— | | |
| Cod-liver oil | 18.51 to 21.32 | } 250 to 303 |
| Menhaden oil | 19.20 | |
| Pilchard oil | 18.60 to 18.75 | |
| Seal oil | 18.90 to 19.60 | |
| Southern whale oil . | 19.31 | |
| Northern whale oil . | 18.85 to 22.44 | |
| Porpoise oil | 21.60 to 21.88 | |
| E. BUTTER CLASS— | | |
| Butter fat | 22.15 to 23.24 | } 241 to 253 209 to 255 |
| Cocoa-nut oil | 24.62 to 26.84 | |
| Palm-nut oil | 22.00 to 24.76 | |

* ALLEN, "The Analyst," 1886, p. 146.

† These numbers $\times .5535$ = percentage of soda (Na_2O — 100 per cent.), or lb. of soda required for 100 lb. of any of the fatty bodies.

SAPONIFICATION EQUIVALENTS—(continued).

| Nature of Oil. | Percentage of* KHO for Saponification, or lb. KHO for 100 lb. Fat. | Saponification Equi- valent, or No. of Grammes of Oil or Fat saponified by One Equivalent in Grammes of any Alkali. |
|------------------------------|---|--|
| F. STEARINS, &c.— | | |
| Lard | 19.20 to 19.65 | } 277 to 294 |
| Tallow | 19.32 to 19.80 | |
| Dripping | 19.65 to 19.70 | |
| Butterine | 19.35 to 19.65 | |
| Goose fat | 19.26 | |
| Bone fat | 19.09 to 19.71 | |
| Palm oil | 19.63 to 20.25 | |
| Cacao butter | 19.98 | |
| G. FLUID WAXES— | | |
| Sperm oil | 12.34 to 14.74 | 380 to 454 |
| Bottle-nose oil | 12.30 to 13.40 | 419 to 456 |
| H. SOLID WAXES— | | |
| Spermaceti | 12.73 to 13.04 | 432 to 441 |
| Bees'-wax | 9.20 to 9.70 | |
| Carnaüba wax | 7.90 to 8.51 | |
| Chinese wax | 6.50 | |
| I. UNCLASSED— | | |
| Shark-liver oil | 14.00 to 19.76 | 284 to 400 |
| Wool fat (suint) | 17.00 | 330 |
| Lanolin | 9.83 | 570.9 |
| Olive-kernel oil | 18.85 | 298 |
| Castor oil | 17.60 to 18.15 | 309 to 319 |
| Japanese-wood oil | 21.10 | 266 |
| Japan wax | 21.01 to 22.25 | 252 to 267 |
| Myrtle wax | 20.57 to 21.17 | 265 to 273 |
| Blown-rape oil | 19.80 to 20.40 | 275 to 284 |
| Colophony | 17.00 to 19.30 | 290 to 330 |

In the case of the glycerides, the saponification equivalent is one-third of the molecular weight, but in case of mono-atomic ethers, like those which essentially constitute sperm oil and bees'-wax, the saponification equivalent is identical with the molecular weight.

* See note † on p. 57.

CHAPTER V.

APPARATUS AND ARRANGEMENT OF THE FACTORY.

APPARATUS.

THE apparatus of a soap factory is of a simple kind, and may be arranged under the following heads:—

1°. *The lye tanks*, for the alkalies.

2°. *The pans*, for effecting the combination between alkali and fat.

3°. *Various appliances for working the product into commercial forms.*

1°. **Lye Tanks, or Vats.**—When the alkalies are causticized at the factory, the operation is performed in cast- or wrought-iron tanks, 6 or 7 feet broad, and 4 or 5 feet in depth, either furnished with a perforated false bottom, or having a coarse piece of matting placed over the plug-hole. From these tanks the lyes, of various strengths, are conveyed to the *reservoirs*. These may be, for convenience, placed at one end of the soap-pan series, and at a somewhat higher level, so that the lyes may be readily run, by means of a *shoot*, into the boilers, as required, or as N N N, Fig. 22, p. 78, or B B B, Fig. 23, p. 80.

If the alkalies are obtained by the soap-maker in the *caustic* state, their solution may be made in cast-iron or sheet-iron kettles.

For the finer qualities of soap, especially toilet soaps, for

which it is necessary to have a perfectly clear and colourless lye, it is advisable to have the vat or tank lined with lead.*

2°. **The Pans.**—In these the combination between the alkali and the fat is effected. They are variously termed *pans*, *coppers*, *caldrons*, *kettles*, or *boilers*, and they differ somewhat in construction, according to the process of soap-making adopted. Speaking generally, large coppers offer advantages over small ones in economy of labour, fuel, and lye. It will be convenient to consider the construction of the pans under the heads of the particular processes for which they are suitable. The chief methods followed may be classified thus:—

1. The *ordinary process (large-boiler process)*: the *open* boiling of an indefinite, *i.e.*, not *exactly* proportioned, mixture of fat and alkali.

2. Processes requiring the mixture of fat and alkali in *calculated proportions*:—

a. The *cold process (little-pan process)*.

b. *Boiling under pressure*.

c. *Open boiling*.

d. *Free-acid process*.

1. Open Boiling.

The pans are made either of cast or wrought iron—in small factories, often of cast iron, either in one piece, or in plates united together by iron cement; in larger factories, more frequently of wrought-iron plates riveted together. They are usually made with a flange at the rim, and above this rim is fixed the *curb*, which is often made of wood, well hooped with iron rings. Their capacity varies according to the quantity of soap to be made at each operation: some-

* DUSSAUCE.

times 15 feet deep and 15 feet in diameter, and capable of turning out 25 to 30 tons at one boiling. It has been ascertained* that for every 100 lb. of fatty matter a capacity of $37\frac{1}{2}$ gallons is required. Hence—

| | | |
|----------------------------------|-----------|------------------|
| 1000 lb. fat require a copper of | 375 | gallons capacity |
| 2000 " " " | 750 | " |
| 3000 " " " | 1000-1125 | " |

In some large American factories the coppers extend through several storeys of the building.

The heating may be accomplished either by fire or by steam. In either case the pans are set in brickwork, and so built round, when fire is used, that the fire shall not play upon the sides, but only on the convexity of the lower part of the boiler; but, even after every attention has been given to the construction of the arrangement on the most scientific principles, there is an enormous waste of fuel.

Heating by steam may be effected either by passing the steam directly into the pan by steam-pipes terminating in a perforated coil resting on the bottom of the pan (*open*, or *wet* method); or (*a*) by a closed coil, or (*b*) by means of a steam-jacket (*close*, or *dry* method). If the steam is discharged directly into the mass of soap, as in the *open* method, some disadvantage is experienced through the weakening of the lye by the condensation of the steam, and, on this account, the use of more concentrated lyes is rendered necessary. The arrangement found to work best is to send the steam through a flat, closed worm about 3 or 4 inches above the bottom of the pan. In this way a pan holding 1000 lb. may be boiled in half an hour, while to do the same by means of a fire would take from three to four hours. Besides, a single steam boiler and one furnace will thus heat

* DUSSAUCÉ, "Treatise on the Manufacture of Soap," p. 344.

several pans at once, and there is no danger of the soap burning. Steam, therefore, affords economy of fuel, labour, and time, and the boiling can be more readily, at any time, controlled. Superheated steam is still more rapid in its operation, and cheaper than ordinary steam.*

The lids of the pans, made either of wood or iron, are arranged so that they may be put down, or taken off, by means of a chain and pulley. The soap is removed either by pumps or by ladling, and the lyes either by pumping or by a pipe fixed to the bottom of the copper.

FIG. 6.

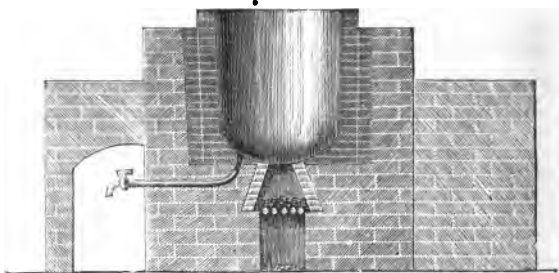


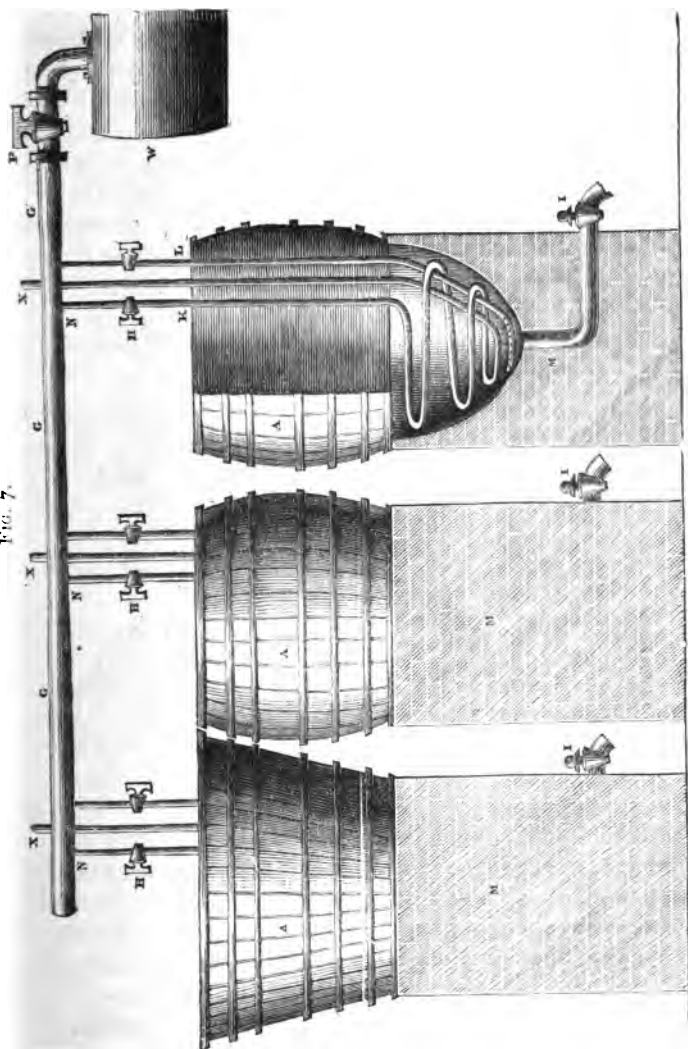
Fig. 6 represents a boiler arranged for heating by the direct heat of a fire.

Fig. 7 is a representation of the arrangement designed by CAMPBELL MORFIT for employing steam heat, and known as *Morfit's steam series*.

In this figure three caldrons, A A A, are shown. In large factories this is a convenient number, though more are often used, but, in a small work, one will answer, though there will always be a loss of time in cleaning it when the charge has to be changed from yellow to white soap. The

* DUSSAUCE, "Treatise on the Manufacture of Soap," p. 350.

FIG. 7.



feeder, *g*, is attached to the boiler, *w*, which is generally fitted against the wall, immediately above the caldrons. The cock *i* is for the withdrawal of the spent lyes. The pipe, *l*, called the *blow-pipe*, serves to communicate, when necessary, additional heat to the contents of the pan, and is also useful to stir up the mass occasionally, an operation more readily accomplished in this way than by a *crutch* in the hands of a workman. Steam is let on or off by the cock *h*. Waste steam passes off through *x*. The current of steam from the boiler may be regulated by the cock *p*.

2. Processes requiring Definite Proportions of Alkali and Fat.

a. Cold process.—The apparatus required for this operation, according to HAWES, who invented it, may be an ordinary caldron (Fig. 8) with the addition of a machine to

FIG. 8.

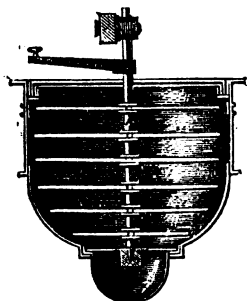
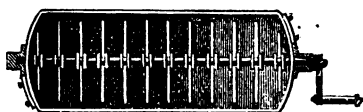


FIG. 9.



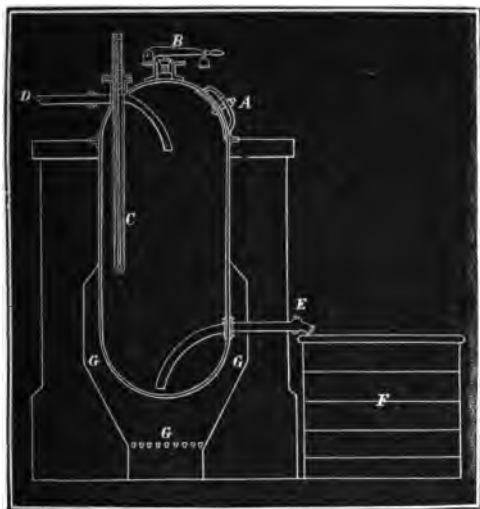
produce the intimate admixture and minute division of the tallow; or a cylinder, as represented in Fig. 9, may be used.

b. Boiling under pressure.—For boiling under pressure, DUNN's apparatus, represented in Fig. 10, may be employed.

The boiler should be furnished with a man-hole, *A*, a safety-valve, *B*, a thermometer fixed in a mercury chamber, *C*,

and all the ordinary appendages of such an apparatus. *D* is the feed-pipe, and *E* the discharge-cock. When in use, the valve is weighted till the temperature in the boiler

FIG. 10.



rises to 310° F., and the boiling is complete in about an hour after that temperature is reached.

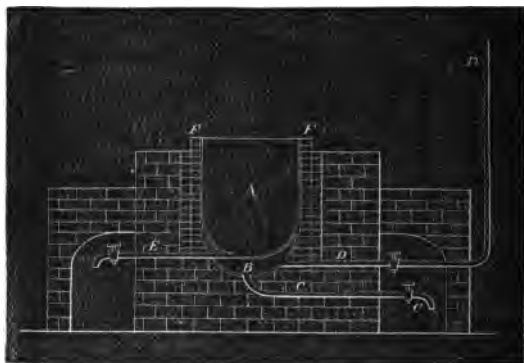
c. Open boiling.—The ordinary open pans already described are suitable for preparing the soaps which fall under this head.

d. Free-acid process.—This is also called *Morfit's process*. The boiler is made of wrought iron, is steam-jacketed, and is fitted with a wrought-iron *stirrer* for thoroughly mixing the ingredients. Fig. 11 is a representation of the steam-jacket pan designed by MORFIT.

A is the interior of the kettle, surrounded by brickwork; *B* is the outer cast-iron caldron, which should fit the inner

kettle tightly so as to prevent any escape of steam; *D* is the steam-pipe from the boiler, fitted with a cock by which steam may be let on or off; *C* is the discharge-pipe for con-

FIG. 11.

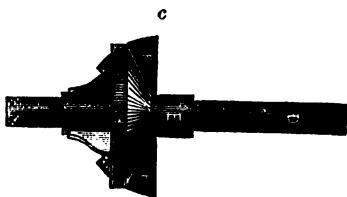
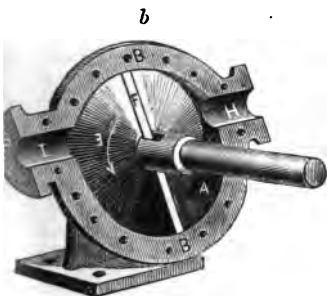
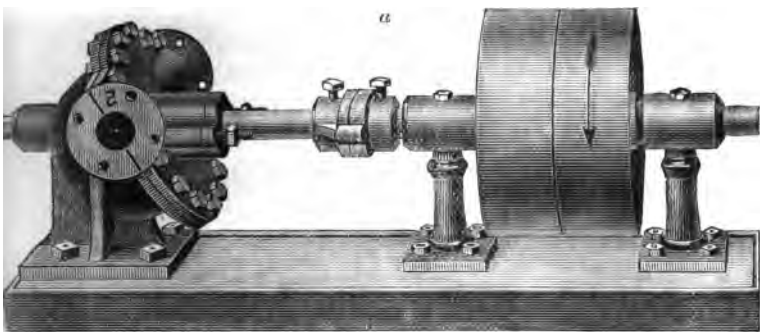


densed vapour—the cock in this pipe may be left slightly open so as to form a safety-valve; *E* is the discharge-pipe of the kettle.

A pump may be conveniently employed for taking off or removing soap, when required, from one pan to another, or for introducing either hot or cold lye, or strengthening change lye. A very serviceable description of pump is made by Hersey Bros., of South Boston, Mass., and is represented in Fig. 12 (*a*, *b*, and *c*). *a* represents the pump complete. When the pump is rotated in the direction of the arrow, the outlet marked *s* is the suction; when rotated in the opposite direction, the opposite outlet becomes the suction, and thus, by giving a few revolutions by hand in this direction, the discharge-pipes may be emptied of their contents. *b* is a view of the interior of the pump when the cover is taken off; when turned in the direction of the arrow, the blade *F* sweeps round, drawing the fluid in at *i*,

and forcing it out at *h*, the contents of the pump being twice emptied at each revolution. The fluid is prevented from passing from one side to the other by the contact of the cone with the cover. *c* shows the cone and blade, and forms the entire working part of the pump. No valve is

FIG. 12.



used, and the operation of the pump is consequently little liable to any derangement.

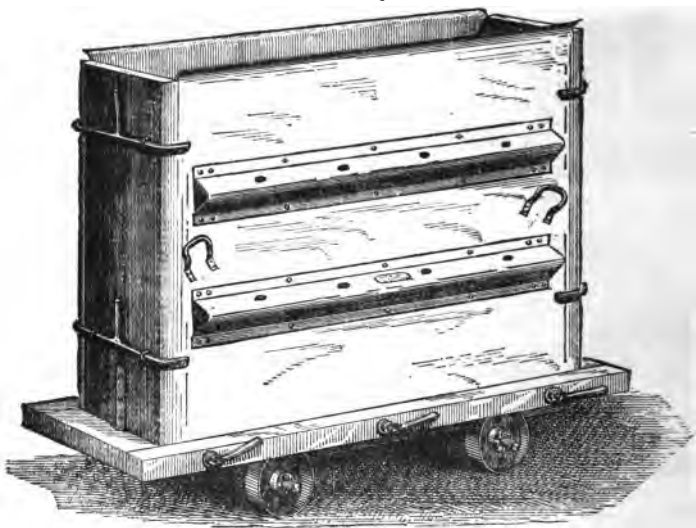
The pump may be set up in any convenient position adjacent to the pan, not more than 10 feet above its bottom, and connected to it by means of a $2\frac{1}{2}$ -inch iron pipe, tapped through the iron plate at a distance of about 2 feet above the worm, or coil. Several pans may be connected with the

pump by iron pipes, with valves placed upon them on the outside of the kettle, so that any one of them may be pumped off and framed without disturbing the others. Inside the pan the pipe has a suitable swing-joint so arranged that it can be raised or lowered at pleasure.

3. Appliances for Finishing the Soap.

Frames.—The frames, which were formerly made only of wood, are now constructed of iron, commonly cast iron, and the wooden ones are chiefly used for mottled soaps, which

FIG. 13.

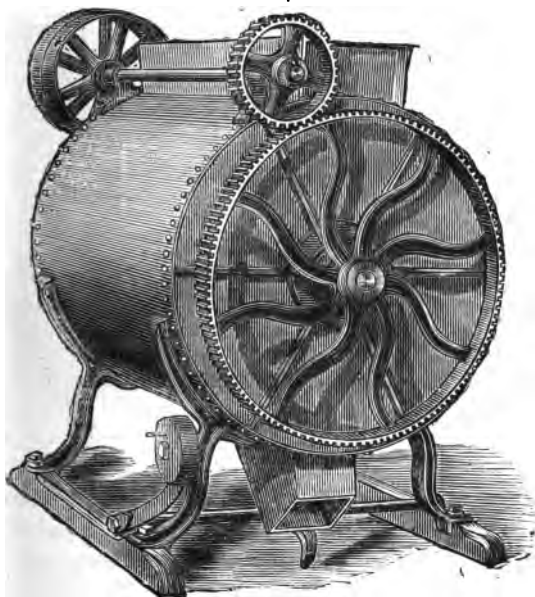


require slower cooling than other descriptions. When soap was subject to duty, the dimensions of the frames were fixed by law, and were required to be exactly 15 inches by 45 inches inside, and not less than 45 inches deep. These dimensions are generally still retained in England, and

hence an English bar of freshly made hard soap measures 45 inches in length.

The wooden frame is made up of a number of separate sections, piled upon each other, and fitting closely together. Each section, having the internal measurement just mentioned, is about 9 inches in depth, and is constructed of wood, about 2 to 3 inches in thickness, lined with thin sheet-iron. These are frequently piled upon one another to the height of more than 20 feet. The bottom of the frame may be of wood, or brick, and furnished with a well to receive the drainings. When the soap has become solid, the frames are removed one by one, and the block of soap remains ready for division into slabs.

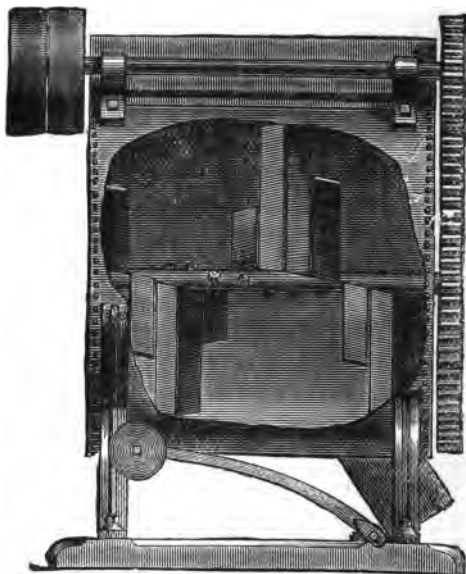
FIG. 14.



Outside view of crutching machine.

Iron frames are now extensively used. Fig. 13 is a representation of WHITAKER's patent frame,* much used by American firms. It consists of two sides of plate-iron, flanged at their upper edges, and strengthened by ribs of corrugated plate-iron, riveted to the outer surface, and running in the direction of the length of the frame. These ribs are intended to prevent the *buckling* or twisting of the side-

FIG. 15.



Working part of crutching machine.

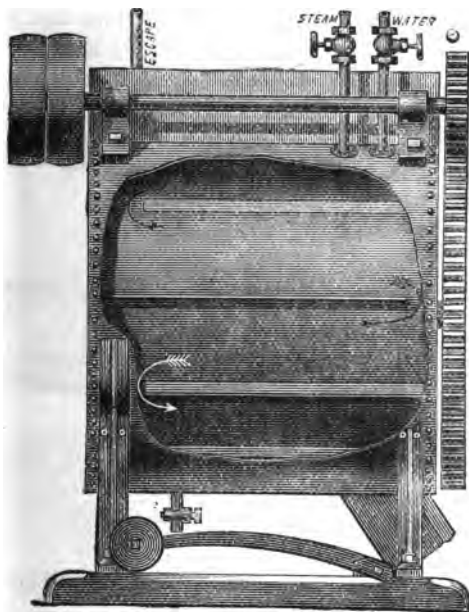
plates. The trouble and expense of the ordinary stays and supports are thus avoided, as the frame is self-sustaining. The sides are connected by ends made of 2-inch plank, secured by clamps. The frame is very light, and easily

* Made by Hersey Bros.

worked. The soap cools sufficiently to strip in twenty-four hours in cold, and in forty-eight hours in warm, weather.

Crutching.—For stirring the soap-paste in the pans or frames, an instrument called a *crutch* is used, consisting simply of a board, to which a long wooden handle is

FIG. 16.

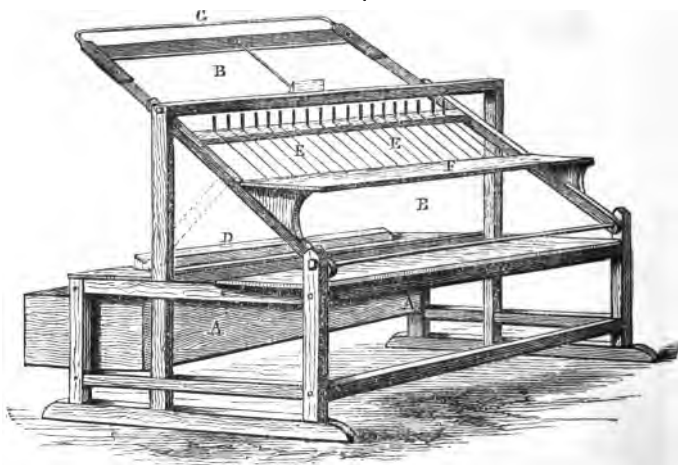


Jacket view of crutching machine.

attached. For mixing various ingredients with soaps, several forms of steam-crutching machines are employed. Figs. 14, 15, 16 are representations of a form patented by STRUNZ (May 13, 1873, and April 23, 1878), and largely employed in the United States. It crutches soap completely within three minutes, and turns out an article of great smoothness.

Cutting and Barring.—The blocks of soap when removed from the frames are marked off on the sides by means of a *scribe*, or *dentier*. This consists of a stick of hard wood, in which are fixed iron teeth. The distance of the teeth from each other is arranged according to the desired dimensions of the bars. The workman then, by means of a brass or steel wire directed in the track of the scribe-marks, divides the mass into *slabs*, which are afterwards subdivided into *bars*.

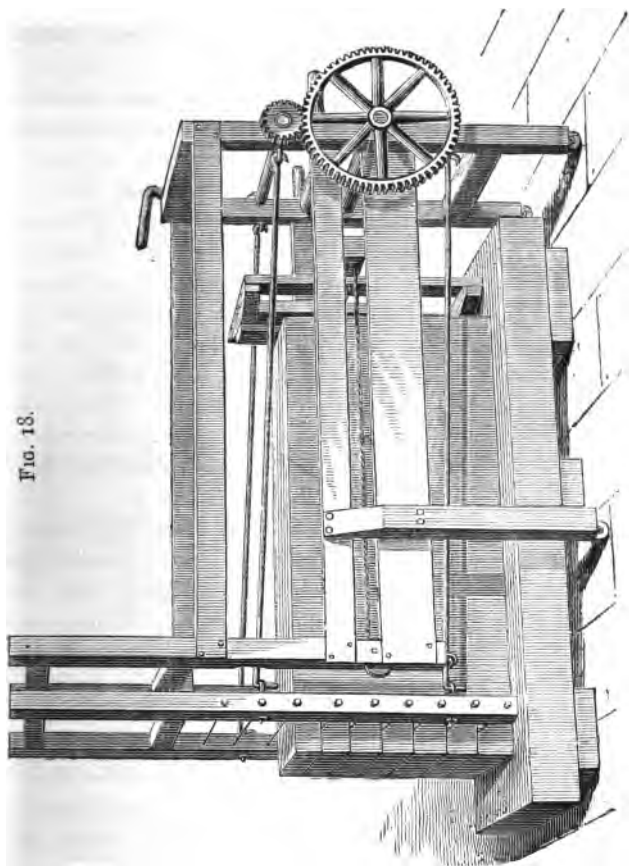
FIG. 17.



The operation of barring may be rapidly accomplished by machinery. Fig. 17 is an illustration of a soap-cutting machine much used in this country.* It consists of a fixed frame of woodwork, A A, and a movable lever-frame, B B, attached to A A by the centre-pin, C. The frames are wide enough to receive a slab of soap 45 inches long by 15 inches wide. This is placed in an inclined position, as

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 664.

shown by the dotted lines, resting on the bar, *D*, of the fixed frame, and against a number of wires forming part of the movable frame. When the lever, *a*, is pressed down, the



wires pass through the slab of soap, dividing this into regular bars, and when the handle is again raised up to the position

shown in the figure, the bars of soap are found on the table, *r*, ready to be removed.

Fig. 18 is an illustration of RALSTON'S champion soap-slabber, made by Hersey Bros., which is considered as effective as it is simple, and is little liable to get out of order.

Fig. 19 exhibits an arrangement, by the same makers, by means of which the three operations of *cutting*, *stamping*, and *spreading* may be carried out. The frame of soap is cut into slabs either by the slabbing machine, of which Fig. 18 is an illustration, or else by the old way of slabbing by hand. In either case the slabs are taken one by one and placed on the *cutting-table*, shown on the right-hand side of Fig. 19. They are forced against a set of wires, and are thereby divided into *bars* by turning the handle seen on the right-hand side of the machine. They are afterwards pushed against the wires shown on the left-hand side of the machine, in a direction at right angles to the former movement, and are thus divided into *cakes*, the size of which is regulated by the distance apart of the wires.

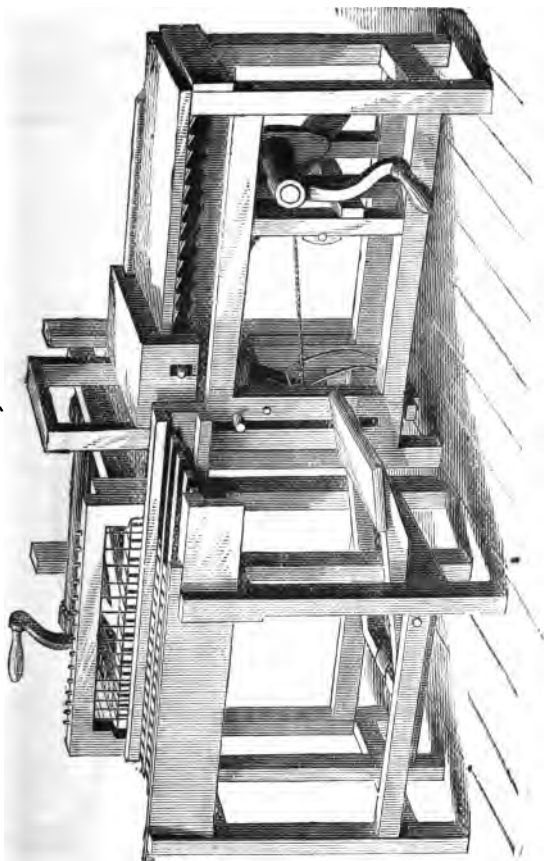
The *stamping attachment* consists of a framework, which is seen in the central portion of the machine, and which, by suitable means, is brought down at regular intervals as the soap passes through, so as to stamp upon each cake some name or simple device. It is intended to be used in cases where the soap is to be put on the market without being pressed.

The *spreading attachment* consists of a series of wooden blocks of such size that, when the soap has passed through the second set of wires, each cake rests upon one of the blocks. These blocks are attached to strips of webbing in such a manner that, when the strips are pulled tight, there is a slight interval between the blocks. To receive the soap, the blocks are pushed close together.

The racks for soap are laid so that the strips of which

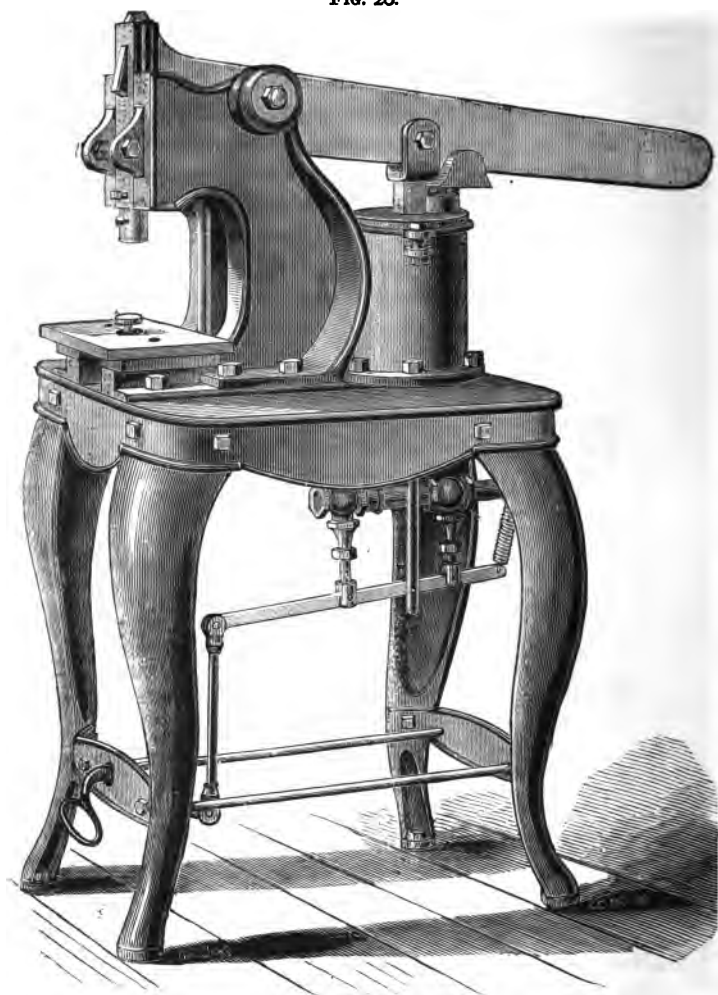
they are formed lie in intervals left between the rows of blocks, and, after the soap has been pushed on the blocks by

FIG. 19.



the action of the cutting portion of the table, a slight pull on the ends of the webbing separates the cakes, so that the racks can be lifted and placed for drying, with the cakes of

FIG. 20.



soap in the best position for that purpose. The treadle, shown on the lower portion of the spreading attachment, is intended to lift each alternate block slightly before they are pulled apart, so that the cakes of soap will separate more readily.

Stamping.—The name of the maker, or the description of the soap, &c., may be put on by means of a stereotype plate and a mallet, or by a stamping machine, such as Figs. 20, 27, 28, 29. By the HERSEY steam press (Fig. 20) a boy can turn out from 1800 to 2000 cakes per hour; a gentle pressure of the foot upon the treadle fills the cylinder with steam, causing the die to descend with great rapidity and power upon the cake, and the instantaneous return of the lever raises it out of the die-box ready for removal. The cakes may vary in weight from a few ounces up to the largest sizes.

ARRANGEMENT OF THE FACTORY.

The following plans for a soap factory, which have proved convenient in actual working, are outlines of those given by DUSSAUCE and CRISTIANI:*

The whole building is of an oblong or square form, divided into three compartments.

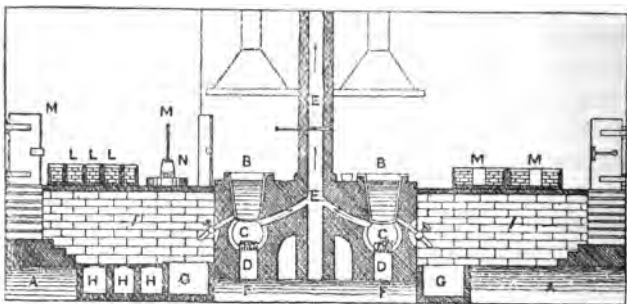
1. The *boiling-house*, containing the kettles, frames, and lye-vats, is most conveniently placed in the centre of the factory and arranged round the large chimney. For a large business, two large boiling-pans answer in most cases, while two other pans may be reserved for making the lyes. If the kettles are to be heated by open fire, or by superheated steam, the furnace is usually in the basement, while the rim

* DUSSAUCE, "Treatise on the Manufacture of Soap," pp. 382-388; CRISTIANI, "Technology of Soap and Candles," pp. 197-217.

of the kettle is extended above the first floor, at a height sufficient to facilitate the stirring.

The *lye-tanks* are best made of cast iron, and are frequently inserted in the ground for the sake of economizing space. They must be well covered—best with cast-iron

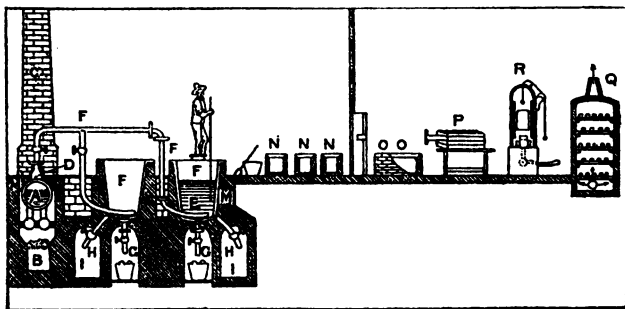
FIG. 21.



lids. But the most convenient arrangement is to have the tanks in an elevated position, so that the lyes can be drawn off.

2. *Store-rooms.*—Adjoining the boiling-house, on one side, should be a warm store-room for the alkalis, and a second room, as cool as possible, for the fatty matters.

FIG. 22.



3. On the opposite side of the boiling-house may be the *barring* or *cutting* room and the *drying* and *packing* rooms.

Description of Fig. 21 (pans heated by open fire):—

A A, *Factory building*.

B B, *Kettles*. C C, *Fireplace*. D D, *Grate*. E, *General chimney*. F F, *Ash-pit*. G G, *Cisterns* for waste lye. H H H, *Vessels* for oils and fats. I I, *Cellars*. L L L, *Lixiviating vessels*, situated above caustic lye-vats. M M (on right of illustration), *Soap frames*; the upper part should be *lower* than the edge of the kettle, so that, after boiling, the soap may, by a *shoot*, be readily run into them. M M (on left of illustration), *Store-rooms*. N, *Apparatus for powdering crude soda*.

Description of Fig. 22 (pans heated by steam):—

A, *Boiler*. B, *Fire-grate*. C, *Chimney*. D, *Dome* from which steam is discharged through the pipe, F F, and the *flat coil*, E E, at the bottom of the kettle. F F, *Kettles*. G G, *Waste-pipes*. H H, *Spent-lye pipes*. I I, *Spent-lye cisterns*. M M, *Foundation of kettles*. N N, *Sheet-iron caustic-lye vats*. O O, *Soap-frames*. P, *Barring-table*. Q, *Drying-room*. R, *Soap-moulding machine*.

The arrangement of a small factory illustrated in Fig. 23 is one that has been found efficient in its results and economical in its working. It may be thus briefly described:—

A A are soap-pans, consisting of a wrought-iron curb, b being the cast-iron bottom. These pans, one of which only is shown in section, are set in brickwork, bound round with wrought-iron tie-bands. c is the cock for drawing off the lyes or spent alkali.

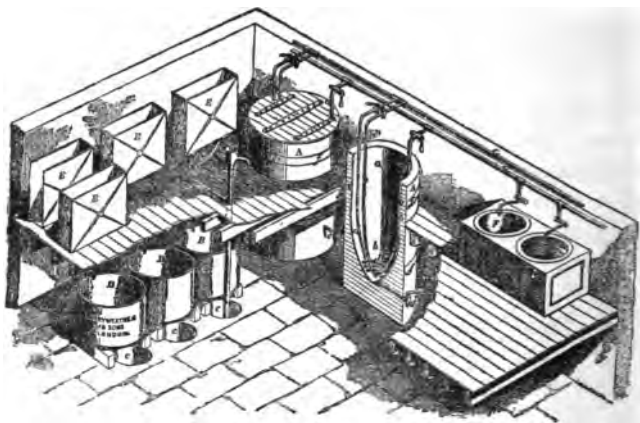
d d the close steam-heating worms, or pipes, connected to the steam and waste mains, G and H.

e e are the open free steam-boiling worms.

f f are the tie-bands for securing the brickwork round the boiler.

Both the pans have covers the same as shown on the pan not in section.

FIG. 23.



B B B are the cast-iron lye or alkali vats, having false bottoms, and being fitted with water-supply.

C C C are the cast-iron pans for receiving the lyes or alkali solution from the vats, *B B B*. The lyes are taken from these pans by means of a pump, through a trough, to the soap-pans, *A A*.

E E E E are the frames in which the soap is cooled, the side and end plates of which are taken off.

F F are steam-jacketed pans for making toilet soap. They are fitted with free steam-boiling worms and all necessary connections, and are placed on a bench as indicated.

G is the main steam-pipe from the boiler.

H is the main waste steam-pipe.

CHAPTER VI.

CLASSIFICATION OF PROCESSES.

DR. W. LANT CARPENTER's classification is as follows: *—

a. Soaps produced by the *direct union* of fatty acids and caustic alkali, or by the decomposition of carbonated alkali by fatty acids.

b. Soaps produced by the action of the *precise quantity* of alkali necessary for saponification upon a neutral fat, without the separation of any waste liquor, the glycerin being retained in the soap. This class includes (1) soaps made by the *cold process*; (2) soaps made *under pressure*.

c. Soaps produced by the *ordinary methods of boiling in open vessels*, working with indefinite quantities of alkaline lyes, the processes being controlled by the experience of the operator. These are again subdivided into (1) *soft soaps*, in which the glycerin is retained, potash being the base; (2) the so-called *hydrated soaps*, in which the glycerin is retained, and of which *marine soap* may be taken as a type; (3) *hard soaps*, with soda for the base, in which the glycerin is eliminated, comprising three kinds—curd, mottled, and yellow soaps.

Dr. C. R. A. WRIGHT† classifies the various processes for the production of soap as follows:—

* Spon's "Encyclopædia," v. 1770.

† Cantor Lectures, "Journ. Soc. Arts," May 1885.

Group I.—Fatty, or resinous, acids in the free state directly neutralized with alkalies (carbonated or caustic). Resulting soap devoid of glycerin.

Group II.—Saponification of fatty glycerides by alkalies, with retention of glycerin intermixed with the soap. In this group are the processes for making (a) soft soaps and marine soaps by *open boiling*; (b) soaps made by *boiling under pressure*; and (c) *cold-process* soaps.

Group III.—Saponification of fatty glycerides by alkalies, with separation of glycerin.

Group IV.—Processes consisting of combinations of the foregoing.

It will be seen from a consideration of the above that the *methods* may be arranged under three main heads—viz., *open boiling*, *boiling under pressure*, and the *cold process*.

1. General Process.

The general method of preparation is the same for all the hard soaps, but there are variations in the details, more especially in the later stages. The following is an outline of the general method:—

1°. *Saponification, Pasting, or Killing the Goods.*—Usually the whole of the fat to be saponified is introduced into the boiler, and at the same time, for every ton, from 150 to 200 gallons of caustic lye, of sp. gr. 1.050 to 1.085 (10° to 17° Tw.), are added, and the whole is gently heated to ebullition. Lye stronger than sp. gr. 1.085 would, at this stage, hinder saponification. After boiling for an hour and a half or two hours, a viscid emulsion, capable of being drawn out into threads, or ribbons, is produced.

2°. *Separation, Cutting the Pan, or Salting.*—To separate the imperfect soap produced, and to allow the spent lye, containing the glycerin, to be withdrawn, a sufficient

quantity of common salt is added, and this, dissolving in the liquid, causes the soap, which is insoluble in the saline solution, to rise to the surface, combined with a definite proportion of water. Thus separated, the soap is called *grain* soap. The spent lyes should contain no caustic soda, and no fat should be thrown up on adding to them a mineral acid.

3°. *Completion of Saponification or "Finishing."*—This part of the operation follows the removal of the waste lye, by pumping or drawing off. It consists in boiling up the granulated soap with fresh, stronger lyes, called *strengthening* lyes, to complete the soap, and to bring it into what is called the *close* state.

If *curd* soap is to be prepared, it is allowed to stand a while, that the lyes may subside, and then the operation is continued as in 5°.

If the *grain* soap contain impurities, such as iron soap, iron sulphide, &c., and if the quantity of water be not in excess after cooling in the frames, a *marbled* or *mottled* appearance results.

4°. *Fitting.*—The unrefined grain soap is apt to contain a proportion of lye entangled in it. To separate this, the curd is melted, with the addition of water or weak lye, and boiled, so as again to produce a homogeneous compound. The mixture is allowed to stand for a considerable time—about two days—when a separation takes place into three layers, and the soap, which forms the middle layer, is then treated as in 5°.

5°. *Cooling and "Cleansing."*—When the soap has remained in the pan a sufficient time to become partially cool, it is ladled out in buckets, or pails, or by other means conveyed to the *frames* to solidify.

Curd soap has then a rough, granulated texture, and is extremely hard, containing only about 20 per cent. of water.

A properly fitted soap will have a *feathery* texture, and contain about 30 per cent. of water.

6°. *Barring and Drying*.—The soap having become cold, the frames are removed, and a compact mass of soap, the size of the frames, remains. This is marked round by the iron-toothed *scribe* or *dentier*, the teeth of which are near or distant from each other according to the size of the blocks desired. The mass is then cut in the places so marked into *slabs*, and these slabs are subdivided into *bars*. These bars are then removed to the drying-room, and piled upon one another cross-ways, interstices being left for the circulation of air to facilitate the drying.

MORFIT thus describes the general method pursued in the United States :—1°. “The strength of the lye employed varies as the fat to be saponified is richer in olein or in solid constituents. The operation is commenced by pouring the lye into the copper to a third of its capacity. This is then heated to ebullition, and the oil is now run in. The reaction is such that a magma is immediately formed. The proper formation of this magma is considered to be the most delicate and important part of the whole process, and, if badly managed, a much greater quantity of lye is required to form the same weight of soap than would otherwise be necessary. After the addition of the fat, the heat is decreased by opening the doors of the furnace, and, when the mixture of fat and lye is complete, if necessary a further quantity of weak lye is added gradually, and with constant stirring during the addition so as to insure thorough contact. The mass should remain homogeneous; the oil should neither rise to the surface nor descend to the bottom.

“If oil should present itself, it is then necessary to add more strong or weak lye, according to the capacity of the caldron. On the other hand, if the lye is in excess, a further quantity of oil must be added, always stirring briskly upon

any addition of new material. The operation requires from eighteen to twenty-four hours for completion, but it may be greatly accelerated by throwing in the *scrapings* or waste of soap already made."

"An excess of soda is recognized by the liquidity and transparency of the paste. When oil is in excess, it rises to the surface. An excessive proportion of common salt in the soda also more or less interferes with the proper formation of the magma, and, if the proportion is very considerable, the use of soap scraps is indispensable."

2°. "The next step in the process is the removal of the large quantity of water which was required for the complete saponification. This is effected by the addition of lye containing common salt, and by afterwards boiling the mixture for from fifteen to twenty hours, with constant stirring. When the mass opens in different places, the separation is complete. The fire is then withdrawn, and the whole is allowed three or four hours' repose, after which the settled waste, or spent, lye is drawn off. A further quantity of lye, charged with common salt, is now added, and the mixture is gently boiled, care being taken to remove from the sides of the caldron any adhering soap, so that all portions may come into contact with the lye. The mass now acquires more consistence, and, after some hours' rest, the settled waste is again withdrawn."

3°. "A fresh quantity of lye, of sp. gr. 1.10 (20° Tw.), is now added, and the mixture again boiled, by which it acquires still greater consistence. After about three hours' further boiling, it is allowed to settle, and the spent lye is again drawn off. This operation is again repeated, with *strong* lye, constant stirring, and gentle ebullition, so that the whole may form a homogeneous mass. At this stage the soap begins to acquire firmness.

"The boiling with lye several times successively serves

not only to complete the saponification, but *to wash* and *purify* the soap. That it may be perfect, it is necessary to repeat the operation four or five times. As soon as complete, the heat should be withdrawn, and the mass allowed to settle and become somewhat cool. It is then ready to be conveyed to the frames."

2. Saponification under Pressure.

BENNETT and GIBBS, of Buffalo, N.Y., took out a patent in 1865 for making soaps by agitation under pressure. This method consists in agitating in a closed vessel, or boiler, fitted with a revolving shaft, or stirrer, the fatty matters with caustic or carbonated alkalies in solution in water while under heat and pressure, in such a manner as to cause a thorough mixing of the fats with the alkaline solution, and the production of a rapid combination of the fatty acids with the base. The pressure is 220 to 280 lb. per square inch, at the temperature of 350° to 400° F. (176.6° to 204.4° C.). At first, if carbonated alkali be used, it is necessary to allow some of the liberated carbonic acid to escape, so as to avoid undue pressure. A batch of soap may, in this way, be made in less than one hour.

The patentees used from 30 to 33 lb. of sodium carbonate at 48°, and 100 lb. of water to each 100 lb. of lard, tallow, or oil. The produce obtained is 200 lb. of soap for every 100 lb. of grease.

The following advantages are claimed for this method :—
(1°) Rapidity; (2°) Quality improved; (3°) Quantity increased; (4°) Labour saved; (5°) Fuel saved; (6°) Cost of materials saved; (7°) Completeness of saponification; (8°) Uniformity of results; (9°) Incorporation of glycerin; (10°) Admissibility of alkaline salts, instead of caustic lye.

DUNN's method (p. 103) is also available for preparing

ordinary soaps under pressure. It differs from the preceding in the employment of caustic, instead of carbonated, alkali, and of a lower pressure (20 to 65 lb. per inch).

3. The "Cold" Process.

By this method the high degree of heat necessary in the ordinary process is entirely dispensed with, and complete saponification is effected at temperatures lower than the ordinary boiling heat. The following is the description of the system as given by WILLIAM HAWES, the inventor:—Two tons and a half of tallow, or any given quantity, are taken and melted at as low a temperature as possible, and then mixed, by mechanical means, with the quantity of lye required to completely saponify the fat. Ordinary soap-boiler's lye is used, preference being given to that made from the strongest and purest alkali. The saponification of the tallow, or other fat, may be ascertained by its absorption or combination with the lye, care having been taken, in the first instance, to use a sufficient quantity of the lye. Twenty gallons of lye, of sp. gr. 1.125 (25° Tw.), are required for every 100 lb. of tallow, but the proportion varies according to the nature of the fat or oil employed. An ordinary soap-pan may be used as the combining vessel, with the addition of a stirrer to facilitate the admixture of the tallow and the lye. Figs. 8 and 9 (p. 64) will convey an idea of the apparatus. For the quantity of fat mentioned above, if the cylinder is used, it should be about 6 feet in diameter and 12 feet in length. When it has been charged with fat, motion is communicated to the machinery, and the lye is then gradually added. In a short time the ingredients will be thoroughly mixed, but the agitation must be continued for about three hours, or until saponification appears to be complete. During the process there is a considerable evolution of heat. After from one to four days,

according to the quantity, it is hard enough for use. As the value of the process chiefly depends upon the lowness of the temperature at which the saponification is effected, it is desirable to transfer the contents of the cylinder, as soon as thickening occurs, to an ordinary soap-pan, where the operation may be finished, either by conversion into yellow soap with the addition of rosin, or into mottled or white soap by finishing lyes in the usual way.

The cold process is very suitable for the manufacture of soap on a small scale, and in such case the mechanical stirrer can be dispensed with.

The advantages obtained by this process are—economy of cost and time, retention of the glycerin, and, when perfumes are introduced, the avoidance of loss which a high temperature naturally causes.

The disadvantages are—the liability of the product to contain an excess of alkali, and the necessity of having very pure materials, because in no part of the operation is there any opportunity of getting rid of objectionable impurities. If the alkali contains too much chloride, it will be necessary to add a proportion of cocoa-nut oil in order to effect the saponification. It is also not uncommon to find, owing to incomplete saponification, that the product contains both unsaponified fat and free alkali.

We shall consider the various commercial soaps produced by these methods, or by modifications of them, in the following order :—

Household, domestic, laundry, or plain soaps.

1. *Curd, or white, soap.*
2. *Genuine mottled soaps.*
3. *Castile, Marseilles, Venetian, or olive-oil soaps—white and genuine mottled.*
4. *Artificially mottled soaps—blue, grey, and red.*
5. *Yellow, or rosin, soap.*

6. *Cocoa-nut-oil, marine, or hydrated soaps.*

7. *Silicated soaps.*

8. *Sulphated soaps.*

Toilet, or fancy, soaps.

Medicinal, or pharmaceutical, soaps.

Oleic-acid, or red-oil, soaps.

Soft soaps.

Industrial soaps.

CHAPTER VII.

HOUSEHOLD, DOMESTIC, OR LAUNDRY SOAPS.

HARD soaps are made with non-drying oils, or solid fats and soda. Their hardness is in proportion to the amount of stearic and palmitic acids which they contain. Soda soaps made with drying oils, such as linseed, are pasty and easily liquefied by a small quantity of water, and approach to the character of soft soaps made with potash. The most important kinds of hard soaps are those made chiefly with tallow, as in England and other Northern countries, and olive-oil soaps, as made in Southern Europe.

1. *Curd, or White, Soap.*

A. ENGLISH METHOD.*—The fatty materials used for the production of hard soaps in this country are tallow, lard, palm oil (well bleached), and cocoa-nut oil, or mixtures of these in almost any proportion. The pan used is the ordinary open boiler (pp. 62, 63, 66), heated either by fire or by *closed* steam-pipes. From 10 to 14 cwt. of tallow are required to produce 1 ton of soap.

1°. The pan having been charged with the fat, weak lye of specific gravity about 1.040 is added (the proportions are

* Founded on GOSSAGE's description—RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 680.

about 200 gallons of such lye to 1 ton of the fat), and the whole is heated, by injection of steam, or otherwise. If the process goes on properly, the fatty matters soon combine with the lye, producing a uniform milky emulsion, from which no watery particles separate on cooling. If such an emulsion is not produced, water, or weaker lye, is added, and the boiling is continued till the combination is complete. At this stage, the application of the tongue shows that the taste of the alkali has passed away, or, in technical language, the lye is *killed*. Repeated additions of stronger lyes are made, and the boiling is continued till the presence of free alkali becomes evident to the tongue. It is then necessary to introduce more fat, followed by stronger lyes, but with care that, at this stage, the alkali shall not be in excess.

2°. The imperfect soap is now separated by the addition of common salt, and, after a few hours' subsidence, the spent lyes are withdrawn from under it. This spent lye contains a portion of the glycerin of the fat, together with sodium sulphate (from the alkali used) and common salt.

3°. The next stage of the process consists in the addition of weak lye to the imperfect soap, and subsequent boiling to bring the contents of the pan into a state of homogeneous mixture, called the *close state*, as distinguished from the *granulated* condition in which the soap separated at the end of the first operation.

Stronger lyes (of about sp. gr. 1.160) are now added till the mixture has a strongly alkaline taste. Sufficient common salt is then thrown in to cause the separation of the soap, and the mixture is boiled for several hours, so that the whole of the fat may be combined with the alkali.*

* This point is well known to the experienced workman by the consistence of the compound. If a little of the mass taken out on a

In this process, attention has to be specially given to the separation of the *alumno-ferruginous* impurities of the lye, which, if not removed, tend to discolour the soap. Their removal is effected by boiling the soap several times with fresh weak lye or water, applying gentle heat, covering the caldron, and allowing time (one to two or three days, according to the quantity of the materials) for the darker-coloured soap, or *nigre*, to settle. The upper stratum of white soap is afterwards ladled out into the cooling frames, curd soap being generally too thick to pump.

In England by far the greater quantity of curd soap produced is made from tallow, or mutton suet, and soda only. Soap thus made is, however, inconveniently hard and difficult of solution. Hence, some manufacturers replace one-fourth of the tallow by as much lard, or olive oil, obtaining thus a soap of superior quality, and less liable to change by exposure. Lard has this advantage over olive oil—that it does not detract from the whiteness of the soap. The advantages gained by the use of lard and olive oil with tallow are thus summed up by MUSPRATT :*—"The soap remains unaltered for a longer period ; it does not emit the disagreeable odour of tallow ; and the saponification is more perfect, as the excess of olein in the lard, or oil, compensates for the large amount of stearin in the tallow, thus inducing a more ready and perfect union of the alkali and fatty acids."

English curd soap is much used in Yorkshire by cloth

trowel is squeezed between the finger and thumb, it will still have a *greasy* feeling if not thoroughly finished ; but if the saponification is complete, it will readily separate from the skin in hard scales. Or a portion may be decomposed by an acid, and, if the saponification is complete, the separated grease is *wholly soluble* in boiling spirits of wine, but not otherwise.

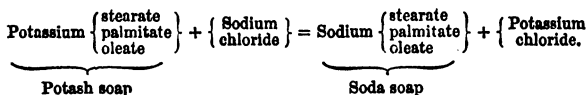
* "Chemistry," ii. 879.

manufacturers, and at Nottingham in the bleaching of lace and stockings.

B. GERMAN METHODS.*—(a) The *old method* of preparing hard soap formerly practised in Germany is of great interest, historically and chemically, and a short description of it may therefore find place here.

1°. The crude tallow is saponified with potash lye prepared from potashes causticized by lime in the usual manner. The first lye has a strength of 20° B. Soon after the boiling has commenced, an emulsion is formed, and, on continued ebullition, the mass becomes clear, having the appearance of a thick syrup, indicating that the whole of the fat has entered into combination with the potash. Turbidity may be due either to excess or deficiency of potash, or to the presence of lime. On treatment of a few drops of the mixture with pure rain-water, a continuance of the milkiness indicates unsaponified fat or the presence of lime. If the latter is the cause, it is removed by the addition of a carbonated alkali; when there is unsaponified fat, more lye must be added and the boiling continued. The milkiness due to excess of alkali disappears on addition of water.

2°. When the clear solution will flow from the spatula in an unbroken stream of the consistence of treacle, and will solidify to a thick jelly when placed on a cold stone, the product is ready for the *salting process*. This consists in throwing salt into the pan and boiling up with the solution of soap. "Double decomposition" takes place analogous to that ensuing between silver nitrate and sodium chloride (see example (e) (2), p. 51), thus:—



* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 674.



This reaction, in conjunction with the excess of common salt, causes the separation of the soda soap and the formation of the under-lye. The soap, insoluble in the brine, coagulates into a whitish mass of small flocks. After resting for some time, the soap is scooped out into the cooling-frame. The soap is obtained quite clean, even from very impure materials, being washed by the salting process. The impurities from the salts in the ash employed, and from the action of the lye on the membranous parts of the crude tallow, are all found in the under-lye.

When all the soap has been taken out, the lye is removed, and the soap afterwards replaced in the clean pan, with an addition of fresh weak lye. The mass is boiled, and a clear solution, as in the first boiling, is obtained, containing chiefly soda soap, but with an admixture of potash soap from the fresh lye. Salt is again added, and the boiling continued. The heat is then removed, the contents of the pan allowed to rest, the soap scooped out again, and the under-lye emptied out.

After this, if crude tallow has been used, about three or four more boilings must be made before the soap is completely saturated with alkali. Less salt is required at every fresh boiling, because there is gradually less potash soap to be decomposed.

A less number of boilings will be sufficient if purer tallow is employed.

3°. The mass is then boiled clear, and if the soap appears satisfactory, the fire is withdrawn, and the product is skimmed off and transferred to the mould.

100 lb. of tallow will produce from 150 to 155 lb. of curd soap, weighed as soon as cut.

Dr. C. R. A. WRIGHT has pointed out * that, in all pro-

* Cantor Lectures, "Journ. Soc. Arts," May 1885.

bability, hard soaps were first manufactured in this way, the use of wood ashes and fatty matters for making potash soaps of a crude character being the earliest traceable kind of soap-making, and that this mutual decomposition is available for the manufacture of hard soda soaps under circumstances when caustic soda is less readily obtainable than potashes—*e.g.*, where wood ashes are available in districts a long way from commercial centres where soda ash and caustic soda can be bought.

This old method, brought to great perfection by long experience, enabled the manufacturer to prepare an excellent soap, but the increasing price of potash and the cheapening of soda have caused it to be nearly abandoned for the modern method of saponification by soda alone.

(b) *Modern German Method.**—The boiling is conducted as follows:—The pan is charged with 190 gallons of soda-lye of 13° B., and 2000 lb. of the best melted suet. The mixture is gently boiled for two hours after it has become milky; then the heat is withdrawn, two hours' repose is allowed, and the lye is run off. Boiling with fresh lye follows, and when the soap, on pressure between the fingers, forms clean solid scales, a few buckets of lye are thrown in to cool it, and again drawn off after settling for a while. The soap is again boiled up with 9 or 10 gallons of fresh lye, and, when fusion is complete, a trial of the paste is made with the spatula. If it runs from the lye, water is added; if it does not run, it must be boiled a little longer, adding a bucket of water containing a third of its weight of common salt, in order to effect the separation of the soap. When this separation appears to be complete, after settling for about an hour, the liquid, which contains the greater part of the lye remaining from the first boiling,

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 678.

generally of a deep bottle-green colour, is drawn off. About eight buckets of water are now added, and the boiling continued till the incorporation is complete. If, on examination, the soap runs from the water, more water must be added, in small portions at a time, till the running ceases, and the pasty mass, when shaken, trembles like a gelatinous compound. The operation is finished by well boiling the contents of the pan, and, unless the soap has a bluish tinge (in which case it should have another washing), the heat is then withdrawn, the pan covered up, and the whole left at rest for a day or more. The soap is then ladled into the frames.

2. *Genuine Mottled Soap—English or London.*

The cheaper fatty matters are usually employed for this description of soap, such as bone-fat, kitchen-stuff, inferior tallow, &c. Lyes prepared from crude sodas are preferred, because the impurities which they contain materially help in the formation of the *strike*, or mottled appearance.

The process, up to the third stage, is conducted in a manner similar to that adopted for curd soap (p. 90). After this, instead of allowing the total subsidence of the *nigre*, the operator inserts a rake, breaking the paste in all directions, and then, thrusting it downwards to the lye, he draws it rapidly upwards so as to cause some of the lye to rise and spread over the surface. As it descends through the viscid mass, the dark-coloured *nigre* leaves veins or marks which, in the cooled soap, remains as mottle. When ready for cleansing into the moulds, the soap is in a gelatinous condition, interspersed throughout with lye. To judge of the proper condition for cleansing requires the experienced care of a good soap-boiler. If iron frames are used, the *ends* of the bars have often no marbling, owing to the too rapid cooling of the parts in contact with the metal, and hence

some makers prefer to use wooden frames for mottled soap.

3. *Castile, Marseilles, Venetian, or Olive-oil Soap—
White and Mottled.*

The process for the manufacture of this soap does not differ greatly from that of other hard soaps. The fatty material used is olive oil, often with the addition of poppy, cotton-seed, or other seed oil, as the soap made from olive oil alone is inconveniently hard. Of course only the cheaper kinds of olive oil are employed, the most suitable being those which contain the largest proportion of stearin, and which, consequently, most readily solidify in the cold.

The operation may be described in four stages:—

- 1°. The preliminary boiling, or *pasting* (*empâtage*);
- 2°. Cutting the pan (*relargage*);
- 3°. Clear-boiling (*coction*); and, in the case of mottled soaps,
- 4°. Mottling, or marbling (*madrage*).

1°. *Pasting*.—The lye, of 8° to 11° or 12° B. (the latter when the oil is thin, the former when it contains more solid matter) is either run into the boiler, or prepared therein by mixing weak and strong lye till the desired strength is reached. It is necessary to be particular about its strength, because, if too strong, or if the quantity of lye be excessive, the solution of the soap formed is hindered, and the first action of the oil and alkali can only take place rapidly and completely when the soap remains dissolved in the lye. This lye should also be, for this stage, as free as possible from common salt (*soft lye*); hence the purer kinds of soda are taken for the first lye, and afterwards soda containing sodium chloride (*salted lye*).

The oil is run in at once with stirring, when the lye has reached the boiling point. An emulsion is soon produced,

and any excess of oil or of lye is then noticed and treated in the manner already described (pp. 84, 85, 91, 93). As soon as the mass has become perfectly uniform, and has acquired the consistence of soap, the heat is withdrawn, and the salting process begins.

2°. *Cutting the Pan, or Salting.*—This operation is performed as detailed previously (pp. 82, 85, 91, 93), or by the use of salted lye at 25° to 30° B. The solution of salt, or the salted lye, is thoroughly mixed with the contents of the boiler. The soap, insoluble in the salt solution, separates in flocks from the excess of water, and by continued boiling, it is at length brought to a granular or curd-like condition. At this point the heat is removed, time is allowed for the lye to deposit, and the liquor is afterwards drawn off.

3°. *Clear-boiling, or Clarifying.*—The lye now added must be so strong that the soap will not dissolve in it. Its strength is accordingly 18° to 20° B., and about 10 per cent. of common salt is added. According to DUSSAUCE, it is preferable to begin this part of the process with *soft* lyes—that is, lyes free from salt. After boiling till the caustic properties of the lye are lost, the liquor is drawn off and replaced by a similar lye, and it may be necessary to repeat the treatment with fresh lye several times, till the soap has greater consistency, and the *alkalinity of the lye remains unaffected*. This shows that the soap is completed, as it will take up no more alkali. The mixture no longer boils smoothly, but in jerks, and the curd, when pressed against the palm of the hand, forms a firm and granular mass, which does not adhere to the skin.

Up to this point the details of the process are nearly the same for both white and mottled Castile soap.

If a white soap is to be produced, the impurities, such as iron compounds, &c., must be separated by further treatment as in 3°a.

3°a. *Liquefying*.—The last lyes having been drawn off, the soap is again treated with weak lye, and heated gently, so that the heavier, dark-coloured soap, or *nigre*, may sink below the lighter mass of purer soap. After settling for a sufficient time in the covered boiler, the upper stratum is ladled off into the frames, and is sometimes, as an additional precaution, poured into these through sieves, so as to keep back casual impurities. X

4°. *Mottling*.—If, instead of a white soap, the object is to produce a mottled soap, impure soda, containing sulphides, is preferred for the lye, and a quantity of ferrous sulphate (green vitriol), about 8 oz. for each cwt. of oil, is added at the end of the preliminary boiling. This is at once precipitated, partly as iron oxide and sulphide, and partly as an insoluble iron soap. In consequence of this addition, and also from the presence of iron and sulphur in the lye, and of ferruginous matters from the pan, the curd obtained at the end of stage 3° has a uniform slate colour. If this were allowed to remain, the effect would not be pleasing, but, instead of directing his endeavours to exclude these impurities, as in the case of the white soap, the soap-maker conducts the operation in such a way as to preserve and arrange them, by diffusing the colour in veins, in order to give a marbled, or mottled, appearance. When the proper consistence of the soap has been attained, the mass is worked about with rakes, so as to bring the lower and darker-coloured parts of the curd to the top. When this has been sufficiently done, the viscid soap is transferred to the frames, where, in about a week or more, according to the quantity, it cools down to mottled soap. By varying the proportion of iron sulphate added, a tint is produced of a lighter or darker hue. By exposure to the air, the iron gets oxidized to the state of sesquioxide, and a reddish tint, called *manteau Isabelle*, is diffused over the bluish mottled mass.

It is thus apparent that in mottled soap the veins and patches of heavy, insoluble, coloured compounds are present because, by special manipulation, they have been intentionally prevented from subsiding, and by the conveyance of the soap to the frames in so viscid a condition that the downward trickling of the coloured impurities should proceed so slowly as only to intensify the desired appearance, and not subside altogether. It is evident also that, if a soap so prepared were thinned by admixture with water, the impurities would more readily subside, and that the veining or mottling would be greatly diminished or entirely prevented. Hence, a *genuine* mottled soap cannot contain more than 33 or 34 or, at most, 36 per cent. of water. Hence, also, as a mottled appearance was formerly a special characteristic of "Castile" soap, and as this was essentially a good soap, a mottled or marbled character came to be regarded as a sign of excellence. So far was this belief carried, that it used to be said there was no need to analyse a marbled soap, as it must necessarily be *genuine*.* This, however, is now by no means the case.

4. *Artificially Mottled Soaps—Blue, Grey, and Red.*

BLAKE and MAXWELL's process may be used to produce these soaps. Two soap-pans are required. In one of these a known quantity of tallow, or bleached palm oil, or a mixture of 80 per cent. of cocoa-nut oil, 14 per cent. of tallow, and 6 per cent. of lard, is boiled with a quantity of soda lyes, carefully calculated by means of the second table on p. 110, and the hydrated soap thus formed is transferred to the other pan, in which a soft curd soap has been prepared from fatty matters and lyes, as calculated from the first table

* RAMPÉL'S "Method of Assaying Soaps," quoted in WATT'S "Art of Soap-making," p. 209.

on p. 110. The mottle is produced by adding to this soap, when in a finished state, colouring matter to impart the desired colour, and in about half an hour after the soaps and colouring matter have been thoroughly incorporated, the soap may be transferred to the frames. For the best descriptions of mottled soaps, the weight of fatty matters used to produce the hydrated soap amounts to from one-fourth to one-half of the fat used to produce the soft curd. For cheaper descriptions, the hydrated soap may be increased till the proportion of fat in the hydrated soap amounts to from two-thirds to one and a half times the weight of fat in the curd soap.

Another way is to prepare a "fitted" soap from the fatty mixture containing cocoa-nut or palm-kernel oil in one pan, and to remove it from the *nigre* to the second pan. Here, for every 1000 lb. of soap, are added 250 lb. of sodium silicate, and the whole is thoroughly incorporated by boiling, until the experienced workman judges that the proper condition for mottling has been attained. The colouring matters mixed with water are then sprinkled into the pan, and, after boiling for a few minutes, the mixture is transferred to the frames.

The colouring matters are—for *blue*, artificial ultramarine, 5 to 10 lb. per ton; for *grey*, manganese oxide, 1 to 3 lb. per ton; and for *red*, vermilion.

5. *Yellow, or Rosin, Soap.*

The distinctive yellow tint of this soap is due to the presence of a considerable quantity of rosin. Several methods are followed in its preparation.

1°. The ingredients are common fat, or inferior tallow, or bone-fat, or red oil, palm oil, and rosin. The proportion of rosin in this mixture should not exceed one-third of the fat; if equal parts are used, the soap produced is soft and dark

coloured. It is usual, in this country, to *partially* make the palm oil or tallow soap, and, when the saponification is nearly complete, to introduce, with the last charge of lye, the coarsely powdered rosin. The contents of the copper are then well mixed together and boiled for some hours, generally with open, or wet, steam, adding more lye whenever necessary, to preserve an excess of alkali till the completion of the saponification. This point is ascertained by cooling a portion of the soap and noting whether it then has a proper consistence and the proper grain, and whether it will wash without leaving a film of rosin on the hands.

The lyes having been drawn off, the paste is next purified, or *fitted*, by boiling up with weak lye (about 8° B.) in order to facilitate the deposition of the impurities. After resting for a while, the lye is again removed, and boiled once or twice more with still weaker lye. After a long interval—from a day or two to a week, according to the size of the pan—there is a separation into three layers: a scum, or *fob*, uppermost, the *nigre* at the bottom, and the pure soap (or *neat* soap, as it is called) in the middle. The scum is next taken off, and the soap is *cleansed*—i.e., the neat soap is removed into the frames.

The dark-coloured *nigre* may be afterwards used for mottling, or for inferior sorts of yellow soap.

2°. If tallow, or other grease, be employed, without any palm oil, the following procedure is sometimes adopted:—2000 lb. of the fat, 600 lb. of rosin, and from 150 to 175 gallons of soda lye of specific gravity 1.075 to 1.150 (15° to 30° Tw.) are run into the boiler, and, when the whole is melted, it is boiled, with continued stirring to prevent the rosin adhering to the bottom and sides of the boiler. If there is a great swelling of the mass, the heat must be

* MUSEPRATT'S "Chemistry," ii. 880.

lessened. The first boiling should be continued not more than two or three hours, on account of the ease with which the combination is effected. After six hours' repose the spent lye is withdrawn, more lye is run in, and the whole is again boiled for about three hours. Another repose of six hours is now allowed, the spent lye is again drawn off, and fresh lye afterwards added. These boilings, &c., are continued day after day till the proper consistence, which is ascertained in the manner already described (pp. 91, 95), has been attained. If the soap is not yet satisfactory, it is requisite to add more lye, and to re-boil; but if the examination shows it to be finished, it is boiled up briskly, the heat is withdrawn, about 6 gallons of lye are thrown in to cool the soap, and two hours afterwards the liquor is run off. From 12 to 16 gallons of water are now added, and the whole is again briskly boiled, stirring constantly till the soap is melted. A little of the boiling paste is now removed on a wooden spatula, and, if it run clear from the lye, more water is added, and the boiling is continued. If it should not run, too much water has already been added, and about a gallon of a strong solution of salt or of lye must be thrown in.

3°. Another and, as some think, better plan* is to make a rosin soap, or, more accurately, an *alkaline resinate*, and a tallow soap *separately*, and to mix the two in the boiler, where they are kept in a state of ebullition for some time, until a uniform mixture results. Salt is then added, and, after treatment similar to that already described, the soap is ready for the frames.

4°. **Dunn's Method.**† — Into each of the ordinary coppers, a circular ring of 1½-inch pipe, perforated with

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 686.

† Quoted in *ibid.*

holes, is fixed just far enough above the bottom to allow the free movement of a stirrer beneath it. This circular ring of pipe is supplied with air from a cylinder blast, or other suitable forcing apparatus, being connected therewith by means of a pipe which passes to the top of the copper, where it is furnished with a stop-cock and union-joint, for the purpose of connecting, or disconnecting, the parts of the pipe within and without the pan. For a clear yellow soap, 90 gallons of lyes of sp. gr. 1.14 made from strong soda-ash are introduced into the pan. The fire is kindled, and about 2050 lb. of grease are added, and, as soon as the lye boils, the blast is set in action. A brisk fire is kept up, so as to maintain the materials as near ebullition as possible. When the lyes are exhausted, more lye is gradually added until the fatty matter is *killed*. 550 lb. of fresh rosin are then added, a bucketful at a time, with more lye occasionally, until 300 gallons, of the strength above mentioned, have been used. The blast is kept in action the whole time if the fire draws well ; if not, it is advisable to stop the blast for a while before adding the rosin, to allow the mixture to approach ebullition. When the whole of the rosin is melted and completely mixed with the soapy mass, and the strength of the lyes taken up, the blast must be stopped, and a brisk boiling given. The whole is then left to rest, that the spent lyes may separate and settle. These are drawn off, and the soap brought to strength on fresh lyes, as in the ordinary process.

During the operation of the blast the soap must be kept in what is technically called an *open* or *grained* state, and for this purpose salt, or brine, is to be added when necessary. Experience proves that it is better not to make a change in the lye during the operation of the blast where lye of the strength mentioned is used, but if weaker lye is employed one or more changes may be made. It is also found

desirable that the soap should be kept in a *weak* state during the action of the streams of air through the materials; otherwise the soap is apt to swell up from the air hanging in the grain, and this is troublesome to get rid of, requiring long boiling. If dark-coloured materials are used, it is well to keep the blast in operation three or four hours after the rosin is melted, provided the soapy mass is kept weak and *open* or *grained*.

When a charge is to be worked upon a *nigre*, such *nigre* should be *grained*, and the spent lye pumped, or drawn off, as usual, and the fresh charge added in the way mentioned above, using less or more lye in proportion to the quantity and strength of the *nigre*, and taking care not to turn on the blast until there is sufficient grease present to make the *nigre* weak.

5°. **Meinecke's Method.***—This is an attempt to produce rosin from turpentine in the soap-pan which shall be at once available for making soap. The rosin is added, as it occurs in white turpentine, and this, on boiling, gives off its volatile oil, which has to be condensed and saved as an incidental product, thereby decreasing the expense of the soap. To condense the spirit of turpentine, the soap-pan must be furnished with a still-head and worm for cooling the vapours. The operation is as follows:—1000 lb. of white turpentine are melted in the copper by steam, with 800 lb. of tallow, or inferior fat, and when the mixture reaches 108° F. it must gradually receive, with constant stirring, 800 lb. of caustic-soda lye containing 30 per cent. of dry soda. The union of the materials is very rapid at this temperature; the acids of the rosin and of the fat are completely neutralized by the alkali, and converted into liquid soap. To promote the

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 687.

vaporization of the essential oil of turpentine, salt or brine is then added, the still-head luted to the copper and connected with the worm, and the contents of the copper are boiled up. The steam and oil of turpentine pass over, and are condensed. When no more oil distils over, the soap is finished in the ordinary manner.

6°. **Jennings' Method.**—To curd soap prepared with tallow or oil and caustic alkali, in the usual manner, is added about 25 per cent. of colophony, 2 to 4 per cent. of sodium carbonate, and about 1 per cent. of aluminium sulphate, common alum, or other double salt of alumina. The mixture is boiled with water till perfect combination is effected. To prevent the rosin from precipitating, a small quantity of dilute sulphuric acid (1 part acid to 9 parts water), amounting to about 2 per cent. of the fats and rosin, is stirred into the mixture.

The composition of *primrose* soap by analysis is :*—

| | South England. | North England. |
|--------------------------|----------------|----------------|
| Fatty acids | 62.3 | 42.66 |
| Soda—as soap | 6.7 | 5.41 |
| „ in other forms | 0.0 | 1.21 |
| Neutral salts | 0.2 | 0.55 |
| Silica | 0.0 | 0.94 |
| Water | 32.8 | 50.40 |
| Total | 102.0 | 101.17 |

Cost of an Ordinary Yellow Soap.—The following calculation indicates, *approximately*, the cost of *production* of 1 ton of ordinary yellow soap at the prices quoted :—

| | £ | s. | d. |
|--|-----|----|----|
| Tallow—11 cwt. at (say) 25s. | 13 | 15 | 0 |
| Rosin—3 cwt. at (say) 5s. | 0 | 15 | 0 |
| Alkali—2 cwt. 3 qrs. (58° at 1½d. per unit), 5s. 6d. | 0 | 15 | 1½ |
| Labour, &c. | 3 | 0 | 0 |
| Total | £18 | 5 | 1½ |

* CARPENTER—SPON'S "Encyclopædia," v. 1796.

6. *Cocoa-nut-oil, Marine, or "Hydrated" Soaps.*

The use of cocoa-nut and palm oils in the manufacture of soaps has increased to a great extent since artificially prepared soda came into general employment. This is well shown by the following statistics:—

| Year. | Imports of | |
|--------------|--------------------|-------------------------|
| | Palm Oil. cwts. | Cocoa-nut Oil. cwts. |
| 1820 | 17,456 | 8,353 |
| 1830 | 213,476 | 8,534 |
| 1840 | 315,503 | 42,428 |
| 1850 | 447,796 | 98,039 |
| 1860 | 804,326 | 194,309 |
| 1870 | 868,270 | 198,602 |
| 1880 | 1,026,378 | 317,828 |
| 1881 | 819,749 | 248,476 |
| 1882 | 801,545 | 136,087 |
| 1883 | 743,512 | 210,874 |
| 1884 | 825,822 | 245,695 |
| 1885 | 898,481 | 185,971 |
| 1886 | 993,091 | 156,667 |
| 1887 | 966,536 | 183,766 |

The behaviour of cocoa-nut oil differs from that of the other fatty matters in the process of saponification. It is difficult to make the saponification begin, but, once started, it goes on with great rapidity, the mixture swelling up enormously. The resulting soap can only be separated from solution in the copper by *very strong* solutions of common salt. The reason of this is that cocoa-nut-oil soap is soluble in *dilute* brine, and is, consequently, available for washing in salt water. Hence it is called *marine* soap. If, however, cocoa-nut-oil soap be prepared in this way, it contains very little water, and becomes so hard that it cannot be cut with a knife. The usual method is therefore not followed in making this soap. As weak lyes will not saponify cocoa-nut oil, the operation is commenced by employing strong lye, of about 20° B.; and, by having the

lye pure and perfectly caustic, the use of salt in cutting the pan is dispensed with. Saponification is also aided by the use of potash lye with the soda.

Pure cocoa-nut-oil soap hardens much too quickly to exhibit any distinct formation of curd, and is, consequently, incapable of marbling by itself. It is very white, translucent like alabaster, exceedingly light, and forms a good lather, but always possesses a more or less offensive odour.

Cocoa-nut oil has the very important property of combining with more water than can ever be incorporated with tallow soap. It really produces no greater quantity of actual soap than an equal weight of tallow, but it can easily be made to absorb one-third more water or lye, and, at the same time, shows no want of consistence or softness, as would be the case with other soaps.

Cocoa-nut oil is not usually employed alone, but is added to other oils for the purpose of producing quickly solidifying soaps containing a large proportion of water, which could not be obtained from tallow, &c., alone. It is even possible* to prepare soap on a large scale in a few hours without salt, and almost without fire, by the use of cocoa-nut oil and tallow, together with strong lye, by merely warming them sufficiently to melt the fat, and keeping them constantly in a state of agitation. Soap prepared in this manner has a finer appearance, and sets in the mould, so that it can be cut. It contains, however, nearly all the water of the lye, as there is very little evaporation in the pan, together with the entire amount of foreign salts, and, in the fresh state, has less resemblance to soap than to stiff dough, taking deep impressions from the thumb, and having a slimy consistence when squeezed between the fingers. When dried for a length of time, there is a copious efflorescence of salts, but it finally

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 683.

acquires the consistence of ordinary soap. "Marine" soaps are often met with containing 70 per cent. of water.

If equal parts of cocoa-nut oil and tallow are used, the smell of the former is scarcely perceptible in the soap. The boiling of such a mixture is continued till a sample exhibits the proper consistence under the thumb. Under the same conditions, tallow could not be saponified alone, but the saponification begins with the cocoa-nut oil, and the presence of the cocoa-nut-oil soap carries on the saponification of the tallow.

Blake and Maxwell's Process.—In this process it was proposed to form a soap by combining saponified materials, in the state called *soft curd*, with a *hydrated soap*, or *neutral soap not deprived of its water*.

The curd soap may be prepared in the usual way, or it may be made, as preferred by the patentees, by means of soda lyes of the strength and in the quantity mentioned below, so as to obtain a soft curd better adapted for combining with a neutral soap. The soap thus formed may be separated from the water, or excess of lyes, by means of salt, or concentrated lyes, in the usual way.

The *rosin soap* is recommended to be prepared as follows:—

About one-third of the rosin to be used is mixed with a small quantity of fatty matter, equal to from 6 to 10 per cent. of the rosin. One-third of the lyes is also mixed with the rosin, and the mixture is slowly melted. The remainder of the rosin is then added gradually, by small portions at a time, as the added portions melt, and, when the whole is melted, the rest of the lye is introduced. Increased heat is then applied till the mixture boils, and this is continued for about three hours, or till saponification is complete, when the mass will have the consistence of thick glue or paste.

The *hydrated soap* is prepared in another pan from any

of the fatty matters mentioned below, either singly, or in combination, and to it are transferred the soft curd, rosin, and tallow soaps. After boiling together for about two hours, the soaps will become thoroughly united, and the compound soap will have assumed an appearance similar to ordinary soap in process of finishing. The soap should be removed to the frames within two or three hours after it is finished, and the frames should be covered so as to retain the heat as long as practicable.

The following table shows the oily and fatty matters which may be used for making the *soft curd*, and the strength and quantity of the soda lyes deemed most suitable for speedily effecting their saponification. The weight of lye required to saponify each 100 lb. of fatty matter may be found by dividing the number of degrees by the strength of the lyes applicable to each kind of fat.

| Fat to be used. | | Quantity of Lye in Degrees Baumé. | Strength of Lye. Degrees Baumé. |
|-----------------|-------------|---|------------------------------------|
| 100 lb. tallow | require . . | 3,800° | 14°-15° |
| „ palm oil | „ . . | 3,200 | 16-18 |
| „ tallow olein | „ . . | 2,800 | 16-18 |
| „ rosin | „ . . | 2,700 | 16-22 |

The fats that may be used for making the *hydrated soap*, and the quantity and strength of the lyes required for saponification, are the following :—

| Fat to be used. | | Quantity of Lye in Degrees Baumé. | Strength of Lye. Degrees Baumé. |
|-----------------|-------------|---|------------------------------------|
| 100 lb. tallow | require . . | 3,800° | 11° |
| „ cocoa-nut oil | „ . . | 4,100 | 16-20 |
| „ palm oil | „ . . | 3,200 | 18-22 |
| „ lard | „ . . | 3,400 | 13 |
| „ tallow olein | „ . . | 2,800 | 18-22 |
| „ olive oil | „ . . | 3,000 | 16 |
| „ rape-seed oil | „ . . | 2,400 | 24-28 |
| „ linseed oil | „ . . | 2,400 | 24-28 |

7. *Silicated Soaps.*

The use of sodium silicate as an ingredient of soap was first proposed by Mr. SHERIDAN in 1835.

It has been stated* that the value of silicated soaps was first publicly and officially recognized at the International Exhibition of London in 1862, when a prize medal was awarded to W. Gossage & Sons, of Widnes; but we find the following paragraph in the "Report of the Juries, Exhibition, 1851" (p. 607):—"The soap called *silicated soap*, now manufactured extensively at Liverpool, is formed by mixing a basic silicate of soda (made by boiling powdered flint in a close vessel, under pressure, with caustic soda) with hard soap in a melted state. It appears to possess remarkable detergent properties, but is liable to feel gritty in the hand." Though they may be useful, therefore, for household purposes, they are unsuitable as toilet soaps.

Sheridan's Process.—The method of preparing the silicate is described on p. 27.

The silicate is incorporated with the soap, previously prepared in the ordinary manner, by mechanical mixture, and, when the mass has been brought into the proper state for solidifying, the whole is placed in the moulds.

Gossage's Process.—GOSSAGE's patent is dated 1854. His plan for the preparation of the silicate, which differs slightly from that of SHERIDAN, is described on p. 28.

In mixing viscous solutions of soluble glass with genuine soap, it is best to commence† the mixing by adding a portion of the solution at a specific gravity of about 1.300, and to add the remaining portions required at increasing specific gravities, so that the average specific gravity of the

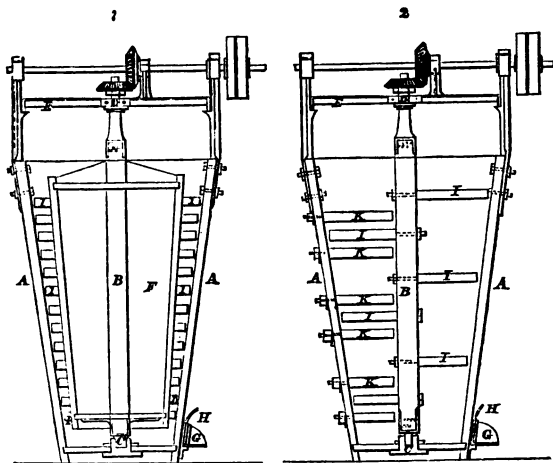
* SPON's "Encyclopædia," v. 1786.

† RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 713.

whole solution used may be equal to that which has been found by previous trials to yield a compound soap of proper hardness when using a genuine soap of the composition employed.

The temperature of the silicate and of the soap-paste should be about 160° F. at the moment of mixing, and, to promote homogeneity, the mixture is stirred up by machinery. This consists* of a large tub or vessel, *A* (Fig. 24), having the shape of an inverted cone of about 26 inches

FIG. 24.



internal diameter at its lowest part, 3 feet 6 inches at the upper part, and 6 feet deep. It is furnished with a central upright shaft, *B*, supported by a foot-step, *C*, fixed to the bottom of the tub, and, by a journal, *D*, adapted to a metallic bridge-piece, *E*, which is fixed over the vessel and secured by screw-bolts to its sides. At the upper part of

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 713.

the shaft is a bevelled cog-wheel working in gear with another bevelled cog-wheel fixed on a horizontal shaft, *S*, which is made to revolve by a band passing round the driving-pulley, *P*, and also round another driving-pulley. The upright shaft is driven at the rate of sixty to eighty revolutions per minute.

To the upright shaft *B* is fixed a closed tub or vessel, *F* (Fig. 24, 1), of such a diameter as to admit of its being placed within the larger vessel, *A*, leaving a space of about 2 inches at the lower, and 6 inches at the upper part; and to the outside of this inner vessel are attached, by means of screws or otherwise, a number of projecting blades, *II*, made by preference of sheet iron, of such a length as to approach within about $\frac{1}{2}$ inch of the inside of the vessel *A*. A spout, *G*, having a movable stopper, *H*, is adapted to the lower part of the vessel *A* for the purpose of running off its contents. The projecting blades *II*, instead of being attached to an inner vessel, may also be affixed to the inside of the upright shaft, and in that case it is best to attach other projecting blades, *KK*, to the inside of the vessel *A*, in such a manner as to allow the blades *II* to revolve between them.

When this apparatus is to be used for the production of compound soap by mixing genuine soap with the silicate solution, it is necessary to ascertain previously the highest temperature at which the mixture will become too thick to run from the mixing apparatus. For this purpose, a preparatory mixing of the neat soap with the silicate is made by means of paddles, or crutches, in a vessel capable of containing about $\frac{1}{2}$ ton of soap, the soap and viscous solution being added at such temperatures as will yield a mixture having a temperature at least 10° higher than the temperature referred to. The contents of the preparatory vessel are then transferred to the mixing apparatus, and a rapid

revolving motion is communicated to the projecting blades. The stopper of the spout *G* is then withdrawn, so as to allow the compound soap, in the state of perfect mixture, to flow from the mixing apparatus, and further quantities of mixed soap and silicate are then supplied. The mixed compound soap is then transferred to the ordinary frames, in which it solidifies on cooling.

Way's Method.—The alkaline silicate is prepared by one of the methods described on pp. 27, 28, 29.

To produce 100 lb. of soap, the operator puts into the soap-pan 11.5 per cent. of bleached palm oil, 11.5 per cent. of cocoa-nut oil, and 30.6 per cent. of soda lye of 36° Tw. These ingredients are boiled till the soap becomes stiff, and then there is added 44 per cent. of the solution of silicate of 36° Tw. The boiling is now continued till the soap becomes thin and limpid, when 2.4 per cent. of common salt is thrown in, and the boiling continued for three or four hours. After this the soap may be cleansed, either at once, or after it has been allowed to stand for a few hours.

If *open* steam is used, it is best to have the solution of silicate and the lye of greater strength than that mentioned, in proportion to the quantity of water which is condensed from such steam into the soap-pan.

Other siliceous matters, such as powdered soap-stone, porcelain earth, pipe-clay, and fuller's earth, are also used for mixing with soap instead of soluble glass.

Davis's Alk-alumino-silicic Soap* is a mixture of ordinary soap with fuller's earth, pipe-clay, and pearl-ash or soda, by which the cost of the soap is said to be much diminished, while it is claimed that its detergent properties are improved. It is prepared by adding, to every 126 lb. of soap-paste, 56 lb. of fuller's earth, slaked or dried, 56 lb. of

* RICHARDSON and WATTS, "Technology," vol. iii. pt. i. p. 714.

dried pipe-clay, and 112 lb. of calcined soda or pearl-ash, all reduced to powder, and sieved as finely as possible. These ingredients are then thoroughly incorporated by stirring or crutching. The mixing must be very perfectly and rapidly done before the pasty mass cools. To obviate any objection against the use of this soap for washing white linens, a modification of the above process is proposed, by which the use of fuller's earth is omitted, leaving the proportions, for every 120 lb. of soap, 112 lb. of dried pipe-clay and 96 lb. of calcined alkali. A soap thus prepared is said by the patentee to be useful for general purposes at sea, and for washing white linen in *salt* water.

For the preparation of a soap for washing white linen in *fresh* water, the process is still further modified by using 112 lb. of soap-paste, 28 lb. of dried pipe-clay, and 36 lb. of calcined soda; and to prepare a toilet soap, either for fresh or salt water, 28 lb. of fuller's earth, slaked or dried, and 20 lb. of calcined soda are mixed with 112 lb. of perfumed curd soap.

DUNN devised and patented a special boiler for combining soap with sodium and potassium silicates under pressure (see p. 65). The soap is prepared from tallow 7 parts, palm oil 3 parts, rosin 3 parts, caustic-soda lye (21° B.) 140 to 150 gallons. These having been placed in the boiler, heat is applied till the pressure is sufficient to permit the temperature in the boiler to rise to 310° F. This temperature is maintained for an hour, and the soap is then discharged into the vessel at the side of the apparatus. The silicate is prepared as previously described (p. 29).

8. Sulphated Soaps.

These are prepared by a process patented by Dr. NORMANDY. The object is to impart hardness to soaps made from inferior fats, and also to soaps containing large

quantities of rosin. Without this addition, such soaps are apt to be too soft, and, dissolving too freely in water, are very wasteful. The process thus enables a large class of fats, otherwise unsuitable, to be employed in soap-making.

The soap is first prepared in the usual manner, and when ready for cleansing, the salts are crutched in. For every 80 lb. of soap the proportions are 28 lb. of sodium sulphate (Glauber's salt) and 4 lb. of potassium carbonate, or 2 lb. of potassium carbonate and 2 lb. of sodium carbonate. When the whole has been thoroughly mixed, the soap is ready for the frames.

Another process for the preparation of salinated soaps is that patented by NORMANDY and SIMPSON. The soap may be prepared in the usual way from tallow, bone-fat, lard, palm oil, &c., and after the soap has been *curded* by means of salt, or strong lye, the lye is allowed to settle down, and, after it has been drawn off, a certain quantity of fresh lye and cocoa-nut oil is added, and the whole well boiled till a homogeneous mass results having the appearance, except as regards colour, of *fitted* yellow soap. The desired proportion of sodium sulphate, sulphite, or hyposulphite is next introduced, the mixture boiled, and the soap afterwards transferred to the frames. This method, it is claimed, will yield a mottled soap of better consistence and appearance than is obtainable from the same fatty materials in the ordinary way, and without separation of lyes in the frames.

Sodium hyposulphite crutched into the soap increases its hardness, like the sulphate, and the soap so treated is less liable to effloresce. It has also the property of removing the chlorine, which bleached fabrics have a tendency to retain, and by which they are exposed to deterioration.

HOFFMANN, MILLER, URE, and MUSPRATT all commend the usefulness of this process, but these soaps are not so much used as formerly.

CHAPTER VIII.

TOILET, OR FANCY, SOAPS.

THE manufacture of these soaps is either carried on as a separate business, or as a branch of the ordinary soap-maker's work, or of the perfumery business. The *stock* soap is either a specially prepared article—cold-process soaps being largely used—or a soap prepared in the ordinary manner.

It will be convenient to consider this branch of soap-making under the following heads:—

1°, *The materials*; 2°, *The apparatus*; 3°, *The manipulation*; 4°, *Formulæ*; 5°, *The French system*.

1°. The Materials.

These are chiefly white curd soap, fitted soaps, and soaps prepared from palm and almond oils. Cocoa-nut-oil and rosin soaps are also used for some toilet soaps. They should all be of superior quality.

2°. The Apparatus.

a. For the Preparation of the Soap.

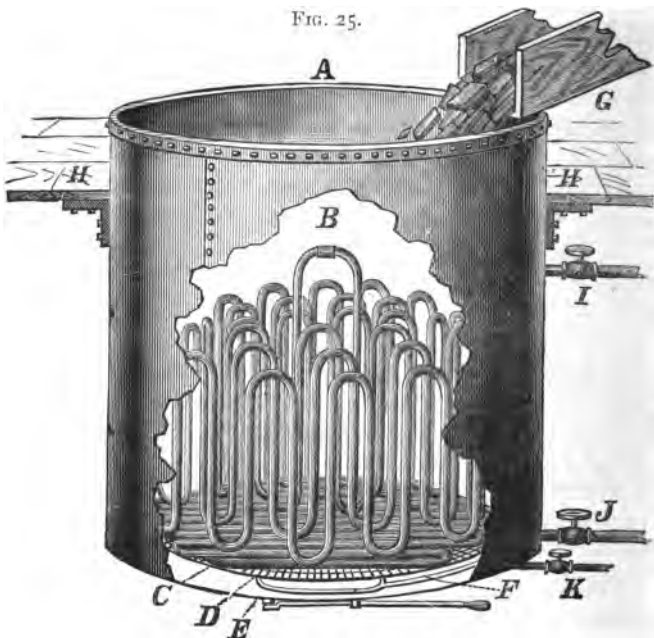
The selected soaps have first to be sliced. This is accomplished either by a cutter, or planing-knife, fixed on a strong wooden bench, and furnished with a drawer to receive the soap shavings as they are sliced off from the bars. (Fig. 30, p. 136.)

Or a cutting machine turned by a handle may be employed where larger quantities are operated upon. The bar of soap is pushed down an inclined plane against the edge of one of the blades, the handle is turned, and the shavings fall into a box placed underneath. By this machine, 2 cwt. of soap may be cut in an hour. (Fig. 31, p. 136.)

b. For Re-melting the Soap.

The most convenient pans for this purpose are small steam-jacketed pans, of 2 cwt. to $\frac{1}{2}$ ton capacity, according

FIG. 25.



A, The shell. *B* and *C*, Steam-coils. *D*, Grating for soap to rest upon. *E*, Discharge-gate. *F*, Small pipe for admitting direct steam through perforations. *G*, Feed-spout. *H H*, Floor. *I*, Dry steam. *J*, Exhaust steam. *K*, Open steam.

to the extent of business; or a WHITAKER re-melter (Fig. 25) may be employed.

The method of using this apparatus is as follows:—Fill the re-melter with soap, close the discharge-gate, *E*, and let the open and dry steam on for ten minutes. Then shut off the open steam, open the discharge-gate, and run off the soap into the steam *crutcher* till the latter is full, and run the *crutcher* from three to five minutes until the soap is thoroughly mixed. As fast as the soap lowers in the re-melter, add more stock, so as to keep the vessel full, as the soap will thus melt more quickly. The open steam should be let on two or three times, for ten minutes at a time, while filling the *crutcher*, some kinds of soap requiring more than others.

This machine is also used for re-melting soap scraps, with the object of saving *fillings*, such as sodium silicate, talc, and other substances.

c. Frames.

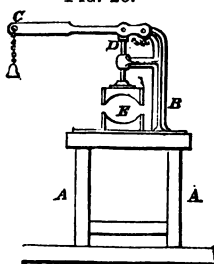
These are smaller than those used for household soaps.

d. Moulding.

For moulding the tablets, various kinds of apparatus are employed. Fig. 26 will give the reader an idea of the *modus operandi*.

A A is a table to which the press is fastened by bolts and screws. *E* is the lower portion of the mould; the upper portion is attached to the piston *D*, which is worked by the lever *C*, connected with the cast-iron pillar *B*.

FIG. 26.

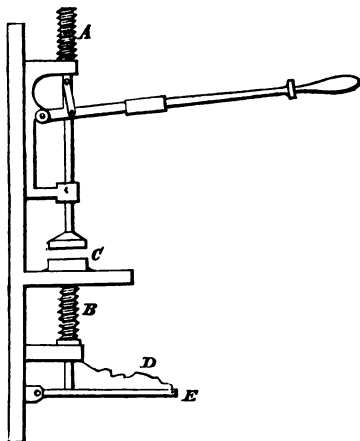


e. Stamping.

For this purpose the press Fig. 27 may be employed.

In this machine* there are two spiral springs, *A* and *B*, by which the cake of soap is immediately expelled from the box *C*, as soon as it is pressed. *D* is a rope suspending a

FIG. 27.



wooden rod, *E*, which serves as a support to the bottom of the die during the pressure. The box *C* is movable, being merely fastened by screws, and, when necessary, may be replaced by others of different sizes. The die from which the tablet is to receive a device, or name, is screwed to the top of the box *C*, and may also be changed when required.

Another form of stamping-press is that shown in Fig. 28, which is worked by hand.

Fig. 29 represents a stamping-press worked by steam.

3°. The Manipulation.

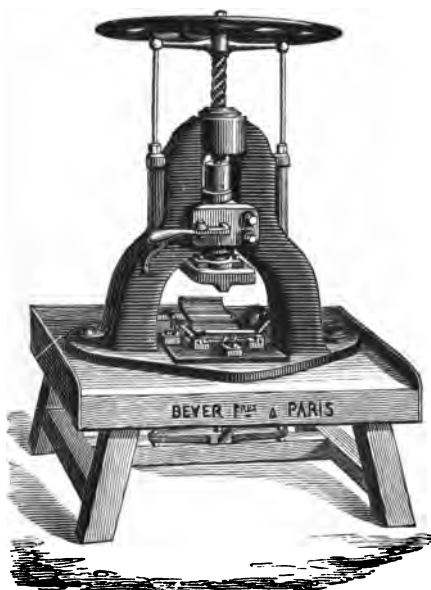
The numerous varieties of fancy soaps may be classed as (a) *opaque* and (b) *transparent*.

* MORFIT, p. 185.

a. Opaque Toilet Soaps.

In the manufacture of these soaps, the operator may either (A) prepare the article directly by the *little-pan* or *cold process*; or (B) he may re-melt and refine, and afterwards perfume soaps prepared in the ordinary manner. Or, to prepare the finest toilet soaps, he may adopt the French system (p. 136).

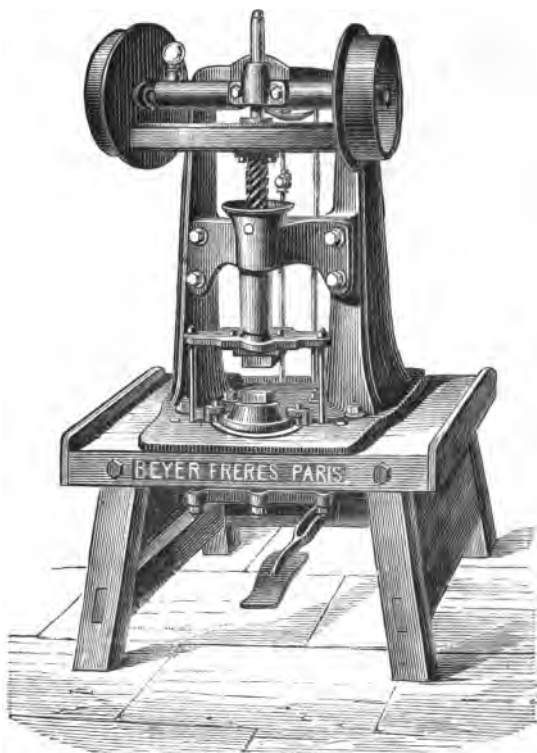
FIG. 28.



A. COLD PROCESS.—The selected fats, such as clarified beef marrow, clarified lard, sweet-almond oil, cocoa-nut oil, castor oil, and other fats of good quality, are melted together, and, if necessary, strained. Some makers now add

the alkaline lye to the melted fat; others heat the lye, and add the melted fat to it. In either case the added materials are introduced gradually with continual stirring, and care is

FIG. 29.



taken that the temperature does not rise much above 150° F. (65.5° C.). The next stage is to run the soap into the cooling-frames, and allow it to repose.

The low temperature at which this operation is conducted

is extremely favourable to the use of delicate perfumes. These are, of course, best introduced at as late a stage of the process as possible, so as to prevent loss, yet before it is too late to secure complete admixture with the mass.

The advantages and disadvantages of the cold process have been already specified (p. 88).

B. RE-MELTING.—The soap for this purpose should be a good yellow fitted soap of recent manufacture, and as neutral as possible.

1°. After having been sliced by one of the machines previously mentioned (p. 117), it is transferred to the melting-pan. It must not, however, be all put in at once, but, after the first portions have been melted and *crutched*, so as to produce uniformity, a little more of the cut soap is added, the pan covered till this has also become fluid, and the whole again stirred. Other portions are then introduced, and successively melted and crutched as before, so as to effect intimate mixture. When the paste begins to cool, the desired colouring matters are mixed with it, and afterwards the selected perfume, reserving the latter to the last so as to avoid any unnecessary loss by evaporation. At this stage also, if desired, a portion of glycerin may be introduced.

2°. FRAMING.—The soap is now ready for the frames, into which the pasty mass may be transferred by ladles. The frames are covered with cloths, so that the cooling may be gradual.

3°. FINISHING.—In a day or two it will be sufficiently hard to cut into bars and tablets of any desired size. The cakes are then *trimmed* at the edges and corners, moulded, and stamped.

Savonettes, soap-balls, or wash-balls are shaped by rotating blocks of soap upon a *soap-scoop*, made of brass with sharp edges, or the paste may be first formed into

balls by hand, and, when quite dry, finished by turning them with a lathe.

The surface of tablets or of savonettes may be polished, either by rubbing with a little spirit on a cloth, or by exposure to the action of wet steam for a few seconds.

b. Transparent Soaps.

Two methods are in use for the manufacture of transparent soaps—(1°) Solution of *stock* soaps in alcohol; (2°) The *cold* process.

1°. PREPARATION BY SOLUTION OF SOAP IN ALCOHOL.—It has been long known that a concentrated hot solution of soap retains its transparency on cooling. This fact is applied to the production of transparent soaps. As any non-soapy matters that may be present in the stock are, with the exception of free caustic alkali, insoluble in strong spirit, a transparent soap properly prepared by the alcoholic process from a good soap is necessarily of a high degree of purity, and is justly valued for toilet purposes. Makers do not all operate in exactly the same way, but the following is an outline of the process generally:—

1. Yellow soap of good quality, reduced to shavings, and dried, is introduced into a still of sufficient capacity together with alcohol (strength about 55 to 60 o.p.). Sometimes the shavings are previously powdered, and in this country, owing to the high spirit duty, methylated spirit, instead of pure alcohol, is employed, in the proportion of about 5 gallons to 1 cwt. of dried soap. Most makers also add a certain proportion of glycerin. The still is heated by steam, or by a hot-water jacket, as the direct action of fire would interfere with the appearance of the product.

2. Moderate heat is continued till about one-fifth to one-third of the spirit has passed over.

3. The clear residue, free from any deposited matters, is

run into moulds to form bars, and when these are cold they are cut into cakes.

4. The cakes, after drying sufficiently, are bevelled, polished, and stamped.

The cakes are not at first transparent, and require to be kept in the drying-room for some months before they are ready for sale. During this time evaporation of the remaining alcohol and water takes place, the colour deepens, and much of the odour of the methylated spirit goes off. If too much spirit is left in the soap at first, it is liable to become opaque, and, if there is too little, the soap will not harden properly. The finished soap contains only from 9 to 12 per cent. of water, and no spirit.

Scents and colouring matters, when desired, are mixed with the dissolved soap at the commencement of the process. The colouring matters are introduced in alcoholic solution—for *red*, tincture of alkanet; for *yellow*, tincture of turmeric, annatto, or saffron; for *orange*, a mixture of alkanet and turmeric; for *green*, tincture of chlorophyll, or a mixture of blue and yellow; for *blue*, tincture of indigo-carmin; &c.

2°. THE COLD PROCESS.—Certain kinds of soaps* prepared by the cold process, especially castor-oil soap, have a natural tendency towards a somewhat transparent appearance, which is increased by the addition of spirit, glycerin, sugar, or petroleum. With the employment of a considerable proportion of sugar (15 to 30 per cent.) a comparatively large amount of tallow is admissible without interfering with the transparency, provided that *complete* saponification is insured. Dr. WRIGHT gives the following formula for the production of a transparent soap by this process, which will be without great excess of free alkali or of sugar:—

* Dr. C. R. A. WRIGHT, Cantor Lectures, May 1885, p. 25.

Heat to 149° F. (65° C.) a mixture of tallow 20 parts, palm oil 12 parts, castor oil 8 parts, and then gradually run in 20 parts of caustic-soda lye at 38° B. When intermixed, crutch in 20 parts of strong alcohol, 20 parts of glycerin, and 10 of syrup containing half its weight of loaf-sugar. Colours and perfumes may be added as desired.

As an illustration of the materials sometimes used in this class of soaps, WRIGHT quotes the following formula :*—

Melt the following with agitation :—10 kilos. cocoa-nut oil, 10 kilos. castor oil, 8 kilos. neutral tallow, and saponify them at 122° F. (50° C.) with 14 kilos. of caustic soda at 38° B., and continue stirring until pastiness sets in. Add 8 kilos. loaf-sugar in 8½ litres of water at 185° F. (85° C.), taking care to bring it in gradually. As soon as the soap begins to solidify at the sides, the boiler is jacketed with a water-bath, kept at 176° F. (80° C.), until the soap has attained the proper consistency and the scum has separated. Add 20 to 30 per cent. of *loading*, agitate well, and then stir in a boiling solution of 1 kilo. crystallized soda in a litre of water; dye, perfume, and finish off the batch as usual. The *loading* is made from mineral oil and soap shavings, the petroleum being previously deodorized by means of bleaching-powder solution and hydrochloric acid, and subsequent treatment with chalk to remove adhering acid. 30 kilos. of the oil thus purified are heated to 122° F. (50° C.), mixed with 2 kilos. of well-dried soap shavings, and heated until a sample taken out solidifies on cooling.

On this formula WRIGHT makes the following useful observations:—"It is evident from the above that even without the *loading* the resulting mass would not contain as much as half its weight of actual soap, for the ingredients consist of 28 kilos. fatty glycerides (representing a little

* "Journ. Soc. Chem. Ind." April 1883.

more than the same weight of anhydrous soda soap—about 29 kilos.) and $32\frac{1}{2}$ kilos. of water, soda, and sugar, so that, when 30 per cent. of loading is added, the resulting mass would not contain much more than one-third its weight of actual soap. On the other hand, the total alkali used (partly as caustic-soda solution, partly as crystals) represents about 113 per cent. of the amount chemically equivalent to the fatty matters, furnishing, consequently, a soap with an excess of *free alkali* equal to one-eighth of that combined as soap—a quantity very far in excess of that compatible with good quality as regards injurious action on tender skins. The quantity of sugar prescribed represents some 13 per cent., reckoned on the mass without loading, and about 27 per cent. of the actual soap formed.

“This formula, apart from the loading, results in the production of an article of distinctly better quality than most of the transparent soaps of this kind now sold in Great Britain, for these soaps usually contain a still larger excess of alkali (ranging from 15 to 25 per cent., and even more being often found), whilst the amount of actual soap in tablets fresh from the factory (and not dried by exposure in shop windows) rarely exceeds 45 per cent., so that these articles are about as much a compound of sugar-candy and soda crystals as they are soaps, if not more so.”

These soaps are often termed *transparent glycerin* soaps.

The following formulæ are said to give satisfactory results:—

1. Melt together 500 parts of suet, the same quantity of Ceylon cocoa-nut oil, 250 parts of castor oil, 50 parts of palm oil, and 500 parts of glycerin. Saponify the mixture at about 75° C. with 650 parts of soda lye of 1.38 sp. gr. The soda solution should be added gradually, and the whole well stirred during the saponification, which will be completed in about five minutes. The soap is now removed

from the source of heat, and mixed with 600 parts of strong alcohol (or methylated spirit), the whole being well stirred until it is clear. 150 parts of simple syrup are then added, together with the perfumes. It is then poured into moulds.*

2. 20 lb. tallow, 12 lb. palm oil, 8 lb. castor oil, 20 lb. 38° lye, 20 lb. 96 per cent. alcohol, 20 lb. glycerin, 5 lb. sugar dissolved in 5 lb. water. Heat the tallow and palm oil, add the lye, and saponify; then add the alcohol, and, when the combination is complete, introduce the glycerin. The soap may be perfumed with oil of bergamotte 250 grams, citron 90 grams, lavender 20 grams, neroli 30 grams, rosemary 5 grams, and a few drops of otto of roses dissolved in 1 lb. of 96 per cent. alcohol and coloured with saffron substitute.†

CRISTIANI‡ gives the two following formulæ:—

3. *Transparent Soap*.—Tallow 209 lb., caustic-soda lye 40° B. 94.6 lb., alcohol 110 lb.

To the melted grease add one-half the alkali, keeping the heat as low as possible—about 120° F. When, with constant stirring, the fresh lye is combined, add the remainder of the lye, to which has been previously added the alcohol, the heat being well regulated. Saponification takes place rapidly. Add the perfume, cool, pour into the frames, and continue the cooling very gradually. The transparency will not be apparent till the soap has been exposed to the air for some time. To perfume the quantities given above, 2.2 lb. of mixed essences will be required.

4. *Transparent Glycerin Soap*.—Tallow (mutton) 44 lb.,

* "Pharm. Zeitung," 1879, p. 719; "Year Book of Pharmacy," 1880, p. 344.

† "Seifensieder Zeitung," 1884, p. xxiii.

‡ "Treatise on Soap and Candles," pp. 422, 423.

cocoa-nut oil 44 lb., castor oil 22 lb., glycerin (pure) 22 lb., caustic lye 40° B. 57 lb., alcohol (96 per cent.) 48.4 lb., water 9.9 lb.

Melt the grease at 104° F., and add the alkali gradually, keeping the heat low to prevent evaporation, and stir constantly. When the lye has been absorbed, after three or four hours' stirring, add the alcohol, which should be warmed, and stir till the whole becomes cool. Then add the glycerin, and, when this has been mixed, the water and perfumes. Turn into frames, pouring slowly. Very superior, if well made.

A cheap transparent soap may be made as follows:—Cocoa-nut oil 10 kilos., castor oil 10 kilos., tallow 8 kilos., caustic-soda lye 38° B. 14 kilos.

Saponify at 112° F. (50° C.), and stir till pasty. Then add gradually 8 kilos. loaf-sugar in 8½ litres at 185° F. (85° C.); cool and frame.

4°. Formulæ.

Ammoniated Soap.†—A soap made from 8 parts of stearic acid, 4 cocoa-nut oil, 1 potash, 1 soda, 6 water, is cut into shavings and placed in a retort, in which it is subjected to the action of gaseous ammonia, at a pressure of 15 lb. per square inch, till thoroughly permeated by it.

Almond Soap.—Oil of almonds by weight 21 oz., solution of caustic soda (sp. gr. 1.334) by weight 10 oz. Add the lye to the oil in small portions, stirring frequently, leave the mixture for some days at a temperature of from 64° to 68° F., stirring occasionally, and, when it has acquired the consistence of a soft paste, put it into moulds till

* "J. Soc. Chem. Ind." 1883, p. 181.

† C. R. HUXLEY, English patent 3441, March 17, 1885.

sufficiently solidified. It should be exposed to the air for one or two months before it is used.

Beef-marrow Soap.*—To 500 lb. of beef marrow add 250 lb. of caustic-soda lye of 36° B., stir constantly and gently, and heat the mass till it becomes soluble in water. In this state dilute with 2000 parts of boiling water, and pour in 1000 parts of brine (containing 180 parts of common salt), with constant stirring. After allowing some time for repose, pour into the frames, and leave for a day or two to set thoroughly.

Bitter-almond Soap.—Pure white soap 10 kilos., oil of bitter almonds 120 grams. Not coloured.

Or, white tallow soap 56 lb., oil of almonds $\frac{1}{4}$ lb. For inferior kinds, *nitro-benzol* is employed instead of oil of almonds.

Floating Soap.—Good oil soap 14 lb., water 3 pints. Melt together by aid of steam or water bath, and assiduously beat together until the mixture has at least doubled its volume. The capacity of the pan for 14 lb. of soap should be about 18 gallons. Frame and cool. The thickness of the soap in the frames should not be more than 6 or 7 inches. In about a week or less it will be ready for cutting. Perfume, as desired. Colour with $\frac{1}{2}$ to 1 drachm of vermilion per lb.

Glycerin Soap.—Any mild soap, being melted, has glycerin intimately mixed with it in the proportion of $\frac{1}{10}$ th to $\frac{1}{25}$ th of the weight of the soap.

Perfume with oil of bergamotte or rose-geranium mixed with a little oil of cassia, to which sometimes a little oil of bitter almonds is added.

Honey Soap.—White Marseilles soap 4 oz., honey 4 oz., benzoin 1 oz., storax $\frac{1}{2}$ oz. Mix well in a marble mortar. When thoroughly mixed, melt over a water bath, pass

* MORFIT, "Treatise on Soap," p. 244.

through a fine sieve, and run into moulds. Divide into cakes.*

The article commercially vended under this name rarely contains any honey. It may be prepared as follows:—

Palm-oil soap and olive oil of each 1 part, curd soap 3 parts; melt together.

Perfume with oil of verbena, rose-geranium, or ginger-grass.

Or, a neat yellow soap is mixed with 5 per cent. sodium carbonate, or silicate ($59\frac{1}{2}^{\circ}$ B.), the whole crutched, and perfumed with oil of citronella.

Lard Soap.—This soap is prepared by the cold process, as follows:—Melt 112 lb. of lard by gentle heat, and add half the lye, prepared by dissolving 56 lb. of caustic soda to mark 36° B. Agitate well without allowing the mixture to boil, and when the incorporation is complete the remainder of the lye is gradually introduced. The temperature is kept under 149° F. When the paste has sufficient consistence, and has no greasy feel when pressed between the fingers, it may be run into the frames. The desired perfume is added while the soap is in the pasty state. In about two days it will have become sufficiently solid to be cut into tablets and pressed. This soap is very hard, and of a brilliant whiteness.

Miahle's Neutral Soap.—In a communication to the French Academy,† M. Miahle describes a soap which he states combines the advantages of being prepared without heat, and thus avoiding the loss of the glycerin in combination with the fatty matters, and of being free from that alkalinity generally present in soaps prepared in the cold. In its preparation the ordinary toilet soap, made without

* DUSSAUCE, "Treatise on the Manufacture of Soap," p. 638.

† "Pharm. Journ." iii. 665.

heat, is cut into shavings and exposed, in a properly closed chamber, to the action of carbonic acid gas. The soap absorbs a quantity of the gas proportionate to the quantity of caustic soda which has escaped saponification, and by the transformation of the free alkali into bicarbonate it loses all its causticity. It then constitutes a perfectly neutral soap, containing all the glycerin of the fatty bodies employed in its manufacture, and a certain quantity of bicarbonate of soda.

Samphire Soap is Messrs. Field's recently patented article, which is saponified by the use of iodized potash, obtained from seaweed ashes, with palm oil and olein. The resulting soap is subsequently milled, after completely expelling the water, and is de-alkalized by the introduction of an ammoniacal salt.

Savon au Bouquet.—White tallow or lard soap 10 kilos.*

Perfume with oil of bergamotte 15 grams, neroli 15 grams, sassafras 10 grams, thyme 10 grams.

Colour with 100 grams brown ochre. The oil of neroli may be replaced by oil of lavender, and oil of cloves, 10 grams, may also be added.

Savon à l'Huile de Cannelle (*Cinnamon Soap*).—Pure palm soap 5 kilos., tallow soap 5 kilos.

Perfume with oil of Chinese cinnamon 80 grams, sassafras 20 grams, bergamotte 30 grams.

Colour with 80 grams yellow ochre and 20 grams burnt sienna.

For inferior descriptions, oil of cassia is used instead of oil of cinnamon.

Savon au Fleur d'Oranger.—White tallow soap 6 kilos., pure palm soap 4 kilos.

* 1 kilogram = 2.20 lb. Avoir.

Perfume with oil of Portugal 140 grams, oil of amber 10 grams. Or with oil of geranium 40 grams, oil of neroli 50 grams.

Savon au Musc.—White tallow soap 5 kilos., pure palm soap 5 kilos.

Perfume with oil of bergamotte 50 grams, roses 5 grams, cloves 5 grams, musk 10 grams.

The musk is prepared thus :—Pound 10 grams of musk in a mortar, with an equal weight of sugar, and 5 grams of pure potash; then add 160 grams of alcohol gradually, triturate for a quarter of an hour, pour the mixture into a flask, and leave it for two to four weeks, shaking it from time to time. Then filter, add the whole of the filtrate to the 10 kilos. of soap, and afterwards the other perfumes.

Colour with 80 grams brown ochre.

Savon à la Rose.—White tallow or lard soap 10 kilos.

Perfume with oil of roses 40 grams, cloves 15 grams, cinnamon 10 grams, bergamotte 30 grams, neroli 10 grams. Or with oil of roses 25 grams, geranium 60 grams, cloves 15 grams, Chinese cinnamon 10 grams.

Colour with 60 or 80 grams of vermilion.

Savon à la Vanille.—White tallow soap 10 kilos.

Perfume with tincture of vanilla 500 grams, oil of roses 5 grams.

Colour with 100 grams of burnt sienna.

Savonettes, or Wash-balls.*—These are made of any of the mild toilet soaps, scented at will, and sometimes with the addition of starch. The spheroidal form is given to them, as described on p. 123.

1. Curd soap 3 lb., finest yellow soap 2 lb. (both in shavings), soft water $\frac{3}{4}$ pint. Melt by a gentle heat, and stir in powdered starch $1\frac{1}{2}$ lb. When the mass has con-

* COOLEY'S "Encyclopædia," ii. 1464.

siderably cooled, add essence of lemon or bergamotte $1\frac{1}{2}$ oz., and make into balls.

2. *Camphor*.—Melt spermaceti 2 oz., add camphor cut small 1 oz., dissolve, and add the mixture to white curd soap $1\frac{1}{2}$ lb., previously melted by the aid of a little water and gentle heat, and allowed to cool considerably. These balls should be covered with tin-foil.

3. *Honey*.—Finest yellow soap 7 lb., palm oil $\frac{1}{4}$ lb. Melt, and add oil of verbena, rose-geranium, or ginger-grass 1 oz., or oil of rosemary $\frac{1}{2}$ oz.

4. *Mottled*.—(a) *Red*: Cut white curd or Windsor soap (not too dry) into small square pieces, and roll these in powdered bole or rouge, either with or without the addition of some starch; then squeeze them strongly into balls, observing to mix the colour as little as possible. (b) *Blue*: Roll in powder blue, and proceed as before. (c) *Green*: Roll the pieces in a mixture of powder blue and bright yellow ochre.

By varying the colour of the powder, mottled savonettes of any colour may be produced.

5. *Sand*.—Soap (at will) 2 lb., fine sand 1 lb.; perfume if desired. For finer qualities, finely powdered pumice-stone is substituted for sand.

6. *Violet*.—Palm-oil soap 4 lb., starch 2 lb., finely powdered orris root 1 lb.

Shaving Paste.—1. Naples soap 4 oz., powdered Castile soap 2 oz., honey 1 oz., essence of ambergris and oils of cassia and nutmegs of each 5 or 6 drops.

2. White wax, spermaceti, and almond oil of each $\frac{1}{4}$ oz.; melt, and, whilst warm, beat in two squares of Windsor soap, previously reduced to a paste with a little rose water.

3. White soft soap 4 oz., spermaceti and salad oil of each $\frac{1}{2}$ oz.; melt together and stir till cold. Scent at will.

When properly prepared, these pastes produce a good

lather, with either hot or cold water, which does not dry on the face.

Windsor Soap.—*Plain.*—The best kind is made from olive oil 1 part, tallow 8 or 9 parts, saponified with caustic-soda lye, and scented, after removal from the pan, with oil of caraway and a little oil of bergamotte, lavender, or origanum, in the proportion of about 2 lb. of the mixed oils per cwt. of soap. A little oil of cassia, or of almonds, or of the essences of musk and ambergris may be also added. The oil of caraway may be replaced by a mixture of equal parts of the oils of rosemary and lavender.

Ordinary plain Windsor soap is made from curd soap, scented, while pasty, with oil of caraway, and a little oil of bergamotte, lavender, or origanum, in the proportion of about 1½ lb. of the mixed oils per cwt.

Brown.—The colour of this variety was originally the effect of age upon the plain white soap, but is now produced by the addition to the above of a little brown colouring matter, such as caramel, umber, or brown ochre.

*Weise's Formula.**—40 lb. tallow and 15 to 20 lb. olive oil are saponified with soda lye of 19° B., and the soap is treated with lye of 15°, and finally with lye of 20°, the process being conducted as for a curd soap, except that no excess of alkali is to be used. When boiled clear, the soap is left in the boiler for six or eight hours, then completely separated from the lye, placed in a flat mould, and pressed till it no longer exhibits any flux, to prevent it from mottling. To perfume the above-mentioned quantity, add oil of cumin 10 oz., oil of bergamotte 6 oz., oil of lavender 3 oz., oil of origanum 1 oz., and oil of thyme 3 oz.

Another formula is the following:—Hard curd soap (made from good tallow 9 parts, olive oil 1 part) 100 oz.,

* "Dingl. Polyt. J." cxxxv. 237.

scented with oil of caraway 1 oz., oil of lavender $\frac{1}{4}$ oz., and oil of rosemary $\frac{1}{4}$ oz.

Rose Windsor is the plain variety coloured with vermillion or iron oxide, and perfumed, after the soap has been transferred to the frame, with essence of roses.

*Violet Windsor** is composed of 50 parts of lard, 33 parts of palm oil, and 17 parts of spermaceti, perfumed with essence of Portugal and a little oil of cloves.

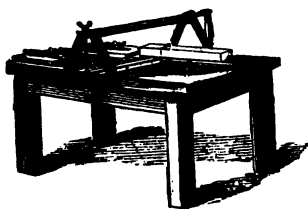
5°. French System.

The French have devised special machinery for the manufacture of the finest kind of toilet soaps. The "stock" soap, or basis, should be made from the purest materials. Usually, it is prepared by the *cold* process.

The mode of procedure is as follows:—

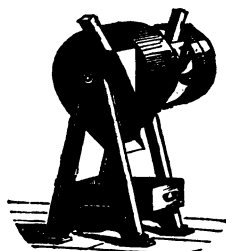
1°. CUTTING.—The soap having been cut into bands by the *hand cutter* (Fig. 30) is passed to the *rotary cutter*

FIG. 30.



BEYER'S hand cutter.

FIG. 31.



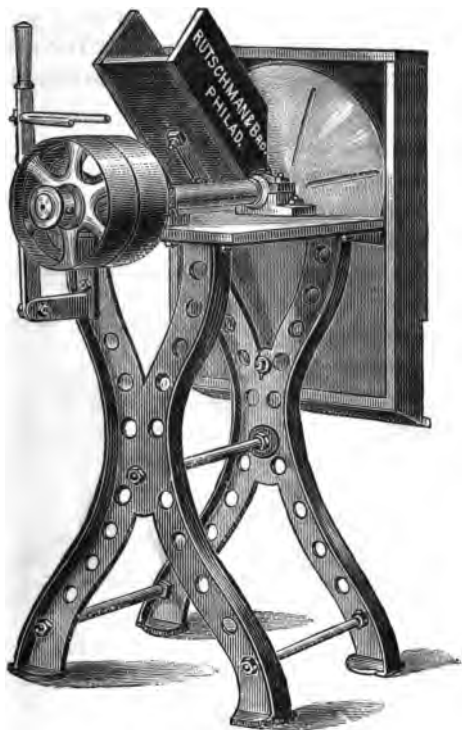
BEYER'S rotary cutter.

(Fig. 31), or to a RUTSCHMAN automatic soap-chipper (Fig. 32), by which it is reduced to thin shavings. This machine is usually placed in the drying-room, in order that

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 707.

during the process the shavings may become somewhat drier,

FIG. 32.



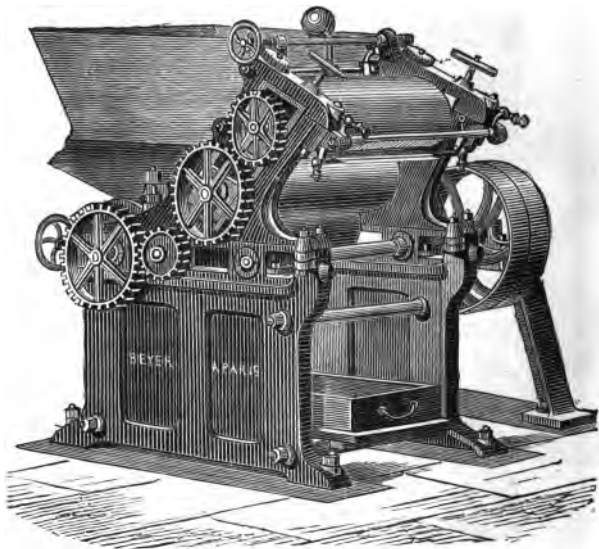
Automatic soap-chipper.

2°. CRUSHING AND GRINDING.—The dry shavings are now ready to be placed, with the desired perfumes and colouring matters, in the hopper of the *crushing-mill*, Fig. 33 or Fig. 34.

This machine is mounted on a frame cast in one piece, and carries three or four granite rollers. The motion of

the rollers draws the soap shavings between the first and second rollers, which are so geared that the second revolves more quickly than the first, and the soap is thus not only crushed, but also undergoes a rubbing action. The increased speed of the second roller has the effect also of passing the crushed material along so as to place it between

FIG. 33.

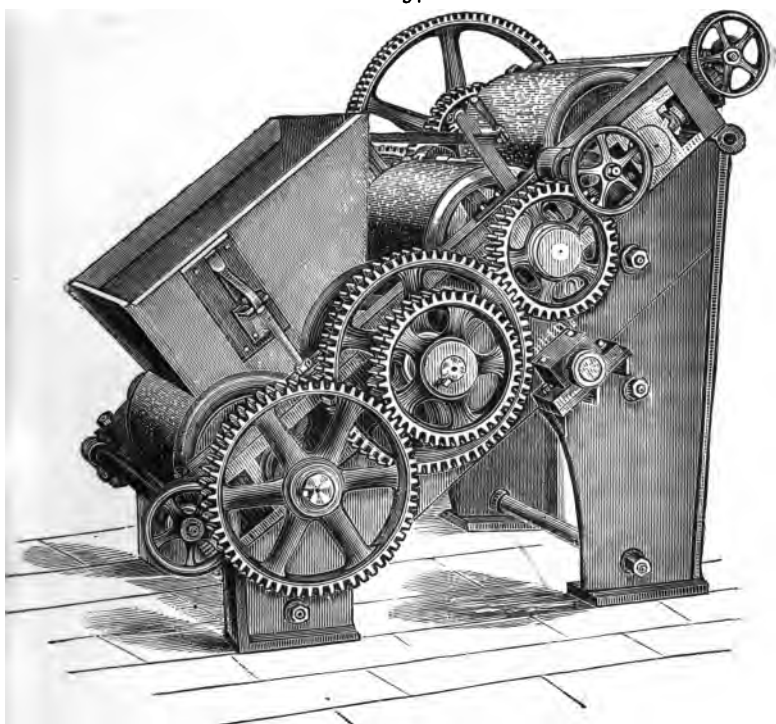


BEYER'S crushing-mill.

the second and third rollers, where it undergoes a second crushing. The third roller, revolving at a still higher speed than the second, causes the soap to be seized and crushed again between the third and fourth rollers. The soap paste is removed from the last roller by a steel scraper, and returned to the hopper, from which it is again passed through the mill. This triple crushing by the suc-

cessive passing of the soap between the rollers is technically called in France *passee* (passage). Each passage of 30 kilograms (about 60 lb.) occupies five minutes.

FIG. 34.



RUTSCHMAN'S crushing-mill.

Three or four passages, or more, are generally required to effect perfect amalgamation of the mass, the exact number depending on the nature of the materials. When the workman judges that the operation is finished, he presses a button, acting on two scrapers, and these fall

in front of the fourth roller, and the separated ribbons of soap are received in a small waggon, by which they are conveyed to the plotting and squeezing machine (*boudineuse-peloteuse*). In some factories, however, the crushing-mill is so placed that the ribbons can be directly thrown from it to the feeding-hopper of the plotting machine.

FIG. 35.

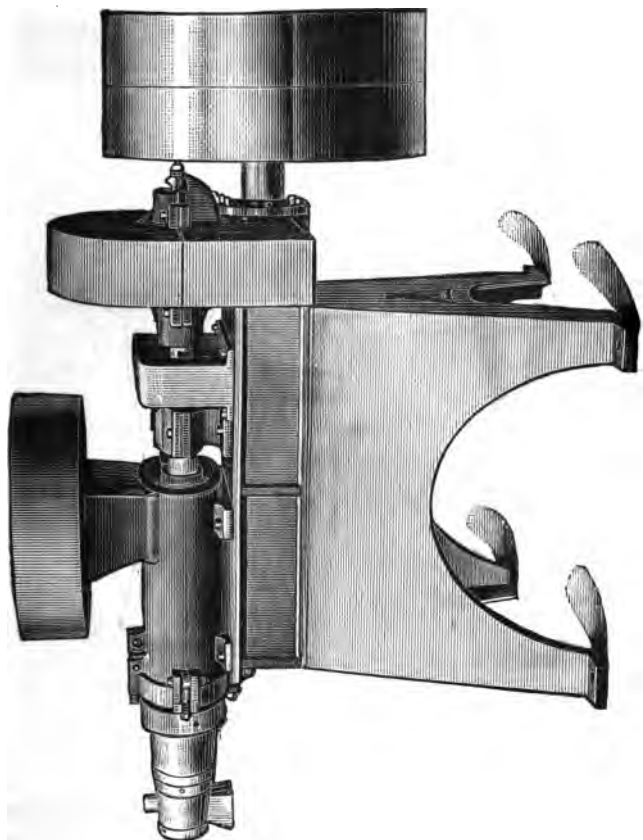


BEYER'S continuous plodding machine.

3°. PLOTTING OR PLODDING.—The object of the plotting machine is to compress the ribbons and shape them into perfectly homogeneous and compact bars, and its use has tended greatly to the development of the manufacture of toilet soaps. A representation of this apparatus is given in

Figs. 35 and 36. In this machine, below the hopper, there is a powerful screw propeller, conical in shape, and fitting closely the conical barrel. Owing to this form, the ribbons

FIG. 36.



RUTSCHMAN'S compound helix continuous plodder.

of soap, falling from the hopper upon the larger part of the revolving screw, are forced towards the mouth of the barrel with increasing pressure. The brass mouthpiece is fitted

with gauge-plates for altering the size and shape of the bar as it issues therefrom. These two machines are capable of turning out 10,000 cakes of soap in one day.

In another form of plotting machine the soap is pressed by means of a hydraulic ram through a cylinder, and *squirted* through a mouthpiece of the required dimension and shape.

FIG. 37.



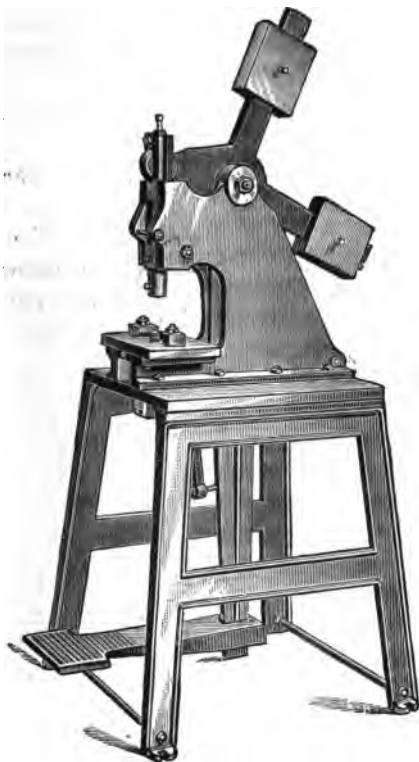
RUTSCHMAN'S cake-cutting machine.

4°. CUTTING INTO CAKES.—From the plotting machine the bars are transferred to a cutting machine, worked either by the foot (Fig. 37) or by steam, and cut into blocks or cakes of the desired size.

5°. STAMPING.—These cakes are afterwards moulded and

stamped, either by a stamping press worked by hand or foot or by steam (Fig. 38 and Figs. 20, 27, 28, 29, pp. 76, 120, 121, 122).

FIG. 38.



RUTSCHMAN'S soap press.

By Dr. C. R. A. WRIGHT'S patented process * a soap free from uncombined non-volatile alkali may be produced under the French system from stock soaps containing free alkali.

* English patent 7573, February 10, 1885.

A quantity of an ammoniacal salt (such as the chloride or sulphate), equivalent to the average amount of free alkali in the stock, is dissolved in the smallest possible quantity of warm water and added to the shavings before their first passage through the mill. During the successive grindings the ammonia and ammonium carbonate formed from the neutralization of the free alkali are practically entirely removed by evaporation, which readily takes place from the thin ribbons scraped off from the rollers.

The chief advantages of the *milling* process are that the most delicate perfumes can be mixed with the soap without loss, as there is but little heating during the operation, and that as the cakes produced contain less water than those formed by the re-melting process, they require a less time in the drying-room before being ready for sale, and will not afterwards shrink or lose weight.

CHAPTER IX.

MEDICINAL SOAPS.

DECHAN gives the following results obtained in his valuable investigation into the character of the soaps of pharmacy: *—

“Generally speaking, the samples examined are to be relied on for the quality and complete saponification of the fat employed, though in a few instances the purity of the oil might have been called in question. In almost every case the combined alkali is in excess of the quantity required to form the normal salt, and in several of the samples there is a considerable percentage of free alkali. This is unfortunate, especially as regards the free alkali, because the value of the soaps for many pharmaceutical purposes, such as excipients for certain pill masses, bases for suppositories, &c., depends to a considerable extent on the complete combination of the alkali with the fat.

“*Sapo Durus, B.P.*—There is some difference of opinion as to whether ‘hard soap’ is synonymous with ‘white Castile soap.’ Mr. Squire says ‘the *Sapo durus* of the Pharmacopœia refers without doubt to the white Castile soap.’ But if it is meant, as seems to be intended, that *foreign* Castile soap only is referred to, then, with all deference to this authority, there is very considerable ‘doubt’ in the matter. Some firms supply the same soap indis-

* “Pharmaceutical Journ.” April 25, 1885.

criminally, but others make a distinction, as will presently be shown, and it would be well if we had some authoritative declaration to guide us. For example, Nos. 2 and 11 were supplied at the same time by one firm, and it is perfectly evident that the soaps are quite distinct; No. 1, on the other hand, bears a much stronger resemblance to Nos. 10 and 11 than it does to No. 2. The principal constituents, olive oil and soda, are the same in both classes, so that the main distinction between them, No. 1 excepted, is that *Sapo durus* contains a higher percentage of fat, and consequently it is of more value. We may therefore quite fairly infer that sample No. 1 is a specimen of *Sapo castil. alb.*, and if this much be allowed, then we can see a very sharp distinction between the two classes of soaps; *Sapo durus* containing nearly 7 per cent. more fat than *Sapo castil. alb.* This difference is certainly too much to allow to accident, so it must be accounted for in some other way. CHRISTISON in treating of these soaps says that they are chiefly imported, especially from Spain, 'but of late years hard olive soap has been manufactured in England' ('Dispensatory,' p. 280); consequently, it appears quite possible that there is another distinction between these two soaps, apart from that shown by the analysis—viz., that *Sapo durus* is of English manufacture, whereas *Sapo castil. alb.* has always been recognized as a foreign product. Should this be correct, then they are not one and the same, notwithstanding Mr. Squire's statement to the contrary, and it would evidently be of some value to have this authoritatively decided, because there can be no two opinions as to their relative value as shown by the analysis. All the soaps responded to the tests given by the B.P. with the exception that none of them were entirely soluble either in water or rectified spirits, the largest percentage of insoluble matter being 1.8 in the case of No. 1.

"*Sapo Animalis*.—The finest quality of this soap is made from pure tallow which has undergone the process known as 'rendering,' and this alone ought to be used for pharmaceutical purposes. The fat from which the specimens examined had been manufactured was of a uniform and good quality, which could not be said of some of the other classes. With the exception of No. 7, all the samples contained a larger percentage of uncombined alkali than did those of *Sapo durus*, and on this account, even if no other reason existed, ought not, under any circumstances, to be preferred to the latter, which is of a decidedly milder type, and therefore much better suited for those galenical preparations in which soap is a necessary constituent.

"*Sapo Castil. Alb.*—The analytical results of the different specimens indicate that much care is bestowed on the manufacture of this article, with the view evidently of producing a soap containing as near as is practicable a uniform percentage of fatty matter, the greatest variation in this direction being 2.4 per cent., which, considering the *modus operandi* of soap manufacture, is exceptionally small. It is also worthy of note that one sample (No. 9) did not contain the slightest trace of uncombined alkali, being, in fact, the only one of the twenty specimens examined which showed absolute freedom from what must be considered a most objectionable ingredient. The fact that this sample is of continental manufacture ought to have some meaning to British manufacturers, showing, as it does, that it is quite possible to manufacture a perfectly neutral soap, whereas the efforts of many home makers in this direction end in utter failure. Like *Sapo durus*, *Sapo castil. alb.* answered fairly well to the B.P. tests, and if the difference in the percentage of fat be taken into account, the latter can be quite appropriately substituted for the former, and *vice versa*.

"*Sapo Castil. (Mottled)*.—This soap is decidedly of an

inferior character as compared with *Sapo castil. alb.* It contains a larger proportion of free alkali, and the fat is also much lower in quality. This ought, in my opinion, to prevent its being used for either *Sapo durus* or *Sapo castil. alb.*, for which it is sometimes substituted. The mottled appearance of the soap was produced in some cases with ultramarine, and in others with iron salts. The materials added for the purpose of mottling in no way enhance the value of the soap, but, if anything, have an opposite tendency, and the fact that soap manufacturers should persist in mottling the soap can only be explained by the demand for such by a taste which may be characterized as uninformed and antiquated.

"There seems to be a common impression that the mottled Castile is better than the white, this opinion being founded probably on PEREIRA's statement that the white soap is purer than the mottled, 'but it is a weaker soap (*i.e.*, it contains more water).' It is possible that when this was written the relative composition of the soaps may have been different to what it is now, but it is remarkable that in every case in the under-noted table [p. 150] the samples of mottled soap gave higher percentages of water than those of the white. If it is meant that the white is weaker in the sense of being less irritating, the contention might be admitted; but this is of course no advantage, so that the mottled variety is in every respect decidedly inferior.

"There also seems to be a common notion that soap contains a very large percentage of water, one writer stating recently that it contained 40 per cent. more or less. This is quite true if it be applied only to the common scouring soaps, but decidedly erroneous when the statement is made, as in this case it was, with reference to the soaps under consideration.

"*Sapo Mollis*.—The percentage of free alkali in the

samples of this soap is very remarkable, one sample containing as much as 0.8 per cent. The quantity of combined alkali in excess of that required to form the normal potassic salt is also much greater than in that of the others, the mean of the four samples examined being 4.25 per cent. The composition of *Sapo mollis* is liable to vary to a much greater extent than any of the other classes, and for this reason the soap is not so much to be depended on. The cause of this irregularity is mainly due to the process of manufacture, which depends more on the operative in charge of the work than is the case in the manufacture of hard soaps.

"That something ought to be done to reduce the percentage of free alkali in soaps required for pharmaceutical purposes will be readily admitted, and that soaps can be produced which do not contain this irritating and corrosive agent we have sufficient evidence in the results given in the table [p. 150]. It remains with those who have a right to speak in the matter to make it known that a soap containing free alkali ought not to be used in pharmacy."

Aromatic Mouth Soap (ZALMON'S)*.—1 lb. of neutral soap, prepared from fat of the best quality, is dissolved in cold distilled water; about 3½ oz. finely sifted cuttle-fish bone are added to the solution, and the whole is evaporated at a gentle heat. When the desired consistency is nearly reached, add ¾ drachm each of peppermint oil, sage oil, virgin honey, and wine vinegar, or lemon oil. Mix the whole quickly by stirring, and pour into suitable moulds to cool. Colouring matter may be added as desired.

Aromatic Antiseptic Tooth Soap.†—Castile soap 1 lb., pumice-stone in fine powder 1 oz., thymol 20 grains, oil of wintergreen 30 drops.

* "Chemist and Druggist," 1880, p. 13.

† *Ibid.* 1884, p. 73.

DECHAN's Table of Analyses of Pharmaceutical Soaps.

| | | Fat. | Com- bined Alkali. | Free Alkali. | Sul- phate. | Chlo- ride. | Silica. | In- solu- ble Matter. | Water. | Inso- luble in Alcohol. |
|-----------------------|----------------|------|--------------------------|-----------------|----------------|----------------|--------------|--------------------------------|--------|-------------------------------|
| 1 | Sapo durus | 72.2 | 9.02 | 0.24 Tr. | 0.33 0.00 | 0.80 Tr. | 0.00 0.00 | Tr. | 20.00 | 1.8 |
| 2 | " | 83.0 | 9.60 | Tr. | 0.00 | Tr. | 0.00 | Tr. | 10.00 | Tr. |
| 3 | " pulv. | 83.0 | 11.40 | 0.10 | Tr. | Tr. | 0.00 | 0.10 | 8.00 | 0.2 |
| 4 | " | 88.0 | 9.65 | Tr. | Tr. | Tr. | 0.00 | 0.60 | 4.60 | Tr. |
| 5 | " animalis | 80.2 | 9.30 | 0.31 | Tr. | 0.45 | 0.00 | 0.71 | 10.00 | 1.2 |
| 6 | " | 76.0 | 10.37 | 0.31 | 0.20 | 0.70 | 0.00 | 0.40 | 14.00 | 1.6 |
| 7 | " | 78.8 | 10.00 | 0.12 | 0.25 | 0.30 | 0.00 | 0.50 | 11.00 | 1.2 |
| 8 | " | 78.4 | 8.60 | 0.40 | Tr. | Tr. | 0.00 | Tr. | 15.00 | 0.4 |
| 9 | " castil. alb. | 77.9 | 9.48 | 0.00 | Tr. | 0.27 | 0.00 | 1.00 | 12.00 | 0.6 |
| 10 | " | 75.5 | 8.90 | 0.25 | Tr. | Tr. | 0.00 | 0.80 | 16.00 | Tr. |
| 11 | " | 77.4 | 9.20 | 0.10 | 0.30 | 0.30 | 0.00 | 0.70 | 12.50 | 0.8 |
| 12 | " | 76.0 | 10.00 | Tr. | Tr. | 0.60 | Tr. | 1.00 | 12.50 | 1.0 |
| 13 | " (mottled) | 69.4 | 9.18 | 0.15 | 0.10 | 0.79 | 0.50 | 0.84 | 20.00 | 1.3 |
| 14 | " | 64.8 | 8.60 | Tr. | 0.14 | 0.40 | Tr. | 0.70 | 26.00 | 1.0 |
| 15 | " | 71.8 | 9.04 | 0.28 | 0.20 | 0.50 | 0.10 | 1.20 | 17.30 | 1.8 |
| 16 | " | 66.5 | 8.84 | 0.33 | 0.10 | 0.30 | Tr. | 0.60 | 23.50 | 1.2 |
| 17 | " mollis | 51.0 | 10.60 | 0.20 | 0.19 | 0.80 | 0.17 | 1.00 | 39.50 | 1.8 |
| 18 | " | 48.0 | 10.32 | 0.21 | 0.20 | 0.60 | 0.10 | 1.10 | 42.00 | 2.0 |
| 19 | " | 45.0 | 14.90 | 0.80 | 0.30 | 0.80 | 0.30 | 1.00 | 40.50 | 2.0 |
| 20 | " | 50.0 | 14.60 | 0.30 | 0.25 | 0.70 | 0.10 | 1.10 | 36.00 | 0.8 |
| <i>Mean results:—</i> | | | | | | | | | | |
| | Sapo durus | 81.5 | 9.92 | 0.08 | 0.08 | 0.20 | 0.00 | 0.20 | 10.65 | 0.5 |
| | " animalis | 78.3 | 9.57 | 0.28 | 0.11 | 0.36 | 0.00 | 0.40 | 12.50 | 1.1 |
| | " castil. alb. | 76.7 | 9.14 | 0.09 | 0.07 | 0.29 | 0.00 | 0.90 | 13.25 | 0.6 |
| | " (mottled) | 68.1 | 8.90 | 0.19 | 0.13 | 0.50 | 0.15 | 0.83 | 21.70 | 1.3 |
| | " mollis | 48.5 | 12.60 | 0.38 | 0.23 | 0.70 | 0.17 | 1.00 | 39.50 | 1.0 |

Shave the soap into ribbons, beat it into a paste with a little water, and add first the pumice-stone, and then the thymol and oil of wintergreen dissolved in a small quantity of alcohol.

Castor-oil Soap (for *Linimentum Saponis Compositum*).

—According to M. S. HAMMER,* this soap seems to answer best for this liniment, and may be prepared by the following process :—

Saponify 2 pints of castor oil with 6 oz. of caustic potash and 2 pints of water by heating till a transparent mixture is obtained ; then add a saturated solution of 8 oz. of sodium chloride, stir until cool, allow to subside for a day, decant the liquid portion, cut in pieces, and dry for use.

Chlorinated Soap (*Sapo Calcis Chlorinatæ*).—Castile soap in powder 11 oz., chloride of lime (dry) 1 oz. Mix, beat them to a mass with rectified spirit q.s., holding in solution oil of verbena, or of ginger-grass, $\frac{1}{4}$ oz. Lastly, form the mass into flat tablets, and wrap in thin sheet gutta-percha. Said to be well adapted for hospital use, for removing stains from the skin, and for preventing infection from contagious diseases.

Camphorated Sulphur Soap.†—12 kilos. of cocoa-nut oil, 6 kilos. of soda lye (38° B.), 1 kilo. of potassium sulphate dissolved in $\frac{1}{2}$ kilo. of water, and 160 grams of camphor, which is to be dissolved in the melted cocoa-nut oil.

Gall Soap.†—1 kilo. of galls is stirred in 25 kilos. of melted cocoa-nut oil, and then saponified cold with $22\frac{1}{2}$ kilos. of soda lye (38° B.). The soap is coloured with 350 grams of ultramarine green, and perfumed with 75 grams lavender oil and 75 grams cummin oil.

Iodine Soap.†—10 kilos. cocoa-nut oil, 5 kilos. lye

* "Proc. Cal. Pharm. Soc." 1883, p. 50 ; "Year Book of Pharmacy," 1883, p. 313.

† "Year Book of Pharmacy," 1883, p. 313.

(38° B.), and $1\frac{1}{2}$ kilo. of potassium iodide, dissolved in $\frac{1}{2}$ kilo. of water.

Disinfecting Soap (JEYE'S *Improved*).—Gas tar is distilled and the light oil rejected; 16 parts of the heavier oil, 32 parts of cocoa-nut oil, and 16 parts of caustic soda at 35° B. are saponified in a jacketed pan, with or without the addition of rosin, and sodium sulphate and carbonate.*

Liquid Soaps (KINGZETT'S).†—KINGZETT prepares liquid soaps for employment as insecticides by dissolving rosin or crude turpentine in alcohol, and saponifying with potash. To this is added an alcoholic solution of a fatty acid soap and various disinfectants. Or,‡ crude turpentine, or rosin may be dissolved in "Sanitas" oil, or rosin spirit, or rosin oil, and then saponified by caustic-alkali solution of sp. gr. 1.300. Camphor is added to insure a permanently liquid product, and this may be medicated by addition of thymol, &c. Or,§ petroleum spirit, or thymol, may be used instead of, or in conjunction with, the "Sanitas" oil mentioned in the last patent.

Mercurial Soaps.—1. *Sapo Hydrargyri*.—Dissolve 4 oz. of mercury in the same weight of nitric acid without heat; melt in a porcelain basin, over a water-bath, 18 oz. of veal suet, and add the solution, stirring the mixture till the union is complete. To 5 oz. of this ointment add 2 oz. of solution of caustic soda (sp. gr. 1.33) till a soap is formed which is completely soluble in water.

2. *Sapo Mercurialis*.—Castile soap (in powder) 4 oz., corrosive sublimate 1 dr. dissolved in rectified spirit 1 oz.; beat to a uniform mass in a mortar.

* English patent 16,427, December 13, 1884.

† " " 3,894, March 26, 1885.

‡ " " 2,210, February 17, 1885.

§ " " 3,855, March 25, 1885.

3. *Sapo Hydrargyri Precipitati Albi* (Sir H. MARSH).—Beat 12 oz. of white Windsor soap in a mortar, add 1 drachm of rectified spirit, 2 drachms of white precipitate, and 10 drops of otto of roses; beat the whole to a uniform paste.

4. *Sapo Hydrargyri Precipitati Rubri* (Sir H. MARSH).—White Windsor soap 2 oz., nitrate of mercury (levigated) 1 drachm, otto of roses 6 or 8 drops, in rectified spirit 1 to 2 drachms; beat to a paste.

Soap Leaves.—These are made by passing continuous paper sheets over rollers through a hot solution of soap, the excess of soap attached to the surface being scraped off. The paper then is conducted over drying cylinders to the cutting machine.*

Tannin Soap.—9 kilos. of cocoa-nut oil are saponified with $4\frac{1}{2}$ kilos. of soda lye, then 250 grams of tannin, previously dissolved in alcohol, are put in, and the whole mixed. The soap is perfumed with 30 grams Peru balsam, 10 grams cassia oil, and 10 grams oil of cloves.†

Tar Soap (*Sapo Piceus*).—Tar 1 part, liquor potassæ and soap (in shavings) of each 2 parts; beat them together till they unite. Action, stimulant, in psoriasis, lepra, &c.

Turpentine Soap (*Sapo Terebinthinæ*; STARKEY'S Soap).—Potassium bicarbonate, oil of turpentine, and Venice turpentine, equal parts; triturate together in a warm mortar, with a little water, till they combine; put the product into paper moulds, and, in a few days, slice it, and preserve in well-stoppered bottles.

Unna's Soaps.‡—UNNA started his experiments by pre-

* REITHOFFER and NEFFE, Vienna, German patent 23,195, June 6, 1882; "J. Soc. Chem. Ind." 1883, p. 543.

† "Year Book of Pharmacy," 1883, p. 313.

‡ "Edinburgh Medical Journ." October 1885; "Year Book of Pharmacy," 1886, p. 282; "Pharm. Journ." xvi. 328.

paring a normal soap of fixed composition, which could be incorporated with various medicinal substances. Though, theoretically, he considered that beef fat was the most perfect, still, practically, he found that an advantage was gained by adding 1 part of olive oil to 8 parts of beef fat. The alkali consisted of 2 parts of soda to 1 of potash, this combination being less apt to blister when medicinal substances were added to the soap. Cocoa-nut oil, though producing a soap which lathers well, was found to make the skin dry after continued use. Even a neutral soap, when constantly used, tends, according to UNNA, to produce an unpleasant roughness, from removing too completely the natural oiliness of the skin. He, therefore, leaves the soap *over-fatty*, that is, besides the fat necessary for perfect saponification, an excess amounting to 3 or 4 per cent. is added. Any secondary addition of glycerin or vaseline he entirely rejects. This soap he terms *over-fatty normal soap* (*über fettete grund Seife*). It may be used as an ordinary washing soap in all forms of inflammatory skin diseases where ordinary soap is forbidden, as in eczema, erythema, and for skins poor in fat with a tendency to dryness; also as a soap for healthy people whose occupation compels them to wash frequently in the course of the day. The composition of such soap is:—

| | | | | | | |
|----------|-------------------|---|---|---|---|-------|
| 16 parts | best ox fat | . | . | . | . | 59.3 |
| 2 " | olive oil | . | . | . | . | 7.4 |
| 6 " | soda lye (38° B.) | . | . | . | . | 22.2 |
| 3 " | potash lye | " | . | . | . | 11.1 |
| <hr/> | | | | | | <hr/> |
| 27 | | | | | | 100.0 |

In this soap about 4 per cent. of oil remains unsaponified. It is of a yellowish-white colour, of a waxy consistence, and quite permanent. It forms an exceedingly good soap for children, and, if rubbed on the hands and wiped off again in a few minutes with a dry towel, it leaves the hands smooth,

and little liable to be injuriously affected by damp, cold, or long-continued contact with carbolic acid.

Over-fatty Marble Soap consists of equal parts of the foregoing, and the finest powdered marble. It will be found useful in thinning down the horny layer in acne. It thus replaces pumice-stone and sand soap, and, while the powdered marble rubs off the scales or the thickened epidermis, the over-fatty normal soap leaves the polished surface smooth and normally unctuous.

Over-fatty Ichthyol Soap.*—This has its special value in the treatment of various forms of rosacea, both in the congestive and cyanotic forms, and can be advantageously employed with hot water. A stronger effect is produced by leaving the soapy lather to dry on.

Wych-hazel Soap.†—The juice of the plant *Hamelis virginica*, or common wych-hazel, is mixed with soap, and with various compounds for toilet purposes which contain soap. Such compounds are said to be beneficial in the case of bruises and lacerations of the skin.

* *Ichthyol*, or fish oil, first prepared by SCHRÖTER, is the distillation product of a peculiar bituminous sulphurous mineral obtained from deposits of fossil fish. According to BAUMANN, sodium ichthyosulphate has the composition represented by the formula $C_{25}H_{22}S_2Na_2O_6$.

† DIMBLEBY, English patent 11,305, August 15, 1884; "J. Soc. Chem. Ind." 1885, p. 459.

CHAPTER X.

OLEIC-ACID, RED OR BROWN OIL, SOAPS— SOFT SOAP—INDUSTRIAL SOAPS.

Oleic-acid or Red Oil Soaps.

OLEIC-ACID, red or brown oil, is a bye-product of the candle manufacture, and, being already separated from glycerin, it readily enters into combination with alkalies, either caustic or carbonated.

Morfit's Process.—The red oil, or other fatty acid, is poured into an open pan, with a fire beneath, to one-third of the depth of the vessel, in which it is agitated and heated by the patentee's *steam-twirl*.* If it is desired to make a grade of soap lower than toilet soap, rosin, in the proportion of 5 per cent. of the acid and upwards, is added in small lumps as soon as the oil has become hot. When, after continued heating and stirring, the rosin is entirely dissolved, finely powdered carbonated alkali is added in quantity proportionate to the homogeneous mixture of fat and rosin, while the swirl is kept slowly revolving. When all the alkali is in, and the swelling-up caused by the escape of carbonic

* This is a sort of rotatory paddle fixed inside the copper, tubular, and perforated at intervals. It is connected by means of a hollow spindle with the boiler, so that steam can be admitted through it at will. Thus heating and mixing are effected simultaneously.

acid has subsided, the paste begins to thicken, and soon assumes the condition of soap. It is then removed to the frames, and left to settle. For *neutral* soaps, the quantity of carbonated alkali should only slightly exceed the proper equivalent proportion, determined by calculation from the combining number of the fat acid which constitutes the "stock." For *strong* soaps the quantity of alkali may be increased.

The advantages claimed for the preparation of soaps by MORFIT'S process are—(1) As the relative proportions of the ingredients are adjusted at the beginning of the operation, there is no waste lye or any other residue. (2) The soap is said to come out promptly, and in greater perfection than can be readily obtained by the usual method of boiling upon caustic lye. (3) The product is always uniform in appearance and composition, and does not shrink or deteriorate by time and atmospheric influence.

Another way of preparing this soap is the following:—1300 lb. of soda lye of 18° B. are boiled in the copper, and to it are gradually added, with constant stirring, 1000 lb. of red oil. The oil is rapidly taken up by the lye, and there is considerable intumescence, which has to be kept down by uninterrupted stirring. As long as the paste continues strongly caustic it must have new additions of oil till only slight alkalinity remains. If, on the other hand, after cooling for two or three hours in the copper, there is a deficiency of alkali, it must be heated with 50 or 60 lb. more lye. The fire is then extinguished, and the paste, after an interval of about twenty-four hours, is removed to the frames, which should be very shallow, as this soap sets slowly.

CARPENTER* describes MORFIT'S method for the preparation of soap from fatty acids as follows:—

* SPON'S "Encyclopædia," v. 1771.

The soda is used in the form of a refined carbonated ash at 52°, every 100 lb. being dissolved in 160 lb. of water in a lead-lined vat, and the solution allowed to settle previous to use. The store-tanks of this, and of the fatty acids employed, are connected with small gauge-tanks or measuring tubes for the purpose of obtaining uniformity of results by the use of exact quantities in every operation.

For the delivery of the soda solutions into the soap-pan a special feeder is provided, so that the flow of liquid may be regulated at discretion; a perforated rose-spout may be advantageously placed under the exit pipe.

The soap-pan is jacketed and furnished with a stirrer, and the steam is either superheated, or used at a pressure of 75-80 lb. The pan has a movable curb above it, so as to give room for the increase of bulk caused by the liberated carbonic acid. The curb, when required, can be drawn aside on a railway.

In making soap with this apparatus, 1000 lb. of oil are run into the pan, with the curb in its place, and heated to 280-320° F. (138-160° C.) according to its quality. At this point, for a *neutral* soap, 190 lb. of soda ash, or, for a *strong* soap, 210-225 lb., dissolved in the proper quantity of water, at 212° F. (100° C.), is let into the pan at such a speed that the time occupied is not less than six nor more than twelve minutes. The whole is kept well stirred, and swells up enormously; but, in five minutes after the last portions of alkali have been added, the mass subsides, and, in fifteen minutes more, changes from a spongy to a clear, soft, brilliant, homogeneous paste. The curb is then removed, and, in about an hour, 100 lb. of boiling water are let in from the rose-spout of the soda-feeder, and the whole is again well stirred. If it is desired to add sodium silicate, or any other substance, it is introduced at this stage, after which the soap is transferred to the cooling frames, and a

fresh batch is proceeded with. Soap thus made has the following composition :—

| | | | | | |
|------------|---|---|---|---|-----------------|
| Water | . | . | . | . | 27.50 per cent. |
| Oleic acid | . | . | . | . | 65.00 " |
| Soda | . | . | . | . | 6.70 to 7.50 " |

When rosin is used, it should be added to the oil while the latter is being heated, or the rosin soap may be made in a separate pan provided with a MORFIT'S steam-twirl: 1200 lb. rosin and 2200 lb. caustic lyes at 11° B. are boiled together, and the thin jelly so produced is transferred in suitable quantities to other pans. This soap contains :—

| | | | | | |
|-------|---|---|---|---|----------------|
| Water | . | . | . | . | 37.7 per cent. |
| Rosin | . | . | . | . | 54.5 " |
| Soda | . | . | . | . | 7.8 " |

According to MORFIT the refined ash of 52°, prepared by the Jarrow Company, Newcastle-on-Tyne, has the following composition :—

| | | | | | |
|-----------------|---|---|---|---|--------|
| Water | . | . | . | . | 1.00 |
| Sand and silica | . | . | . | . | traces |
| Sodium chloride | . | . | . | . | 2.84 |
| „ sulphate | . | . | . | . | 8.04 |
| „ carbonate | . | . | . | . | 88.66 |
| Total | | | | | 100.54 |

Soft Soap.

The article which is known as *soft soap* is not, strictly speaking, a true soap, but rather a more or less impure solution of potash soap in caustic lye, forming at ordinary temperatures a transparent smeary jelly.

Soft soap is used to some extent for washing coarse linen, but it is of far greater importance, as an indispensable and powerful detergent, in linen-bleaching works.

The fatty materials employed in this country for making

soft soaps are whale oil, seal oil, linseed oil, and tallow ; on the Continent, the drying oils, hemp, linseed, sesamé, camelina, and poppy, and the non-drying oils, rape, train, &c. As the first group produce a softer article, it is customary to mix the oils in different proportions according to the time of year, employing more of the drying oils in winter and of the non-drying oils in summer.

Rosin may be introduced, in fine powder, up to $\frac{1}{3}$ rd of the weight of the fatty matters.

In the preparation of the soap, in some works, a portion of the oil is first introduced and heated. Then weak potash lyes, marking from 9° to 11° B., are added ; *moderate* heat is kept up, more oil and lye being alternately added till the whole of the charge has been introduced. Some makers add the whole of the fat at once together with a portion of the lye, and the remainder of the lye after some hours. Gentle ebullition, with great care to prevent boiling over, is continued till the saponification is judged complete. The boiling gradually becomes quieter, the frothy mass subsides, the paste grows more transparent, becomes thicker, and a thick, sticky fluid falls in streaks from the stirrers. As soon as these characters are apparent, stronger lye is gradually added for the purpose of clarification. If a transparent appearance is not readily produced, it is requisite to add some very strong lye. When the combination is perfect, the clear and transparent paste should be free from clots or granules, and there should be no acrid taste. To ascertain if this is the case, a small sample, free from scum, is taken out from the middle of the pan and cooled. If it should neither be of proper consistence nor free from opacity, the boiling is continued, and re-tested in the same way at intervals until the soap is properly finished.

In the cooling of these small samples, peculiar phenomena are noticed, which afford good means of judging of the

quality of the soap. When there is formed round the little patch, dropped on to a piece of clean glass, an opaque zone, a fraction of an inch broad, this is taken as indicating complete saponification, and is called *strength*. When this ring is absent, the soap is said to want *strength*. When the zone, after being distinctly seen, soon disappears, the soap is said to have *false strength*.

Towards the end of the boiling, the soap becomes thicker, the colour darkens, and there is less frothing. When the bubbles become so large as to overlap, they resemble films or lamellæ, and soap-boilers term such appearance *lamination*. A peculiar noise at this point is heard, and it is said the soap *talks*. If, on testing a portion, no opaque zone, or only a slight one, appears after cooling, it may be concluded that the proper proportions have been attained.

When the tests are satisfactory to the experienced operator, the fire is extinguished, or the steam turned off, the soap is left for some time longer to cool, and is then packed in small casks for use. The cooling is sometimes aided by the introduction of a quantity of *cold soap*.

Scotch Method.—273 gallons of whale or cod oil and 4 cwt. of tallow are put into the soap-pan, with 250 gallons of lye, made from American potash, of such strength that 1 gallon contains 6600 grains of real potash. Heat being applied, the mixture froths very much as it approaches the boiling temperature, but is prevented from boiling over by beating down on the surface within the iron curb which surmounts the caldron. Should it soon subside into a doughy-looking paste, the lye has been too strong. Its proper appearance is that of a thin glue. About 42 gallons of a stronger lye, containing about 8700 grains of potash per gallon, are now introduced, and, after a short interval, another 42 gallons; and thus successively till nearly 600 such gallons have been added to the whole. After suitable

boiling to saponify the fats, the proper quality of soap will be obtained, amounting in quantity from the above materials to 100 firkins of 64 lb. each.*

Russian Method.—According to KURRER, a lye containing three-fourths caustic potash and one-fourth potassium carbonate marking 10° B. is added to the linseed, rape, or hemp-seed oil in the boiler. An equal quantity of the same lye is placed in a cistern by the side of the boiler, and is allowed to flow uninterruptedly in a minute stream into the boiler, so that the state of ebullition is not checked. The process is judged complete when the soap flows from the stirrer as a clear slime which can be drawn out in threads between the fingers.

The results by this method are uncertain, and the product is never uniform.

Gentele's Method.—GENTELE found that the potash in soft soaps may be partially replaced by soda without any disadvantage. The product contains a little more water than ordinary soft soap. The best proportions are said to be 1 part of soda to 4 parts of potash lye, and the lyes should be free from salt and other saline impurities, which prevent the clarifying of the soap. A mixture of 100 lb. of red oil, 50 lb. of tallow, and 3750 lb. of hemp-seed oil makes a good stock for this soap.

Soft soaps are more caustic than hard soaps, and contain whatever impurities may be present in the materials. The white granular masses in soft soaps are due to potassium stearate, and are sometimes imitated by the introduction of starch.

* URE's "Dictionary of Arts," &c., iii. 702.

Industrial Soaps.

"Fulling" Soap, or soap for cleansing and scouring woollen fabrics, is a soft soap of the composition*—

| | I. | II. |
|---------------------|------|------|
| Fatty acids | 50.0 | 40.0 |
| Potash | 11.5 | 9.5 |
| Water | 38.5 | 50.5 |

It should contain a slight excess of alkali, but no rosin (which hardens the fabrics), starch, or silicate.

Or,† a *brown-oil* soap, prepared by MORFITT'S process, which should have a stiff body and be slightly strong in alkali, may be used. Its solution in boiling water must cool to a jelly in a reasonably short time. Its suitability in this respect may be ascertained by dissolving, with the aid of heat, 1 oz. of the sample in $7\frac{1}{2}$ oz. of water, and then adding *cold* water up to 16 fluid oz. This should form a jelly within half an hour. Such a soap, when freshly prepared, has, according to MORFITT, the following composition:—

| | |
|---|--------|
| Fat (melting point 84° F.) | 65.00 |
| Combined soda | 6.50 |
| Other salts | 1.40 |
| Water | 27.10 |
| | <hr/> |
| | 100.00 |

Another formula that has been proposed for soap for cleansing woollen fabrics‡ is:—1 part of borax and 32 parts of Castile soap incorporated with water into a thick paste, to which a fragrant essence may be added.

Ox-gall Soap.—The following method gives a satis-

* RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 693; KINGZETT'S "Alkali Trade," p. 175.

† MORFITT'S "Practical Treatise on Soaps," p. 196.

‡ M. S. GOSLING, English patent 5998, May 15, 1885.

factory article :*—Mix together $1\frac{1}{2}$ kilo. ox-gall with 25 kilos. melted cocoa-nut oil. Saponify this mixture by the cold process with $12\frac{1}{2}$ kilos. caustic-soda lye of 38° B. The soap may be dyed by the addition of 850 grams of ultramarine, and, if desired, perfumed with a mixture of 75 grams of lavender oil and 75 grams of caraway-seed oil. Ox-gall soap is useful for scouring woollen goods.

Soaps for Calico Printing and Dyeing.—Soaps from tallow, palm or olive oil are generally employed for calico printing and dyeing, olive-oil soaps being sometimes preferred for Turkey-red dyeing. A good soap for these industries must be as neutral as possible, and thoroughly saponified.

When soaps of the alkalis are used as mordants in conjunction with alum, or tin or lead compounds, there is a combination of alumina, tin, or lead with the fatty acids of the soap, and an insoluble metallic soap is deposited on the fibre.

According to O. SCHEURER,† a soap for brightening colours such as alizarin, or garancin, should, first of all, produce a perfectly white ground, upon which the colour then appears much more brilliant, and, in the second place, it should not attack the colour itself. On comparing, from this point of view, the various soaps occurring in commerce, the Marseilles soap was found to be the best, although the reason for this superiority is not, at first, obvious. A soap which attacked the colours used to be regarded as too alkaline, but on analysis it was found to contain no more alkali than the best soaps. It was especially the oleic-acid soaps which exhibited this injurious alkalinity—attacking all shades of colour. This behaviour is attributed by SCHEURER to the

* "J. Soc. Chem. Ind." 1882, p. 154.

† "Bulletin de Mulhouse," 1882, p. 142; "J. Soc. Chem. Ind." 1883, p. 286.

fact that many so-called alkaline soaps made with oleic acid simply contain both free oleic acid and free alkali, because the saponification has not been complete. Such soaps may be perfected by continuing the boiling. It should be remembered that the combination of the acid and soda requires a considerable time—two kinds of soap, an *acid* soap and a *basic* one, seem to be produced at the beginning of the process, and these gradually unite to form a neutral soap.* The reaction can be hastened either by increasing the temperature or the pressure; thus, at a pressure of 1.5 atmosphere SCHEURER found that a better soap is obtained in two hours than in twelve hours under ordinary pressure. A soap manufactured by DAUMAS D'ALLÉON, of Marseilles, is recommended as the type of that best suited for dyeing and printing purposes. It has the following composition:—

| | | |
|--|-----|---|
| Fatty acids | 55 | { or 9.106 parts Na ₂ O to 100 parts of fat |
| Caustic soda (Na ₂ O) | 6 | |
| Water | 39 | |
| Total | 100 | |

The following method is said to be successfully used at the Zawierciars Works for the preparation of a soap to be used in dyeing and printing:—About 360 litres of water and 69 kilos. of lye at 36° B. are boiled up together, and 140 kilos. of oleic acid added with constant stirring till a uniform mixture is obtained; 3120 litres of water are then added, and the mixture is well stirred till a clear soap solution results.

When the above proportions are used, the oleic acid is sometimes found to be in excess, and some more soda must then be added. To prevent this, a little more soda should be added at the beginning.†

* See also p. 53.

† "Dingl. Polyt. Jour." 247, 12; "J. Soc. Chem. Ind." 1883, p. 286.

Soap for Silk Throwsters.*—This should be the best curd soap of the usual processes—white, and free from odour.

Soap for Silk Dyers.—The soap suitable for *stripping* and *boiling off* gum from silk is a brown-oil soap,* which should cleanse readily without injury to the silk, and be easily rinsed out. It is usual to add to the soap a proportion of sodium sulphate.

In the North of Europe † soft potash soaps, generally made from linseed oil, are used; in the South, hard soda soaps made from olive and other oils are preferred. Of late years, soaps made from oleic acid have been increasingly used. In general, those which are made from oleic acid and linseed oil wash off best; next, those from olive oil and suet, &c. Palm-oil soap does not rinse off so well. For scouring silk to be dyed, oleic-acid soap is most suitable, but for those destined to remain white a good olive-oil soap is preferable.

According to CALVERT, the soft soaps usually made for dyers' use are not indiscriminately applicable to all colours. To produce the maximum effect in brightening the shade, the soap should be composed of—

| | | | For Madder Colours. | | |
|-------------|---|---|---------------------|-----|--------|
| | | | Purples. | | Pinks. |
| Fatty acids | . | . | 60.4 | ... | 59.23 |
| Soda | . | . | 5.6 | ... | 6.77 |
| Water | . | . | 34.0 | ... | 34.00 |
| | | | 100.0 | ... | 100.00 |

Soap for Removing Stains.‡—22 lb. of the best white soap are reduced to thin shavings, and placed in a boiler together with water 8½ lb. and ox-gall 13¼ lb. Cover up,

* MORFIT.

† SPON'S "Encyclopædia," ii. 519.

‡ "Year Book of Pharmacy," 1885, p. 286.

and allow to remain at rest all night. In the morning heat gently, and regulate so that the soap may dissolve without stirring. When the whole is homogeneous and flows smoothly, part of the water having been vaporized, add turpentine 9 oz. and benzine (best clear) $7\frac{1}{2}$ oz. Mix well, and, while still in the fused state, colour with ultramarine, add ammonia, pour into moulds, and stand for a few days before using. The product is said to act admirably.

Another formula, which requires more skill than the former to prevent the soap coming out unevenly, is the following:—Cocoa-nut oil 27.5 lb., tallow 2.2 lb., soapstone 4.4 lb., caustic-soda lye (sp. gr. 1.349) 15.4 lb., ox-gall 0.6 lb., turpentine 0.3 lb., benzine 0.1 lb., brilliant green 0.1 lb., ultramarine green 0.05 lb. Melt the fat, add the soapstone and colour, cool to 68° F. (20° C.), and then add the solution of soda. When all is well united and mixed, add very gradually the gall, continuing the agitation, without intermission, for some time after all has been added. Should any separation take place, cover the boiler for a few seconds, and, if this does not help, fire up again, and continue stirring. Lastly, add the turpentine and benzine. Pour into moulds, and stand before using. This preparation, when properly applied with a brush, is said to remove the most refractory stains without injury to the cloth.



CHAPTER XI.

VARIOUS SOAPS AND SOAP POWDERS.

C. D. Abel's Process.*—This process aims at the production of a hard soap which shall be practically almost completely freed from the lyes, and shall contain much less salt than ordinary curd soap, while at the same time a much harder and more neutral product is obtained, containing also less water (from 20 to 25 per cent.) than that obtained in the ordinary way. The soap, separated by salt as usual, and before its separation from the lye by complete cooling has taken place, is introduced into a *centrifugal machine* driven at a high speed, and is subjected while hot to centrifugal action for from four to at most twenty minutes. By this means the separation of cocoa-nut-oil soap can be perfectly effected.

Cold-water Soap.—This is a recent make of soap which, as CARPENTER states,† was at first made from very soft fatty materials, but containing a very small amount of water. It may also be made by drying "neat-soap," fitted in the ordinary way, till about one-third of its water has been driven off. Sometimes the term is applied to heavily watered soaps. Potassium and sodium carbonates are frequently added to increase the lathering property.

* English patent 6,472, April 17, 1884; "J. Soc. Chem. Ind." 1885, p. 226.

† "Soap, Candles, &c.," p. 195.

The following is the composition of a genuine cold-water soap (CARPENTER):—

| | |
|----------------------------|-------|
| Fatty acids | 70.2 |
| Soda—as soap | 7.3 |
| „ in other forms | 1.8 |
| Silica | 1.6 |
| Neutral salts | 0.4 |
| Water | 22.0 |
| <hr/> | |
| Total | 103.3 |

Eichbaum's Soap.—In order to make a soap from strongly smelling fish fats, F. EICHBAUM* takes 400 kilos. of the fat, 25 kilos. raw palm oil, 250 kilos. lye of 12° B., and warms up. A further similar amount of lye of 15° B. is added, and the thoroughly mixed mass allowed to boil till clear and free from scum, more lye being added when necessary. The mass is then poured in a thin stream through 20° lye, 50 kilos. powdered rosin are added gradually, and then 40 kilos. lye of 20°, and the mass boiled. When ready, the soap is salted in the ordinary way. The addition of the rosin is said to lessen the fishy smell considerably.

Kottula's Compact Neutral Soap.†—This soap is prepared by combining any of the usual fats or oils with concentrated soda lyes and lime liquor. The soda lye is concentrated to about 28° B., and purified by boiling for half an hour with alum, in the proportion of 4 to 4½ lb. to every cwt. of lye. The vessel is then removed from the fire, alum again added, in the proportion of about 2 to 2½ lb. to each cwt. of lye, and the liquid is stirred till the alum dissolves, after which the vessel is covered, and the whole is left to settle and become clear. The lime liquor is pre-

* "J. Soc. Chem. Ind." 1886, p. 495.

† RICHARDSON and WATTS, "Technology," vol. i. pt. iii. p. 721.

pared by combining water with lime, and then adding to each cwt. of lime liquor about $1\frac{1}{2}$ to $1\frac{3}{4}$ lb. of sal ammoniac. The liquid is boiled for about half an hour, and then allowed to settle and become clear; or the sal ammoniac is added to the lime liquor while hot, and stirred for about half an hour.

Ten tons of fatty matter, with or without rosin, 9 tons of lye prepared as above, and 13 tons of lime liquor will produce a superior compact neutral soap, which may be coloured, mottled, or perfumed by the usual processes.

Kottula's Hand or Skin Soap.—The fatty matters are mixed with highly concentrated soda lyes purified with a certain quantity of alum and sal ammoniac, whereby a neutral soap is said to be obtained cheaper and better than by any other process.

The mode of procedure is thus described:—"I prepare the highly concentrated lyes by boiling until they reach, say, about 30° to 33° B., add about 5 lb. of alum to each cwt. of lye, and boil together for about half an hour. I remove the lyes and alum from the heat, and add to each cwt. 1 lb. of sal ammoniac, stir for half an hour, cover, and allow the mass to settle and become perfectly clear. To obtain the lyes stronger than 33° , I make a second addition of alum, but in smaller proportion. To obtain lyes of 42° , I make a third addition of alum, and then add the sal ammoniac. I melt a quantity of any fatty matter used in soap-making, and, while still hot, stir, and add the lyes, prepared as before described, say, to every 100 lb. of fatty matter, about 100 lb. of 30° B., or 90 lb. of 33° B., or 80 lb. of 36° B., or 70 lb. of 39° B., or 60 lb. of 42° B.; continue to agitate the mass till it becomes thick, and when thick it can be transferred to the frames. After the soap is finished, it may be coloured, mottled, or perfumed in the usual way."

of Soap in Small Quantities.*—The
 ali Company, of St. Helens, Lancashire,
 and 98 per cent. caustic soda in a *fine powder*,
 cans holding from 10 lb. to 4 cwt. This
 does not deliquesce or melt away like the
 caustic soda, and any quantity may be taken
 and the remainder will not deteriorate, even
 be left open for some days. No boiling
 ed, and it is perfectly easy to make a few
 at a time with this alkali. The following
 etly followed, will, it is claimed, always suc-

etly 10 lb. of double refined 98 per cent.
 powder (Greenbank), put it into any can or
 ons of water, stir it once or twice, when it
 solve and become quite hot. Let it stand
 us made is cold.

it, and place in any convenient vessel for
 of clean grease, tallow, or oil (*not* mineral
 or tallow be used, melt it slowly over a fire
 l, and of a temperature not over 100° F. If
 heating is required.

lye slowly *into* the melted *fat*, or oil, in a
 ontinuously, at the same time stirring with a
 rrer about 3 inches broad. Continue gentle
 the lye and fat are thoroughly combined and
 consistence of honey. Do not stir too long,
 re will separate again. The time required
 hat with the weather, and the kind of tallow,
 used; from fifteen to twenty minutes is gene-
 t.

ENZIES, "Chemist and Druggist," 1880, p. 339.

4. When the mixing is completed, pour off the liquid soap into any sufficiently large square box for a mould, previously damping the sides with water so as to prevent the soap sticking. Wrap up the box well with old blankets, or, better still, leave it in a warm place until the next day, when the box will contain a block of 130 lb. of soap, which can afterwards be cut up with a wire.

If the grease or tallow be not clean, or contain *salt*, it must be *rendered*, or purified, by boiling with water, so as to throw out the impurities. The presence of salt would spoil the operation entirely, but discoloured or rancid fat is quite admissible.

If the soap turn out streaky and uneven, it has not been thoroughly mixed. If very sharp to the taste, too much soda has been taken; if soft, mild, and greasy, too little. In either case it must be thrown into a pan and brought to a boil with a little more water. In the first case, boiling is all that is necessary; in the others, a little more oil or a little more soda must be added.

Any smaller quantity of soap than the above may be made by taking the ingredients in smaller proportions, but it is not advisable to make more than double the quantity prescribed, as it is difficult to work more by hand. By working successive batches, however, a person could turn out 2 tons of soap in a day simply with apparatus obtainable in any household.

By adding a few drops of an essential oil just when the mixing is complete, a toilet soap is produced. Oil of mirbane (artificial almond oil) is the cheapest, but the perfume is not nearly so pleasant as real almond oil, citronella, or oil of cloves. When made with clean grease, or tallow, or light-coloured oil, the soap produced is quite white.

Sand Soap.—C. ROTH* recommends the following method to prepare a good sand soap:—

100 lb. of cocoa-nut oil are saponified with about 200 lb. of lye at 20° B. The soap is then hardened by the addition of about 8 lb. of salt dissolved in water to a density of 15° B., with the addition of 6 to 8 lb. of soda ash. The mixture is now covered up and the foam allowed to subside. After standing five or six hours the foam is skimmed off, and from 100 to 150 lb. of dry sifted sand is thoroughly *crutched* into the mass, and the crutching is continued till the whole is cool. The soap is very firm and hard.

The soap is especially suited for the use of workmen engaged in rough and dirty avocations. If desired, it may be perfumed by the addition of 100 grams each of the essential oils of lavender, thyme, and coriander.

Sodium Aluminate Soap.—The Pennsylvania Salt Manufacturing Co. issue with their boxes of *Natrona refined saponifier* (see p. 32), the following directions for making soap *without using scales, weights, or measures*:—"Cut out one end of this box, empty its contents into a pan, fill the box three times with cold water, and pour it on the *saponifier*, stirring until the latter is all dissolved. Into another pan introduce as much rendered grease or fat as will fill the same box five times. Now pour the dissolved *saponifier* into the rendered fat, and stir for a few minutes until thoroughly mixed. Let the whole stand till next day. Cut into small pieces, and pour in two more boxes of water. Heat and stir till the soap is all dissolved, and free from lumps. Remove the heat, and when cool cut into bars or cakes.

"In very cold weather the water should be warmed a little. The rendered grease should be about as thick as honey, and not very hot."

* "Seifensieder Zeitung," 1884, p. xxi.

Soap Powders.*

Borax Soap Powder.—Curd soap in powder 5 parts, soda ash 3 parts, sodium silicate 2 parts, borax (crude) 1 part.

Each ingredient must be first thoroughly dried, and all mixed together by sieving.

London Soap Powder.—Yellow soap 6 parts, soda crystals 3 parts, pearl ash $1\frac{1}{2}$ part, sodium sulphate $1\frac{1}{2}$ part, palm oil (bleached) 1 part.

These ingredients are mixed as well as possible without any water, spread out to dry, and then ground into coarse powder. The palm oil imparts an agreeable odour.

Pearl Soap Powder.—Curd soap (powdered) 4 parts, sal soda (crude sodium carbonate) 3 parts, sodium silicate 2 parts.

Dried as much as possible, and intimately mixed.

Soap Extract.—Soap 14.3 parts, anhydrous soda 30 parts, and water 55 parts. Manufactured from crystallized soda and soda soap.†

Washing Powder.—A powdery mixture of 90 parts effloresced soda with 10 parts of sodium hyposulphite and 2 parts of borax.†

Wool-washing Composition.—A mixture of 35 parts of dried soda, 10 parts of powdered soap, and 10 parts of sal ammoniac.†

Universal Washing Powder.—Sodium silicate, with a small percentage of soap and starch powder.†

* "Chemist and Druggist," 1884, p. 73.

† *Ibid.* 1879, p. 243.

CHAPTER XII.

RECOVERY OF GLYCERIN FROM SPENT LYES.

Spent Lyes contain variable quantities of water, glycerin, sodium chloride, sodium sulphate, sodium carbonate, caustic soda, and small quantities of albuminous, resinous, and soapy matters.

The glycerin was formerly wasted, but of late years great attention has been devoted to its recovery, and many processes for that purpose have been patented.

KINGZETT, in a valuable paper on this subject,* classes the various processes as designed to effect the following objects:—

1°. To remove, or destroy, albuminous or soapy matters, together with any residual soap in the spent lyes.

2°. To facilitate the removal of the salt, either by employing means to diminish the solubility of the sodium chloride, in cases where that substance is used, or to substitute another which may be more readily and profitably removed.

3°. To economize the cost of concentrating the lyes to that point at which the glycerin may be at once employed for certain purposes in its then crude condition, or still further purified by distillation.

* "J. Soc. Chem. Ind." 1882, p. 78.

The following are some of the methods by which the separation of glycerin has been attempted:—

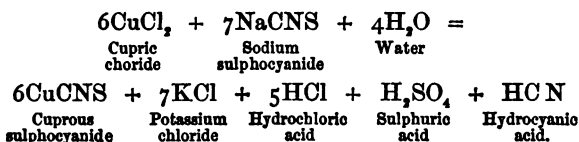
I. **Allan's Method.**—Neutralize with any ordinary mineral acid. After settling, add alum and chloride of lime, or pyroligneous acid, and stir thoroughly, or, before addition of the above, evaporate to the *salting point*. Distil with superheated steam in an apparatus furnished with an exit pipe for the removal of salt as it accumulates.

II. **Allen and Nickels' Method.**—"Lancashire lyes," in addition to the impurities already mentioned, contain sulphides, hyposulphites, cyanides, ferrocyanides, sulphocyanides, &c., from the custom of saponifying with causticized *black-ash* liquor instead of by caustic soda. These impurities make the recovery of glycerin in a satisfactory condition from such lyes a very difficult operation. A. H. ALLEN, of Sheffield, and B. NICKELS, London, have recently, however, patented a process* which promises to overcome this difficulty. The process depends upon the fact† that, when a solution of a copper salt (cuprous or cupric) is added to soap lyes previously rendered neutral or faintly acid, the sulphocyanides are wholly precipitated, together with any sulphides, cyanides, ferrocyanides, or silicates, and also with albuminous, resinous, fatty, colouring, and other organic matters. The precipitate settles with great facility, and the filtered liquid is obtained nearly colourless. The copper is recovered from the precipitate by roasting and treatment with sulphuric acid.

According to ALLEN, the following equation expresses the main reaction which occurs in the removal of the sulphocyanides by a cupric salt:—

* English patent 11,069, August 31, 1886.

† ALLEN, "J. Soc. Chem. Ind." 1887, p. 89.



If the sulphur compounds are not removed, volatile organic sulphur compounds appear in the distilled glycerin, and unfit the product for the uses of the dynamite manufacturer.*

III. Benno, Jappé, & Co.'s Method.—Instead of using sodium chloride to separate soap in the pan, Benno, Jappé, & Co. recommend the use of sodium sulphate. The lyes are then neutralized by acid sodium sulphate, and the salts removed by evaporation and filtration. The glycerin is then purified by distillation.

IV. Clolus' Method.—First neutralize with hydrochloric acid; then remove sodium chloride by means of a turbine, or by dialysis; evaporate to 32° B.; pass hot air to render the glycerin anhydrous, in which the sodium chloride is insoluble, or nearly so; or obtain anhydrous glycerin by evaporation *in vacuo*, and subsequent distillation.

V. Fleming's Method.†—FLEMING proposes to subject the spent lyes to dialysis. He shows that the four soap-works at Neuwied alone produce annually about 1500 tons of waste liquors, containing about 75 tons of glycerin. The percentage of glycerin in the lyes varies from 0.92 to 7.8. The most effectual means for removing the salts contained in the lyes previous to distillation is to subject them to osmotic motion. The lyes are concentrated in suitable pans by steam heat, and then neutralized by sulphuric acid. The quantity of acid required depends upon the amount of

* ALLEN, "J. Soc. Chem. Ind." 1887, p. 88.

† "Dingl. Polyt. Journ." ccxliii. 330-333; "Year Book of Pharmacy," 1882, p. 257.

sodium carbonate present in the lyes. As, owing to the violent evolution of carbonic acid, it is difficult to obtain a perfectly neutral solution, it is preferable to add a slight excess of acid, which, after the precipitation and separation of the sodium sulphate, is removed by lime. The liquor is re-evaporated with steam, a further (small) quantity of sodium sulphate and chloride crystallizing out on cooling. It is now osmosed and concentrated, and, after this operation, is sufficiently free from mineral constituents to be distilled, either *per se* or in conjunction with crude glycerin obtained in the manufacture of stearic acid. The loss of glycerin by distillation is very small, and, as regards the purity of the resulting product, it is shown that it fulfils all the requirements necessary for the successful preparation of dynamite. The great feature of the process is that, unlike molasses, the liquor treated does not attack parchment paper. A large quantity of glycerin remains in the osmose water, and may be recovered by concentrating and distilling the liquid.

FLEMING has also patented the use of a gutta-percha membrane; which, he states, is traversed by salt, but is impermeable by glycerin.

VI. O'Farrell's Method.—Evaporate and treat with methylated spirit, which dissolves the glycerin, and then distil. Or, the lye may be used again in the production of soap till a maximum of glycerin is obtained in a minimum of lye.

VII. Payne's Method.—Neutralize with hydrochloric, sulphuric, or nitric acids. Separate gelatinous and albuminous matters by addition of tannin. Filter, concentrate, and distil off the glycerin.

VIII. Reynolds' Method.*—The lye is first concen-

* Patent No. 1322, June 10, 1858.

trated by evaporation, and the saline matter, which gradually separates, is removed from time to time. When the fluid is sufficiently concentrated (ascertained by the boiling point having risen to 116° C.), it is transferred to a still, and the glycerin distilled off by means of superheated steam introduced into the still. The distillate is next concentrated, and brought to the consistency of a syrup in a vacuum pan. If greater purity is required, it may be obtained by repeating the process, and the little colour that remains may be removed by animal charcoal.

IX. Thomas and Fuller's Method.—Neutralize, evaporate and remove salts, and then add oleic, palmitic, or stearic acid. The neutral glycerides so obtained, after being washed, are treated, as in the candle industry, by the lime saponification process, or by superheated steam.

X. Venables' Method.—The liquor from the soap, either before or after filtration, is neutralized by means of aluminium sulphate, alum, or any soluble salt of aluminium, or any substance containing soluble alumina. The sodium hydrate and carbonate, combining with the acid, precipitate the alumina, and the alumina, combining with some of the organic matters and carrying off the rest, purifies the lyes. Filter, and concentrate. Or, instead of only neutralizing, the salt of aluminium may be added till the lye becomes acid, and it may then be rendered alkaline by addition of caustic lime or any other alkali which may be found convenient. The spent lyes may also be first partially neutralized by the addition of a small quantity of hydrochloric or sulphuric acid; the remaining free sodium hydrate will then be neutralized by the aluminium salt, which may be added to exact neutrality or to excess; in the latter case, the liquid should be afterwards neutralized, or rendered alkaline. Glycerin can then be obtained by distillation.

XI. Versmann's Method.—(1) The lyes are evaporated

until the liquor becomes so concentrated that the salts contained therein begin to crystallize out.

(2) The liquor is then cooled, and filtered to get rid of gelatin and albumen.

(3) Carbonic acid is then passed through the liquid. Sodium bicarbonate is precipitated, and this is separated in the usual way.

(4) After undergoing this treatment, the liquor is made to absorb gaseous hydrochloric acid until the remaining sodium carbonate is converted into chloride, and further until all, or almost all, the sodium chloride has been precipitated.

(5) When the chloride has been separated, the liquor, containing water, glycerin, and hydrochloric acid, is evaporated so as to get rid of the acid, which is absorbed in water for using afresh.

(6) The dilute glycerin remaining can be purified by filtering through animal charcoal, or by concentrating and distilling.*

XII. Young's Method.—Evaporate the lyes by means of superheated steam. Neutralize by sulphuric acid, add calcium carbonate, filter, and treat with a centrifugal machine (such as is used to separate sugar from molasses). Evaporate the separated crude glycerin, and distil.

* VERSMANN, "Chem. News," June 24, 1881.

CHAPTER XIII.

TESTING SOAPS.

It is impossible to know the real composition of a soap, and consequently its value, except by analysis. For many purposes it is sufficient to ascertain the proportion of water, fatty acids, and alkali, while for others a full analysis is desirable.

Samples.—The sampling of soap for analysis requires great attention. The difficulties to be overcome are thus exemplified by R. S. TATLOCK:—A delivery of fifty boxes of Italian olive-oil soap has to be sampled, the goods being sold on the basis of 62 per cent. of fatty acids. The quality of the total contents of each box may be different. The proportion of valuable ingredients cannot be the same in every bar of a given box, from the fact that some of the bars have only their ends exposed to the outside, others their ends and one side, a third series their ends and two sides, while a fourth may be completely inside. Then, again, the bars selected for analysis, for the same reasons, are also in different conditions of dryness, and the sampling by the analyst of each bar for his working sample becomes a matter for grave consideration. The problem is, What proportion of the fifty boxes are to be opened, from what position in the box are the bars to be selected, and in what

* "Journ. Soc. Chem. Ind." 1884, p. 307.

ways are the selected bars to be punched out so as to give an *accurate average* for analysis? Each 1 per cent. of fatty acids represents about £1 12s. 6d. on every £100 value, but probably any hard-and-fast method would be completely upset by the adoption of a different form or size of box.

The following are some of the schemes that have been proposed for conducting the analysis of soap in a systematic manner:—

Dr. Leeds' Method.*—(1) *Water*.—Weigh out about 5 grams in very fine shavings on a dried, weighed, plaited filter. Dry at 110° C. till weight is constant. The loss is water.

(2) *Uncombined Fat*.—Transfer the filter containing the dried soap to a funnel connected with the return cooler, such as is used in the determination of the albuminoids in milk, and connect with the funnel a small tared flask containing 50 c.c. petroleum ether. Or, the filter may be placed in the ordinary Soxhlet apparatus. After complete extraction, distil off the ether, and the residue in the flask, dried at 110°, will be the uncombined fat.

(3) *Free Alkali*, (4) *Combined Alkali*, (5) *Glycerin*.—Allowing the filter with the soap, now free from water and uncombined fat, to remain in the apparatus, attach to it a flask containing about 75 c.c. of 95 per cent. alcohol, and extract.

To the alcoholic solution add a few drops of phenolphthaleïn; if free alkali be present, neutralize with normal sulphuric acid, and calculate the amount of uncombined soda. (Free alkali, if present, may be detected qualitatively, by applying to a freshly cut surface of soap a drop

* "Chem. News," October 5, 1883, pp. 166-8; "J. Soc. Chem. Ind." 1883, p. 479. A tabular arrangement of Dr. LEEDS' scheme is given on pp. 184, 185.

of mercurous nitrate, which will give a greyish tint, or a drop of phenol-phthaleïn, which will give a pink coloration.)

After neutralization, add a large excess of water and boil off the alcohol. To the aqueous solution add a large excess of normal sulphuric acid, noting the quantity added. Boil, cool, and decant through a small filter; wash with hot water, and decant, after cooling, through the filter until litmus-paper is no longer reddened by the washings. The filtrate contains the combined soda and the glycerin; the residue consists of the fatty acids and resin. Neutralize the filtrate with normal soda solution, and calculate the amount of combined soda as Na_2O . Evaporate to dryness, and extract the glycerin with absolute alcohol. Transfer the alcoholic solution to a tared flask, distil off the alcohol, dry at 100°C ., and weigh the residue as glycerin.

(6) *Fatty Acids and Resin*.—With a little petroleum ether, dissolve the small amount of the fatty acids and resin that may be on the filter through which the decantation was effected, add the solution to the larger bulk in the beaker, evaporate off the ether, dry at 100° , and weigh the combined fatty acids. Multiply this result, after deducting the amount of resin, by 0.97; the product is the fatty anhydrides.

(7) *Resin*.—The resin is separated according to the method proposed by GLADDING.* About 0.5 gram of the mixture of fatty acids and resin are dissolved in 20 c.c. of strong alcohol, and, with phenol-phthaleïn as an indicator, soda is run in to slight super-saturation. The alcoholic solution, after boiling for ten minutes to insure complete saponification, is mixed with ether in a graduated cylinder till the volume is 100 c.c. To the alcoholic and ethereal solution 1 gram of very finely powdered neutral silver

* "J. Soc. Chem. Ind." i. 205; "Chem. News," April 14, 1882.

| | | | |
|--|---|--|---|
| Weigh out 5 grams. Dry at | | | |
| Treat with | | | |
| Residue is soap and mineral | | | |
| Extract is soap (<i>fatty anhydrides, resin, and combined alkali</i>), <i>glycerin</i> , and <i>free alkali</i> . Add 2 or 3 drops of phenol-phthaleïn. If necessary, titrate with normal sulphuric acid. | | | |
| Extract is <i>uncombined fat</i> . Dry at 110° C., and weigh. H_2SO_4 used corresponds to <i>free alkali</i> . Calculate as $NaHO$. | Add a large excess of water, and boil off the alcohol. Decompose with excess of normal H_2SO_4 . Note quantity added. Boil, filter, and wash. | | |
| | Filtrate. — <i>Combined soda and glycerin</i> . Titrate with normal soda solution. | | |
| | H_2SO_4 in excess of soda, used corresponds to <i>combined soda</i> in soap. Calculate as Na_2O . | After titration with soda, evaporate to dryness on the water-bath. Treat with absolute alcohol. Evaporate the alcoholic solution to dryness in a tared basin, and weigh as <i>glycerin</i> . | Residue. — <i>Fatty acids and resin</i> . Dry at 110° C., and weigh. Dissolve an aliquot part in 20 c.c. strong alcohol, and, using phenol-phthaleïn as indicator, saponify with soda in slight excess. Boil, cool, and add ether to 100 c.c. Decompose with $AgNO_3$ in fine powder, shake well for ten minutes, and allow to settle. |
| | | Precipitate is <i>stearate, palmitate, and oleate of silver</i> . | Solution. — <i>Resinate of silver</i> . Filter 50 c.c. from the total 100 c.c. Decompose with 20 c.c. HCl (1 : 2). Allow the $AgCl$ to settle, and evaporate an aliquot part of the ethereal solution in a tared dish. Dry at 110° C., and weigh. After applying the correction for oleic acid, the weight corresponds to the <i>resin</i> . This weight, subtracted from the combined weight of fatty acid and resin, gives the <i>fatty acids</i> . |

for Soap Analysis.

100° C. Loss corresponds to water.

petroleum ether.

constituents. Treat with alcohol.

Residue.— Na_2CO_3 , $NaCl$, Na_2SO_4 , sodium silicate, starch, and insoluble residue. Wash with 60 c.c. water.

Filtrate.— Na_2CO_3 , $NaCl$, Na_2SO_4 , and sodium silicate. Divide into four equal parts.

Residue.—Starch and other insoluble matter. Dry the filter, and weigh.

Na_2CO_3 .
Titrate with normal H_2SO_4 , and calculate as Na_2CO_3 .

$NaCl$.
Titrate with $AgNO_3$, or weigh as $AgCl$. Calculate as $NaCl$.

Na_2SO_4 .
Weigh as $BaSO_4$. Calculate to Na_2SO_4 .

Sodium silicate.
Decompose with HCl , and determine soda combined in silicate and silica.

Starch.—Boil with dilute acid to convert into $C_6H_{12}O_6$, and titrate by FEHLING'S solution. Subtract the weight of starch so found from the total residue. The difference is the insoluble mineral constituents.

nitrate is added, and the contents of the cylinder are shaken thoroughly for ten or fifteen minutes. After the precipitate has settled, 50 c.c. are measured off, and, if necessary, filtered into a second graduated cylinder. A little more silver nitrate is added to see if the precipitation is complete, and then 20 c.c. of dilute hydrochloric acid (1 : 2) to decompose the silver resinate. An aliquot part of the ethereal solution is evaporated in a tared dish and weighed as resin, deducting a small correction* (0.00235 gram for 10 c.c.) for oleic acid. The amount of resin subtracted from the combined weight of fatty acids and resin, as found before, gives the fatty acids.

(8) *Sodium Carbonate*; (9) *Sodium Chloride*; (10) *Sodium Sulphate*; (11) *Sodium Silicate*.

(12) *Insoluble Residue*.—The filter in the funnel connected with the return cooler, after treatment with alcohol, contains the mineral constituents of the soap. The contents of the filter are washed with cold water till the washings amount to 60 c.c. The filter is then dried, and weighed. The weight gives the insoluble residue and starch. The starch is converted into glucose with dilute acid, and titrated with FEHLING'S solution. The weight of starch so found, subtracted from the total weight of insoluble residue and starch, gives the insoluble mineral constituents. The aqueous solution of 60 c.c. just mentioned is divided into four equal parts, in one of which is determined the sodium carbonate by titration, and, in the other parts, the chloride,

* Dr. C. R. A. WRIGHT and C. THOMPSON ("J. Chem. Soc." 1886, p. 175) consider GLADDING'S process more satisfactory than any other for the estimation of resin, but they show that this correction-factor is by no means universally applicable. With pure stearic or oleic acid, it is much too large; with acids from castor oil, far too small; but with mixtures such as are likely to occur in the manufacture of soaps the results afforded appear to be not far from the truth.

the sulphate, and the silicate, respectively, by any convenient method.

Filsinger's Scheme.*—(1) *Water*.—In the case of *hard soap*, 5 grams, scraped from the sides and centre of a fresh section, are first very gently warmed, to avoid direct melting, then over a water-bath, and finally in a drying box at 100° C., until the weight remains constant.

For *soft soap*, 10 grams are taken, spread in a thin layer over a large watch-glass, and treated in the same way.

(2) *Unsaponified, or Free, Fat*.—The dry residue from (1) is finely powdered, and washed on a filter three or four times with lukewarm petroleum ether. The filtrates are collected in a weighed beaker, evaporated, dried, and weighed.

(3) *Free Alkali*.—The residue from (2) is digested for a short time with alcohol (95 per cent.), slightly warmed, filtered, the residue on the filter washed with warm alcohol, and the filtrate, to which a few drops of a phenol-phthaleïn solution are added, titrated with $\frac{N}{10}$ sulphuric acid.

(4) *Foreign Bodies*.—These are found, by the usual methods, together with the chlorides, sulphates, and carbonates of the alkalies on the filter in (3).

(5) *Fatty Acids*.—The neutralized alcoholic solution from (3) is mixed with water in a moderate-sized porcelain basin, the fatty acids precipitated by sulphuric acid, and, after melting and settling, 5 grams of dry wax are added. When the whole is cool, the fat-acid wax is removed, washed with water and alcohol, dried without melting, and cooled. The weight — 5 grams = the quantity of fatty acids.

(6) *Glycerin*.—The liquid from the cake of fatty acids is treated with a small excess of barium carbonate, heated,

* "Chemiker Zeitung," April 17, 1884; "Chemist and Druggist," 1884, p. 290.

filtered, the filter washed with hot water, and the filtrate evaporated to dryness. The residue is repeatedly washed with alcoholic ether, the filtrate evaporated in a porcelain dish, dried at a temperature of 70°C. , and weighed.

(7) *Total Alkali*.—10 grams of another portion of soap, prepared as in (1), are dried in a platinum dish, and then heated till all the fatty acids have been destroyed. The porous carbonaceous residue is boiled with water, filtered into a $\frac{1}{4}$ -litre flask, and the filter washed with hot water till the washings cease to give an alkaline reaction. The bulk is then made up, the whole well mixed, and 25 c.c. (= 1 gram soap) of the solution are titrated with sulphuric acid. The result represents the amount of total alkali, and, after deducting the quantity of free alkali, found by (3), the remainder is the proportion of alkali combined with fatty acids, and existing as carbonate and silicate.

(8) *Chlorine*.—The neutral titrated solution from (7) may be used for the determination of chlorine by $\frac{1}{10}$ silver solution.

(9) *Silicic Acid*.—75 c.c. of the solution from (7) are treated with excess of hydrochloric acid, evaporated to dryness, treated with water, filtered, and the residue ignited and weighed as silica.

(10) *Sulphuric Acid*.—The filtrate from (9) is boiled, and, while boiling, barium chloride is added, the precipitated barium sulphate washed, dried, and weighed, and calculated as sodium, or potassium, sulphate.

(11) *Potash and Soda*, if both are present, must be determined in the usual way by platinum chloride.

In many methods of analysis met with in text-books, directions are given to weigh out for each operation small portions (1 to 5 grams) of the sample. In a communication from the laboratory, Owens College, Manchester, the

following objections are taken to this method : *—1°. Soap is extremely variable in composition, and considerable variations are possible in a single sample. 2°. It is continually losing water by evaporation from its surface. As the soap is usually weighed in the form of thin shavings, the surface exposed is, in relation to the weight taken, very considerable.

These two sources of inaccuracy may be obviated thus :—A section is cut through the bar at right angles to its length, weighing 60 to 80 grams. This is dissolved in distilled water by the aid of heat, and the bulk made up to 1 litre (at 60° F.). 50 c.c. are taken out for each of the following operations, immediately after well shaking the liquid, as some of the alkaline salts of the fatty acids separate out from the solution on cooling.

1°. *Total Alkali*.—50 c.c. of the solution are diluted to about 200 c.c., coloured faintly with eosine, and standard acid run in, taking care to stir briskly with a glass rod. The neutral point is extremely well marked by the decolorization of the whole. The cause of the disappearance of the colour is the union of the fatty acids with the eosine at the moment of their complete separation.

2°. *Uncombined Alkali*.—50 c.c. are added to 300 c.c. of a saturated solution of common salt, which, of course, must be neutral to test-paper, and the volume made up to 400 c.c. The neutral alkaline salts of the fatty acids (*i.e.*, true soap) are precipitated. Any excess of alkali present remains in solution, and is determined in an aliquot part of the filtrate. The filter must not be moistened previous to filtration. The total uncombined alkali is calculated therefrom, and deducted from the *total alkali*

* "Chem. News," January 5, 1877; URE'S "Dictionary," iv. 822.

already found. Thus the *combined* and *uncombined* alkali are determined. (This method is less reliable than the alcoholic treatment, pp. 182 and 187.)

3. *Fatty Acids*.—50 c.c. of the solution are introduced into a stoppered separating funnel, decomposed with excess of acid, and agitated with carbon disulphide until the liberated fatty acids are dissolved. The disulphide solution is then drawn off into a tared flask, and the aqueous solution is washed once or twice with small portions of disulphide, and the washings are added to the contents of the flask. The disulphide is then distilled or evaporated off. The fatty acids are purified from the last traces of carbon disulphide by heating the flask for a short time at 100° C. After cooling, the weight, less the tare of the flask, gives the weight of the fatty acids.

Ether may be used instead of CS_2 , but there is this disadvantage, that in the separator it will form the *upper* layer, whereas carbon disulphide forms the *lower*, and hence is more readily manipulated.

4. *Water*.—The direct estimation is effected by evaporating 50 c.c. of the solution to dryness on the water-bath, and finally in an air-bath at from 100° to 120° C. The residue is anhydrous soap, and from its weight the percentage in the sample is calculated.

When thin shavings of soap are dried in the usual manner, the author of the process considers that the last portions of water, amounting to from 1 to 2 per cent., are not driven off.

5. *Mineral Impurities* and *Unsaponified Fat* may be detected by taking the dried soap from the preceding operation, dissolving in strong alcohol, and filtering through a funnel surrounded by a hot-water jacket. The former remain on the filter as an insoluble residue, the weight of which may be readily ascertained.

The alcoholic filtrate is evaporated with successive additions of distilled water. Any unsaponified fat or resin is thus separated from the soap, which remains in the aqueous solution. This solution may be used for 1, 2, or 3.

Estimation of Detergent Value of Soap.

The following volumetric method affords a rapid means of comparing commercial soaps as to their respective detergent powers.* A standard soap is first chosen, by means of which the relative saponifying value of any other soap may be ascertained. The most suitable standard is the mottled Marseilles soap, generally known as Castile soap. The composition of this soap is, in round numbers:—

| | | | | | | | | |
|-------------|---|---|---|---|---|---|---|-----|
| Soda | . | . | . | . | . | . | . | 6 |
| Fatty acids | . | . | . | . | . | . | . | 64 |
| Water | . | . | . | . | . | . | . | 30 |
| | | | | | | | | 100 |

1 gram of this soap will be exactly neutralized by 0.1074 gram pure calcic chloride, or 10 grains by 1.074 grain. Therefore, a solution of 1.074 gram CaCl_2 in a litre of water, or 10.74 grains in 10,000 grains, will suffice to neutralize respectively 10 grams or 100 grains of the standard soap dissolved in the same volume.

PONS† applies the above process in the following way:—

1°. 10 c.c. of the standard calcic solution are placed in a stoppered bottle—holding 70–100 c.c.—with about 20 c.c. distilled water.

2°. 10 grams of the sample of soap are now treated with 100 c.c. alcohol (sp. gr. 0.825) by means of rubbing or shaking with gentle heat; the real soap dissolves, and leaves all mineral or foreign matters, which may be filtered off,

* SUTTON, "Volumetric Analysis," p. 53.

† "Journ. de Pharm. et Chem." April 1865, p. 290.

and afterwards, if necessary, examined. The filtrate is diluted to 1 litre with distilled water.

3°. This solution is then cautiously run from a burette into the 10 c.c. of lime solution, with frequent shaking, until a lather is obtained.

4°. The 10 c.c. of lime solution divided by the number of c.c. of soap solution required will show the richness of the soap compared with the standard. Thus, if 10 c.c. only are used, the soap under examination is of the same quality as the standard; if 15 or 20 c.c. are required, the percentage will be $\frac{10}{15} \times 100 = 66$ per cent., or $\frac{10}{20} \times 100 = 50$ per cent., and so on.

A. H. ALLEN's modification of this process is as follows:—He ascertains what measure of a standard solution of the sample must be added to 50 c.c. of a very dilute solution of calcium chloride, or sulphate, solution, in order to obtain a persistent lather on shaking. The soap solution is made by dissolving 10 grams of the sample, as in the preceding method, in proof spirit (sp. gr. .920), filtering, and diluting the filtrate with proof spirit to 1 litre. The test is made exactly as in determining the hardness of waters, the soap solution being added to the standard hard water in small quantities at a time till a lather is obtained, on shaking, which persists for at least five minutes when the bottle used for the operation is placed on its side. The standard hard water may be conveniently prepared by exactly neutralizing 40 c.c. of decinormal sulphuric or hydrochloric acid by cautious addition of lime water, and diluting the solution to 1 litre, when it will have a hardness of 14 degrees in CLARK's scale.*

M. Cailletet's Method† of determining the Fatty

* "Commercial Organic Analysis," second edition, ii. 250.

† "Bulletin de la Société industrielle de Mulhouse," No. 144, tome xxix. p. 8.

Acids.—A standard acid is prepared by diluting 189.84 grams of strong sulphuric acid to 1 litre at 15° C. Of this acid 10 c.c. neutralize 1.2 gram of soda, and this quantity is therefore sufficient to decompose 10 grams of soap, as the amount of alkali present never exceeds 12 per cent.

Into a tube of 50 c.c. capacity, and divided into 100 equal parts, are poured 10 c.c. of the standard acid and 20 c.c. of turpentine; 10 grams of the sample in thin shavings are then added. The tube is then closed with the stopper, or with a good cork, well shaken for a few minutes till the soap is dissolved, and then left at rest for fifteen minutes, or till the oily solution of the liberated fatty acids has completely separated from the watery liquid.

In reading off the volume of the turpentine solution after the experiment, a deduction of half a division, or $\frac{1}{4}$ c.c., is made, to allow for the diminution of the capacity of the tube owing to the thin film of watery liquid which adheres to the inner surface of the tube. If the oily stratum occupies 53 divisions, or 26.5 c.c., then, deducting 20 c.c. for the volume of turpentine employed, the remainder, 6.5 c.c. (or 65 per cent.), is the volume of fatty acids in the soap.*

Determination of Glycerin.—Many methods have been proposed to effect this. The usual method is to dissolve a known weight of soap in water, acidulate with sulphuric acid, filter off the separated fatty acids, neutralize with sodium carbonate, evaporate to dryness, and treat the residue with strong alcohol, which dissolves glycerin, and leaves behind sodium salts. Dr. WRIGHT† points out that this residue left on evaporation is rarely pure, most soaps containing small quantities of substances derived from the original fats and oils, which are soluble in the acidified

* This \times by their sp. gr. = percentage by weight.

† Cantor Lectures on "Toilet Soaps," May 1885, p. 40.

aqueous fluid, and thus become more or less dissolved out by the alcohol, so that soaps containing no trace of glycerin will still furnish small percentages of alcoholic extract when thus treated. Sodium chloride, being slightly soluble in ordinary alcohol, may also be contained in the extract. By re-dissolving the dried extract in absolute alcohol, and adding one and a half times its volume of ether, a certain amount of substances other than glycerin is generally precipitated, but, in most cases, even this purification fails to yield pure glycerin, especially in presence of sugar.

Dr. WRIGHT found the following method gave fairly accurate results:—The aqueous acid solution obtained after separating the fatty acids as above described is rendered strongly alkaline with aqueous caustic soda, and then dilute copper sulphate solution is dropped in with agitation, until the copper hydroxide thus formed begins to fail to dissolve. The filtered blue solution is compared calorimetrically with a known quantity of a standard solution of glycerin treated side by side in the same way. When sugar is present, the alcoholic extract, obtained as above, must be heated with dilute sulphuric or other acid, for some time, so as to *invert* the sugar. The fluid is then rendered alkaline, and copper sulphate dropped into the boiling liquid as long as suboxide of copper is reduced, after which the calorimetric estimation of the glycerin is proceeded with as before, the comparison being preferably made with a known solution of glycerin and cane sugar treated simultaneously with the sample under examination.

With care and practice, fairly good results can be thus obtained, more especially when sugar is absent. The following figures illustrate the numbers which WRIGHT obtained in analyses for glycerin, the values being percentages:—

| Nature of Soap. | Crude Alcoholic Extract. | Extract purified by Ether. | Glycerin indicated by Copper Test. |
|--|--------------------------------|----------------------------------|--|
| Opaque untinted soap, moderate quality . . . | 7.0 | 6.1 | 6.00 |
| High-class Parisian glycerin soap, not transparent . | 8.1 | 7.9 | 8.00 |
| Cold process soap + much unsaponified fat . . . | 6.6 | 4.9 | 4.75 |
| British so-called glycerin soap, opaque . . . | 7.9 | 7.9 | 0.60 |
| British transparent soap, without sugar . . . | 19.0 | 17.6 | 15.00 |
| Ditto + 10 per cent. of sugar | 6.1 | 4.0 | 0.00 |

Dr. WRIGHT states that the entire absence of glycerin from a toilet soap necessarily, proves that the whole mass has been prepared either by a boiling process, or by saturating a free fatty acid, as oleic acid, with alkali, or by both processes combined. On the other hand, the presence of a quantity not far removed from the percentage of combined alkali, expressed as Na_2O , suggests that the whole has been probably prepared by the cold process, for, as ordinary oils and fats are substantially tri-glycerides, 1 equivalent of fatty matter will yield 92 parts of glycerin, and fatty acids equivalent to 93 of Na_2O . When larger quantities of glycerin are present, extra glycerin must have been added to the materials during the manufacture of the soap. When small quantities only are present, constituting only a fraction of the percentage of combined alkali, expressed as Na_2O , the soap is probably a blended mass, consisting partly of boiled and partly of cold-process soaps.

MUTER'S METHOD.*—This may be used for the determination of glycerin in soap and soap-lyes. The process is based

* "Analyst," 1881, p. 41; "Year Book of Pharmacy," 1881, p.

on the power of glycerin to arrest the precipitation of cupric hydrate by alkalies. The *modus operandi* is as follows:—

(1) Take 1 gram of absolute glycerin and wash it into a long, stoppered, graduated tube, having a stop-cock at 50 c.c. from the bottom.

(2) Add 50 c.c. of a strong solution of potassium hydrate (1 in 2) and then a weak solution of cupric sulphate very gradually, and with constant shaking, until a fair amount of cupric hydrate is produced which remains undissolved; make the whole up to a given bulk, close the tube, and set it aside to settle.

(3) When perfectly clear, run off from the tap into a beaker a given volume of the deep-blue liquid, and add to it the slightest possible excess of nitric acid.

(4) Pour in a definite excess of ammonium hydrate, bring the beaker under the burette charged with volumetric solution of potassium cyanide, and run in till decolorized.

The number of c.c. of the cyanide used, after calculating to the whole bulk originally in the tube, represents 1 gram of glycerin. The result has, however, to be corrected by going through a blank experiment, with the same amounts of everything, but *without glycerin*, and deducting the c.c. of cyanide taken from that previously found. This precaution is necessary because copper hydrate is not quite insoluble in the strong alkali, but, once made and deducted, the difference gives the true value in glycerin of the cyanide solution, and, when that has been thus standardized, any number of estimations can be quickly made.

The glycerin to be determined must first be isolated, as free from intermixture as possible, as previously described.

Determination of Carbolic Acid in Soap.*—(1) 5 grams of the soap are dissolved in warm water, with addition of from 20 to 30 c.c. of a 10 per cent. solution of caustic soda, according to the proportion of phenols believed to be present.

(2) The cooled solution is then agitated with ether, and the ethereal layer separated and evaporated at a low temperature. The weight of the residue gives the amount of hydrocarbons, &c., in the quantity of the sample taken. The odour towards the end of the evaporation and that observed on heating the residue will give considerable information as to the nature of the admixture. Odours suggesting gas-tar and burning gutta-percha are very common.

(3) The alkaline liquid separated from the ether is then treated in a capacious separator with an excess of strong brine, which completely precipitates the fatty acids as sodium salts. The liquid is well agitated to cause the soap to filter, and is then passed through a filter. In cases where the soap does not readily coagulate, an addition of a small quantity of tallow or palm-oil soap, previously dissolved in water, will usually overcome the difficulty. The precipitated soap is washed twice by agitating it with strong lime, the washings being filtered and added to the main solution, which is then diluted to 1 litre.

(4) 100 c.c. of this solution (= 0.5 gram of the sample of soap) are then placed in a globular separator, and acidulated with dilute sulphuric acid, when it should remain perfectly clear. Standard bromine water is then added from a burette, the stopper of the separator inserted, and

* A. H. ALLEN, "Analyst," 1886, p. 103; "Year Book of Pharmacy," 1886, p. 138.

bolic acid) has been employed, the bromo-derivative will be deep yellow, orange, or red, with little or no tendency to crystallize, and the weight will not afford even a rough indication of the amount of coal-tar acid present.

It must be borne in mind that in this process a loss of 2 or even 3 per cent. of carbolic acid is liable to occur through evaporation.

The following table shows some of the results which Mr. ALLEN obtained by the analysis of representative samples of commercial carbolic soap:—

| No. | Description of Soap. | Phenols. | |
|-----|-------------------------------|-------------|-----------------|
| | | Percentage. | Nature. |
| 1 | Medical carbolic, 20 % pure . | 30.50 | Pure phenol |
| 2 | " " 20 % " . | 17.00 | " " |
| 3 | Carbolic toilet, 10 % . | 3.60 | " " |
| 4 | " " 10 % . | 3.40 | " " |
| 5 | Domestic carbolic . | 4.80 | " " |
| 6 | " " . | 6.40 | Common carbolic |
| 7 | Carbolic soft, 10 % . | 9.90 | " " |
| 8 | " " 10 % . | 8.20 | " " |
| 9 | " " . | 0.16 | " " |
| 10 | Transparent carbolic . | 3.20 | Pure phenol |
| 11 | " coal-tar . | 1.50 | " " |
| 12 | No. 1 carbolic . | 2.50 | Common carbolic |
| 13 | " " . | 5.40 | " " |
| 14 | No. 2 " . | 4.40 | " " |
| 15 | " " . | 3.50 | " " |
| 16 | Carbolic . | 2.60 | Impure phenol |
| 17 | " . | 1.10 | Common carbolic |
| 18 | " . | 0.50 | Impure " |
| 19 | Disinfecting . | none | |
| 20 | Sanitary . | 0.75 | Impure phenol |

Free Alkali in Toilet Soaps.—In view of the objectionable effects produced by excess of alkalinity in toilet soap, and of the circumstance that the best British and foreign makes are found by analysis to contain only very small quantities of free alkali, expressed as anhydrous soda, Na_2O , not exceeding 0.20 to 0.25 per cent. by weight (the com-

bined alkali being usually 7 to 9 per cent., or some forty times as much), WRIGHT* classifies toilet soaps in three grades, from the point of view of the amount of free alkali present—viz.:

First Grade.—Soaps which are, if not neutral, at any rate so devoid of free alkali that the amount of total alkaline matter present, in forms other than actual soap, does not exceed $\frac{1}{40}$ th part (2.5 per 100) of the alkali combined as soap.

Second Grade.—Soaps in which the free alkali, although exceeding the above limit, does not exceed $\frac{3}{40}$ ths of the combined alkali (7.5 per 100).

Third Grade.—Soaps in which the free alkali exceeds the second limit.

Comparison of Soaps.†—GASSLER has compared two German resinous soaps with an English cold-water soap, and considers that the former are superior to the latter.

| | German Soaps. | | English Cold-water Soap. |
|-------------------|---------------|-------|--------------------------|
| | I. | II. | |
| Fatty acids . . . | 56.25 | 53.65 | 46.87 |
| Resin | 14.75 | 17.35 | 23.13 |
| Soda | 12.75 | 12.55 | 12.00 |
| Talc | — | — | 1.00 |
| Water | 16.25 | 16.45 | 18.00 |

The tables of analyses on p. 202 are of interest, as giving the comparative compositions of some colonial and English soaps.‡

Cost of Soap.—The question is sometimes asked, “Can the first cost of a soap be deduced from the figures obtained

* Cantor Lectures on “Toilet Soaps,” May 1885.

† “J. Soc. Chem. Ind.” 1882, p. 371.

‡ “Colonial and Indian Exhibition Reports: Oils and Fats,” by LEOPOLD FIELD, p. 278.

Analyses of Household Soaps.

| | Ivory Bar. | Cold Water. | Telephone. | Klensodor Electric. | Cook's "Prim-rose." | Paton & Charles' Cold-water. | Cook's Bleached Palm. | Good Kind of Imitation Castile. |
|--|------------|-------------|------------|---------------------|---------------------|------------------------------|-----------------------|---------------------------------|
| Fatty anhydrides + resin | 68.33 | 69.85 | 62.70 | 50.70 | 62.28 | 65.70 | 66.18 | 61.45 |
| Combined alkali (reckoned as Na_2O) | 7.72 | 8.00 | 7.92 | 6.88 | 7.12 | 9.28 | 7.83 | 8.46 |
| Excess of alkali (reckoned as Na_2CO_3) | 2.64 | 2.22 | 3.91 | 5.05 | 0.14 | 0.58 | 0.68 | 3.18 |
| Water, &c. | 21.31 | 19.93 | 25.47 | 37.37 | 30.46 | 24.44 | 25.31 | 26.91 |
| | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Resin by GLADNIG's process | 25.00 | 24.00 | — | — | 15.40 | — | — | — |
| Free alkali (reckoned as Na_2O) | 1.54 | 1.30 | 2.29 | 2.95 | 0.01 | 0.34 | 0.40 | 1.16 |
| *Molecular weight of fatty acids | 283 | 280 | 254 | 238 | 280 | 230 | 270 | 234 |

Analyses of Fancy Soaps.

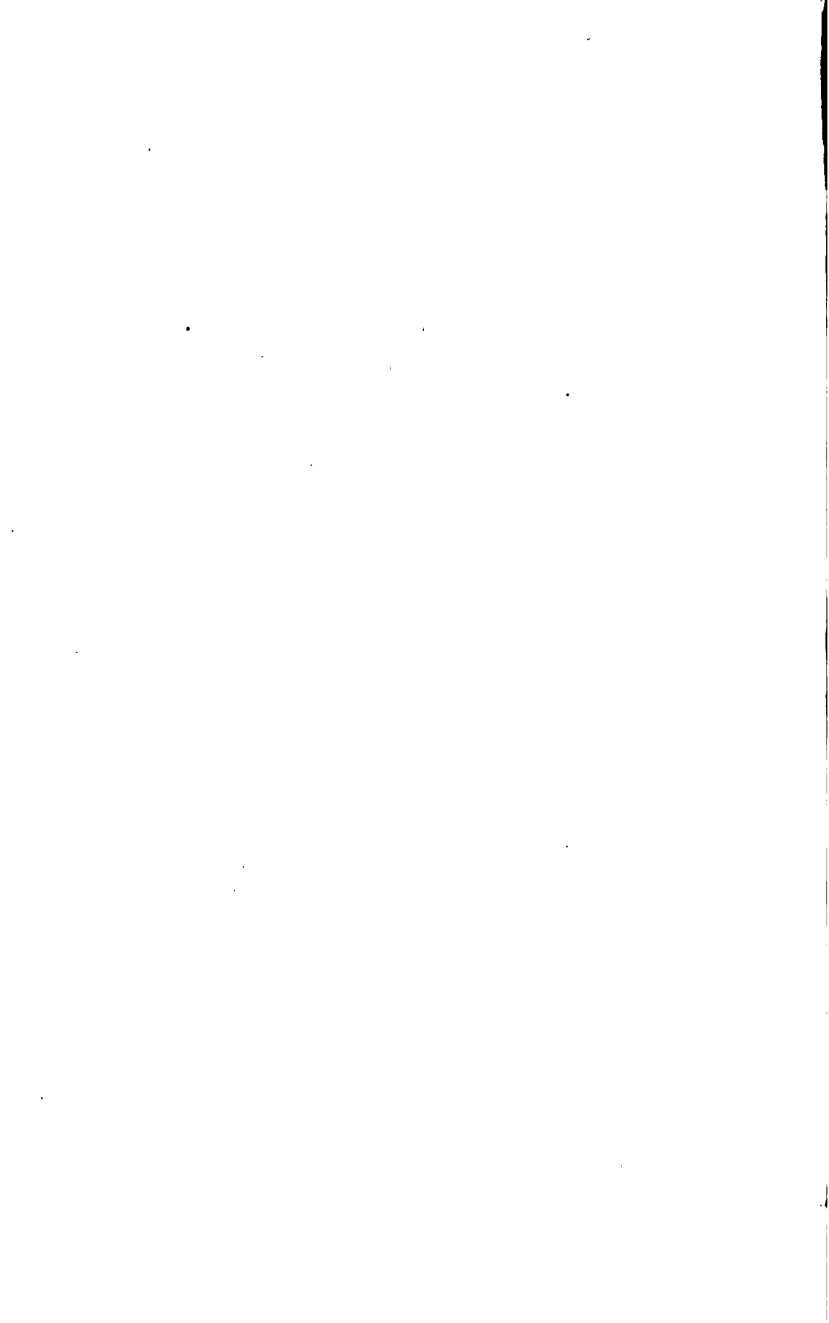
| | Barsalou. | | Bouquet. | Violeta. | Boehm's "Rose." | Wright's Coal-tar. |
|---|-----------|--------|----------|----------|-----------------|--------------------|
| | Mottled. | White. | | | | |
| Fatty anhydrides, &c. | 70.15 | 71.65 | 72.39 | 62.39 | 76.60 | 72.10 |
| Combined alkali (Na_2O) | 9.53 | 8.55 | 8.82 | 8.96 | 9.21 | 9.73 |
| Excess of alkali (Na_2CO_3) | 0.87 | 2.82 | 1.76 | 2.02 | 0.13 | 0.34 |
| Water, &c. | 19.45 | 16.98 | 17.03 | 26.93 | 14.06 | 17.83 |
| | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Free alkali (as Na_2O) | 0.51 | 1.65† | 1.03† | 1.18§ | 0.07 | 0.14 |
| *Molecular weight of fatty acids | 237 | 261 | 263 | 223 | 267 | 239 |

* These numbers are obtained by dividing the percentage of fatty acids (combined as soap) by the percentage of combined alkali (as Na_2O) and multiplying the quotient by 31 (the equivalent of Na_2O). A comparison of these molecular weights with the saponification equivalents given on pp. 57, 58, aided by the knowledge of the specific gravity of the fatty acids and their melting points, will afford considerable insight into the kind of fatty material used in the manufacture of the soap. The method of taking these specific gravities and melting points is described in "Oils and Varnishes," pp. 211-215.

† Too alkaline.

§ Bad soap; very alkaline; much cocoanut oil.

on analysis?" It is difficult always to do this with perfect accuracy. If, however, the percentages so obtained be divided by 5, the quotient will be in *cwts. per ton*, and a calculation as to cost may then be made at the current prices of materials, as in the example given on p. 106. It is necessary, however, that judgment be exercised in forming conclusions from the results of such a calculation.



PART II.—CANDLES.

CHAPTER I.

DEFINITION AND HISTORY.

Definition.—A candle may be defined as a cylinder of fat surrounding a fibrous thread, or wick ; “a contrivance in which, for the purposes of illumination, a wick of fibrous material is employed to effect the combustion of fatty bodies;”* “a wick surrounded by a coating of wax or tallow;”† “a cylindrical or slightly conical rod, formed of solid fat, enclosing a bundle of parallel or twisted fibres of cotton, called the wick, through which the melted fat is drawn up to the region of combustion.”‡

The chief point of difference between a candle and a lamp is that in the former the fuel is a solid, which is gradually liquefied in the required quantity by the heat of the flame, whilst in the latter the fuel is, at common temperatures, a fluid.

History.—The early history of candles is involved in

* “Jury Reports, Exhibition 1851,” p. 615.

† L. FIELD, Cantor Lectures, “Solid and Liquid Illuminating Agents,” 1883, p. 7.

‡ RICHARDSON and RONALD’S “Technology,” vol. i. pt. ii. p. 425.

some obscurity. The Hebrew word translated candle in the Old Testament probably means lamp. Possibly the torch (Lat. *tortium*, a twisted thing) may have been the earliest form of the candle. PLINY* mentions that the books found in the grave of NUMA were in a box bound round with *candles*. These candles were, it is thought, pitched rope. PLINY also states† that the pith of "brittle rushes," which grow in marshy districts, separated from the rind, was used for making *watch-candles* and funeral lights to burn by dead bodies whilst lying above ground. He does not, however, say anything as to the nature of the fat employed, so that it cannot be certainly inferred whether the reference is to candles or to a kind of lamp.

BECKMANN‡ has recorded that the Emperor Constantine, about the beginning of the fourth century, caused the city of Constantinople to be illuminated with lamps and *wax candles* on Christmas Eve.

APULEIUS distinguishes wax and tallow candles by the terms *cerei* and *sebaei*.

In the Saxon period we find that wax candles were not, as a rule, made by professional chandlers, because the well-known candles of King Alfred were manufactured by his *chaplains*, whom he commanded to supply wax in sufficient quantity, and to weigh it in such a manner, that, when there was so much of it in the scales as would equal the weight of seventy-two pence, six candles were to be made thereof, each of equal length, so that each candle might have twelve divisions marked across it. Six of these candles, lighted in succession, burned exactly twenty-four hours. §

* "Natural History," xiii. 13.

† *Ibid.* xvi. 37.

‡ "Hist. of Inven." BOHN's ed., ii. 174.

§ ASSER's "Annals," translated for BOHN's "Six Old English Chronicles," p. 84.

FOSBROOKE* states that in the Middle Ages wax candles were made of various sizes, some exceedingly small, and others weighing as much as 50 lb. He also states that they were made in moulds, and that the wicks were formed of twisted tow.

According to DUCANGE, persons who made and sold candles, or *candelarii*, were known in the middle of the thirteenth century.

In the fifteenth century "mould" candles were introduced by the Sieur DE BREZ.

GILBERT WHITE, writing in 1789, thus describes the method of making rush candles practised in Hampshire:—The proper species of rush for this purpose seems to be the *Juncus conglomeratus*, or common soft rush, which is to be found in most moist pastures, by the sides of streams, and under hedges. These rushes are in best condition in the height of summer; but may be gathered, so as to serve the purpose well, quite on to autumn. As soon as they are cut, they must be flung into water and kept there, for otherwise they will dry and shrink, and the peel will not run. At first a person would find it no easy matter to divest a rush of its peel or rind so as to leave one regular, narrow, even rib, from top to bottom, that may support the pith; but this, like other feats, soon becomes familiar, even to children. When the rushes are thus far prepared, they must lie out on the grass to be bleached and take the dew for some nights, and afterwards be dried in the sun. Some address is required in dipping them in the scalding fat, or grease, but this knack also is to be attained by practice. 1600 rushes, weighing 1 lb., are coated with 6 lb. of tallow, so that 228 lights weigh 1 lb. and cost a little over 5d.†

* "Encyclop. Antiq." p. 472.

† "Natural History of Selborne," p. 220.

In the year 1799 WILLIAM BOLTS took out a patent in England for improving the form, quality, and use of the candle, the specification of which probably contains the first account of an attempt to improve the quality of candles made from tallow and other animal fats, by subjecting the material to a considerable pressure during the act of cooling, and which is, in effect, the preparation of the so-called *stearin* from fats. He likewise describes a solid candle with a short wick, which is placed in a holder, and kept pressed on the end of the candle by a spring, or else the candle is placed in a tube and pressed against the wick by a spiral spring; as well as other contrivances, some of which have been revived and successfully carried out in our own days.*

In 1830 the number of candle-makers in Great Britain was 2695, who paid £500,048 14s. 1d. duty; since the repeal of the duty in that year no record has been kept of their number.

Very little improvement took place in the manufacture of candles till after the discovery by CHEVREUL of the true nature of fats (see p. 51). In 1825 GAY-LUSSAC and CHEVREUL took out a patent for making *stearic acid* candles—the badly combustible glycerin being removed, and the oleic acid being separated to be used in soap-making. But it was not till 1834 that they may be said to have succeeded in making their candles perfect.

The kernels of the candle-nut tree (*Aleurites moluccana*, Willd.), a native of the islands of the Pacific, are used in Fiji, the Hawaiian Islands, and Tahiti, when threaded on the mid-rib of a palm leaf, reed, or stick of wood, as a substitute for candles. In Tahiti and Fiji the tree is called *Futui*, and *Doodoe* is its title in Pitcairn's Island. In the

* "Jury Reports, Exhibition 1851," p. 617.

history of the "Mutiny of the *Bounty*" it is stated that the rooms in Pitcairn's Island were lighted up with torches made of *doodoe* nuts strung upon the fibres of the palm leaf. The nuts are also so strung and used by the Sans Blas Indians in Central America, and a child is in attendance to knock off each nut as it burns out. Each nut burns about ten minutes.* They yield a light which was considered good a century ago, but is now thought dull, smoky, and ill-smelling.

In Java the kernels are cleaned, crushed, and mixed with sufficient cotton or cocoa-nut fibre to give them the consistence of stiff suet. This paste is then rolled round a split reed or bamboo to form a kind of candle or torch. These burn more regularly than the contrivance just mentioned, but they consume more rapidly than tallow candles, and give out an unpleasant odour, so that they are used only by the poorer classes. This tree must not be confounded with the candle tree (*Parmentiera cerifera*) nor with the candle-berry tree (*Myrica cerifera*).†

* M. C. COOKE, "London Medical Record," 1860.

† "Chemist and Druggist," 1879, p. 149.

CHAPTER II.

MATERIALS AND THEIR PREPARATION.

Materials.

THE materials chiefly used in the manufacture of candles are the following:—

1. **Animal Fats.**—Tallow; lard; stearin.
2. **Vegetable Oils.**—Cocoa-nut oil; palm-oil and palm-kernel oil; piney oil, or tallow.
3. **Waxes.**
 - (a) *Animal.*—Bees'-wax; Pela, or Chinese wax; spermaceti.
 - (b) *Vegetable.*—Carnauba; Chinese vegetable tallow; Japan; myrtle; palm.
 - (c) *Mineral.*—Paraffin; ozokerit.
4. **Fatty Acids.**—Coco-stearic acid; palmitic acid; stearic acid.

1. **Animal Fats.**—TALLOW.—This has already been spoken of in treating of soap (p. 18), and various processes for its purification have been described (pp. 32, 39–43).

Tallow consists chiefly of—

Stearin—stearic glyceride, $C_3H_5(C_{18}H_{35}O_2)_3$;

Palmitin—palmitic glyceride, $C_3H_5(C_{16}H_{31}O_2)_3$; and

Olein—oleic glyceride, $C_3H_5(C_{18}H_{33}O_2)_3$

—the first predominating, but varying in proportion according to the species, age, food, &c., of the animal from which it is obtained. The greater the proportion of *stearin*, the harder will be the fat and the higher its melting point.

The two great and inherent disadvantages of tallow as a candle material are due to the presence in it of fluid *olein*, and to the *glycerin* in combination with stearic anhydride in the *stearin*. The former lowers the melting point of the fat, and produces a great tendency in the candle to *gutter*, while the *glycerin*, both of the *olein* and of the *stearin*, being with difficulty consumed, diminishes the intensity of the light, and at the same time causes an unpleasant odour, by giving off, as a product of combustion, the highly pungent *acrolein* (C_2H_3COH), which is very perceptible when a tallow candle is blown out.

The *setting point* of tallow is found for technical purposes as for paraffin (English method), p. 229.

LARD.—See p. 18. STEARIN.—See p. 231.

2. **Vegetable Oils or Fats.**—COCOA-NUT OIL (see p. 21).—This oil or fat is now more utilized for night-lights than for the manufacture of candles. Candles have been made from it, but not satisfactorily; the untreated fat has too low a melting point, and the presence of *glycerin* makes it as objectionable as tallow. The caproic and caprylic acids also give rise to unpleasant odours. The chief brands are Cochin and Ceylon, of which the former is the whiter and sweeter, and is therefore more suitable than the latter for night-lights.

PALM OIL (see p. 22).—Lagos oil is the brand commanding the highest price. Other brands are Brass, Bonny, Old and New Calabar, Whydah, Accra, &c.

PALM-KERNEL OIL (see p. 22).

PINEY OIL, OR TALLOW.—This is obtained by roasting, grinding, and boiling with water the seeds of *Vateria indica*, or piney tree. The oil rises to the surface and is skimmed off. When cold it is a solid fat, melting at about 95° to 97° F. (35 – 36° C.), sp. gr. 0.926. Colour, white or pale yellow, and odour somewhat fragrant.

4. **Waxes.**—The substances known as waxes are obtained partly from animal and partly from vegetable sources. The term wax was formerly confined to bees'-wax, but candles are now frequently called *wax* candles, though made from solid paraffin, or *paraffin wax*.

Waxes proper chiefly consist of members highest in the series of fatty acids, $C_nH_mO_2$, partly free and partly in combination with alcohol radicals. They differ from fats in being less readily saponified, and in yielding no glycerin when so treated. The soap formed is also very sparingly soluble in water.

The waxes are solid at common temperatures—melt below the temperature of boiling water—are sparingly soluble or insoluble in water—soluble in ether, chloroform, carbon disulphide, and in the volatile and fixed oils.

Animal Waxes.—**BEES'-WAX.**—This is obtained by melting the honeycomb in water after the honey has been removed, straining the liquid mass, re-melting the defecated portion, and then casting into cakes, or discs.

Pure bees'-wax has a pleasant odour, a pale yellowish-brown colour, and a specific gravity of 0.960 to 0.969. It is brittle at 32° F. (0° C.), softens and becomes plastic at 88° to 90° F. (31.1–32.2° C.), and melts at 145° to 155° F. (62.77–68.3° C.).

As met with in commerce, the wax varies in colour from very pale yellow, or almost white, to a dark mahogany shade. But, however different in colour, and from whatever country obtained, the chemical composition, according to HEHNER, who has very thoroughly investigated it,* does not vary to any great extent. The following are HEHNER's results from the analyses of eighteen samples of English wax :—

* "Analyst," 1883, p. 16.

| | | Average. |
|---|--------------------|----------|
| Free acid, calculated as cerotic, $C_{26}H_{53}CO.OH$ | } from 13 to 16 %. | = 14.40 |
| Saponifiable matter, calculated as myricin, $C_{16}H_{31}(C_{20}H_{41})O_2$ | | |
| Myricyl palmitate | | |
| | „ 86 to 89.6 %. | = 88.09 |
| | Total | 102.49 |

In all cases the sum of the cerotic acid *plus* myricin is higher than 100, the average being as given above. The tendency of these figures is to show that English bees'-wax consists almost completely of cerotic acid and myricin, but that it also contains a small quantity of a substance of a lower molecular weight, probably the *cerolein* of LEWY.

In the case of seventeen foreign samples the fluctuation of composition was found to be more considerable than the above, but this HEHNER considers to be due to a greater degree of sophistication.

Before wax is employed for the manufacture of candles it is necessary to bleach it. All waxes do not bleach with equal facility. According to BARCLAY, English, Hamburg, Odessa, Portuguese, Mogadore, Zanzibar, East and West Indian, and North American waxes bleach readily, while those from Cuba, Dantzic, Königsberg, Gambia, and Gaboon are only bleached with difficulty, and seldom acquire a good colour.

There are two methods of bleaching wax—(a) *Atmospheric*; (b) *Chemical*.

(a) *Atmospheric Bleaching*.—1°. The wax is cut up into small pieces and placed in a vat, into which steam is admitted through a perforated coil. A small quantity of very dilute sulphuric acid (in the proportion of 1 pint of strong acid for each ton of wax) is added, and the whole boiled and well agitated for some time. The impurities separate and subside. This operation is called "clearing down."

2°. The melted, bright wax is next caused to pass into a tank, the bottom of which is perforated with holes about

the size of an ordinary quill. Through these holes it trickles in thin streams on to a revolving cylinder, or drum, half of which is immersed in a cistern of cold water. The motion of the cylinder carries up a layer of water, on which the wax falls, and becomes divided into exceedingly thin ribbons. These ribbons, by the revolution of the cylinder, are carried under the water, and are removed by a rake as they rise to the surface.

3°. The ribbons are then spread evenly and thinly on long canvas sheets, and placed in the open air, so as to be exposed to the influence of the sun and air, for a period varying from about four to ten weeks, according to the weather. Frequent turning is required so as to expose every portion to the sunlight, and frequent sprinkling with water is also necessary. Once, or perhaps twice, during the period mentioned, the wax is re-melted, separated into threads again, and spread out as at first.

It has been observed that in rainy weather the wax gets a greyish tinge, which cannot afterwards be removed.

A wax that yields to the atmospheric process is termed *kind*, while one that is not so readily bleached is called *stubborn*.

(b) *Chemical Bleaching*.—Wax may be bleached by chlorine, or by bleaching powder, or by WATTS' chrome process. When chlorine is used, substitution products are formed; and when the wax is subsequently burnt, hydrochloric acid is given off. The greenish colour which remains after the chrome process may be removed by boiling the wax several times with solution of oxalic acid. When treated by these methods, wax becomes highly crystalline, and is unsuitable for candle-making. Hence these methods need not be here described.*

* For WATTS' chrome process, and other methods, see "Oils and Varnishes," pp. 192-200.

PE-LA, PIH-LA, OR CHINESE WAX.—This is produced upon the young branches of *Fraxinus chinensis*, or wax tree, by an insect (*Coccus pe-la*). On being scraped from the trees, the crude material is freed from impurities by spreading it on a strainer, covering a cylindrical vessel which is placed in a caldron of boiling water. The wax is received into the former vessel, and, on congealing, is ready for the market. It is perfectly white, translucent, and shining. It has a marked crystalline structure, and melts at about $82-83^{\circ}\text{C}$. (180°F). It is much harder than spermaceti, and not unctuous to the touch. Its sp. gr. is 0.809–0.811. It is tasteless and inodorous, and crumbles into a dry inadhesive powder between the teeth. It is soluble in essential oils and naphtha, insoluble in water, and scarcely affected by boiling alcohol, acids, or alkalies. Chemically it is cerylic cerotate ($\text{C}_{27}\text{H}_{55}\cdot\text{C}_{27}\text{H}_{53}\text{O}_2$). The quantity which finds its way to this country is now very small.

In China, candles are made of the substance itself, but it is more commonly mixed with softer fats, and used for coating more easily fusible material, thus preventing guttering. It is often coloured red with alkanet root, and sometimes green with verdigris.*

SPERMACETI.—This is the solid fat which is dissolved in sperm oil in the head-cavity of the sperm whale, or cachalot (*Physeter macrocephalus*), and which, after death, separates as a solid. The name appears to have been given under the erroneous belief that the substance was the *spawn* of the whale tribe (*sperma ceti*). The head-matter, as it is called, is not the only source of spermaceti, as the blubber or body-fat, after melting and cooling, also yields a deposit of it. The following is an outline of the method of separating the crystals of spermaceti from the oil:†—

* RONALDS and RICHARDSON, "Technology," vol. i. pt. ii. p. 464.

† "Jury Reports, Exhibition 1851," p. 626.

1. *Bagging*.—The oil is filtered through long cylinders of bagging, lined with linen, tied, at one end, to the nozzle of a feed-pipe communicating with a tank elevated about 6 feet, and, at the other end, tied up with string. The oil, pressed upon by the weight of its own column, readily passes through, while the bags retain the solid portion. The spermaceti, at this stage, is of a dingy-brownish colour, and is called *bagged sperm*.

2. *First Pressing*.—The bagged sperm is next placed in hempen sacks, and subjected to a pressure of about 80 tons in a hydraulic press, by which the greater portion of the adhering oil is removed.

3. *Second Pressing*.—The pressed sperm is now melted and crystallized by slow cooling, and, after being ground to powder, is folded up in square pieces of bagging and then subjected to the action of a much larger hydraulic press, capable of exerting a force of 600 tons. The oil which runs from this press contains a small quantity of spermaceti, and is therefore returned to the bags to be filtered.

4. *First Refining*.—The spermaceti is next melted in a large iron vessel, and boiled for some time with a solution of caustic soda or potash, which readily saponifies the sperm oil still adhering to the spermaceti, whilst it has scarcely any action on the spermaceti itself. By this means the sperm oil is removed in the form of soap.

5. *Hot-pressing*.—The purified spermaceti is next removed from the boiler, and run into flat tin-moulds to crystallize. It is then again ground to powder, placed in linen bags, interleaved with horse-hair mats and previously heated iron plates, and pressed in a *horizontal* hydraulic press, heated by steam.

6. *Second Refining*.—The hot-pressed spermaceti is now removed and boiled with a strong alkaline lye, the temperature reaching 235° F. (113° C.). By this final operation it

becomes as colourless as water, and has only to be cast into blocks for the convenience of storing.

Spermaceti, thus purified, consists mainly of cetylic palmitate ($C_{16}H_{33}.C_{16}H_{31}O_2$). It is white, scaly, brittle, neutral, inodorous, and nearly tasteless. Its sp. gr. is 0.943 at $15^{\circ} C.$, and it melts at about 110° to $120^{\circ} F.$ ($43.3-48.8^{\circ} C.$).

Vegetable Waxes.—CARNAUBA, OR STONE-WAX.—This occurs as a thin film on the leaves, stalks, and berries of the Carnauba palm (*Copernicia cerifera*), a native of Brazil. The leaves, &c., are collected and dried, and the wax can then be peeled or boiled off, melted in earthen pots, and turned out when cold. It is of a yellowish colour, and very hard and brittle. When bleached, it is quite white. Its sp. gr. is about 0.995–1.000 and its melting point $182-185^{\circ} F.$ ($83.3-84.9^{\circ} C.$).

Its chemical composition is uncertain. STÜRCKE* found it to contain myricyl alcohol ($C_{30}H_{62}O$), free and in combination as myricyl cerotate, to the extent of about 45 per cent.

It is used sometimes to harden candles, but only in very small quantity, as 2 per cent. of the wax would cause the candle to crack. *Heel-balls*, for rubbing on the heels of boots, &c., contain 50 to 60 per cent. of this wax mixed with a little blacking, rosin, and soft wax.

CHINESE VEGETABLE TALLOW.—This is found enveloping the kernels in the nuts of *Stillingia sebifera* (*Excoecaria sebifera*, Müll.). According to Dr. PORTER SMITH, the fat is obtained in China from the seeds in the following manner:—(1) The ripe nuts are bruised and the pericarp separated by sifting. (2) They are then steamed in wooden cylinders, with

* LIEBIG'S "Annalen," ccxxiii. 283–314; "Year Book of Pharmacy," 1885, p. 204.

numerous holes at the bottom, which fit upon kettles or boilers. The tallow is softened by this operation. (3) The mass is then gently beaten with stone mallets, to separate the tallow from the albumen of the seeds, and afterwards sifted through hot sieves. (4) To remove the still remaining brown testa of the seeds, the tallow is poured into a cylinder made up of straw rings put one on the top of the other, the whole placed in a rude press, and the tallow squeezed through in a pure state.

The product is a hard, white, tasteless, odourless solid. It chiefly consists of tri-palmitin. Its melting point is about 104° F.

JAPAN WAX.—Although called wax, this substance is, strictly speaking, a fat, as it consists of palmitin or glycerin palmitate. The chief sources from which it is commercially obtained are *Rhus succedanea* and *Rhus vernicifera*, which, according to Prof. J. REIN, of Marburg, were introduced into Japan probably from the Loochoo Islands.

According to A. MEYER,* the most usual plan for obtaining the wax is the following:—The previously well-dried fruits are ground either by mill-stones, or with wooden pestles in mortars, or by bamboo-flails. The shells and epidermis are separated by sifting and winnowing, and the mass is then heated in canvas bags over boiling water, in order to melt the fat, which is then pressed out. The crude tallow is now boiled with dilute lye, whereby it becomes granular and more readily bleached. The bleaching by sunlight and subsequent melting are repeated till the product is pure and white. 400 lb. of seeds will yield 100 lb. of wax. When freshly broken, the fractured surface of the imported article is almost white, or, sometimes, slightly yellow.

* "Year Book of Pharmacy," 1880, p. 220.

ish-green. Its odour is like that of tallow, and disagreeable. Its sp. gr. is 0.916 (MEYER), 0.99 (FIELD*), 0.984–0.993 at 15° C. (ALLEN). It melts at 52–53° C. (125.6–127.4° F.) (MEYER) when old, and about 42° C. (about 107.6° F.) when recently solidified. According to FIELD, its melting point is 48.89° C. (120° F.). According to other observers, its melting point varies from 120° to 130° F. As it contains glycerin, it gives off the smell of acrolein during combustion.

MYRTLE WAX (MYRICA WAX, MYRTLE TALLOW, OR BERRY WAX) is a solid fat of a pale-green colour obtained by boiling off the coating of the berries of *Myrica cerifera* in Louisiana, and of *Myrica cordifolia* at the Cape of Good Hope. Its sp. gr. is 1.005, and its melting point 47–49° C. (116.6–120.2° F.) (MOORE). It contains palmitic and myristic acids, with a little glycerin, but its exact composition appears not to have been yet ascertained. Candles made from this wax were exhibited at the Colonial and Indian Exhibition, 1886, by Messrs. Hall & Zinn, of the Cape of Good Hope. They burned with a smoky flame and with a strong odour of tallow, but without guttering.†

PALM WAX.—From the trunk of *Ceroxylon andicola*. It does not melt below the temperature of boiling water, according to some observers, but according to others the melting point varies from 161.6–186.8° F. (72–86° C.).

c. **Mineral Waxes.**—PARAFFIN is found native, or is obtained by the distillation of petroleum, bituminous shales, cannel coal, lignite, wood-tar, or peat.‡

Refining Paraffin.—There are several methods practised

* Cantor Lectures on "Solid and Liquid Illuminating Agents," 1883, p. 18.

† "Colonial and Indian Exhibition Reports, 1887," p. 276.

‡ For details of preparation, see "Oils and Varnishes," p. 153 *et seq.*

for effecting the purification of crude paraffin. In some of these naphtha is used, in others it is dispensed with.

The following process is a combination of the action of *naphtha* and *hot-pressing*, by which the quantity of naphtha, which has a powerful solvent action on paraffin wax, is much reduced:—

1. The crude solid is placed in a centrifugal machine, by which paraffin oil is expelled.

2. The residual mass is cast into cakes, placed in layers on cocoa-nut matting, on hollow iron plates containing water to regulate temperature, and submitted to hydraulic pressure. As much as possible is squeezed out in the cold, and then the temperature is raised gradually to from 35° to 40° C., by which means the paraffins of lower melting points are squeezed out, the object being to produce a solid with a high melting point, so as to make it approximate to the character of wax or spermaceti. This operation leaves the cakes of a dark-brown colour.

3. To further purify these cakes, they are melted, heated to 155° C., and 2 per cent. of sulphuric acid added, to remove any bodies of the C_nH_{2n} series, or olefines, still present.

4. The cakes are again melted with soda, cooled, and repressed. They are then well washed with hot water, cooled, mixed with cold colourless naphtha to assist filtration, and then filtered through animal charcoal to remove colouring matters.

5. The product is next placed in steam-jacketed wrought-iron cylinders, and superheated steam is passed through to remove naphtha. The residue is then pressed, and cast into cakes.

Another way of carrying out the naphtha process is to melt the scale with a certain proportion of naphtha. The mixture is then either allowed to cool in suitable vessels, or

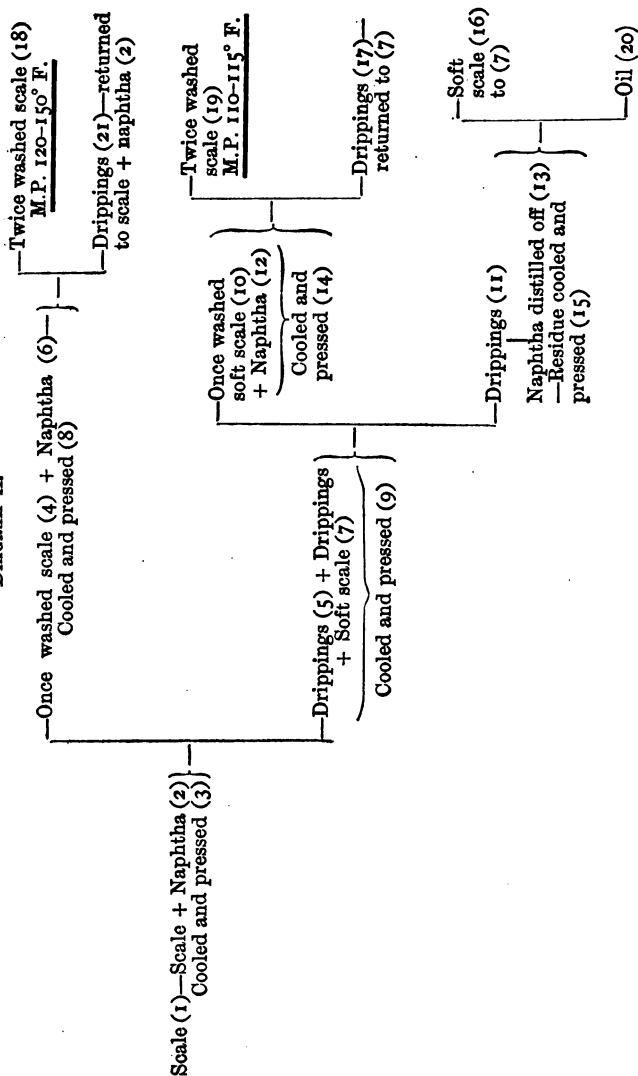
it may be cooled by artificial means. The cooled mixture is subjected to hydraulic pressure, when the objectionable portion is carried away by the naphtha. This operation is repeated two or three times, or until the desired degree of purity is obtained. Diagram A. (p. 222) will make the process clear.*

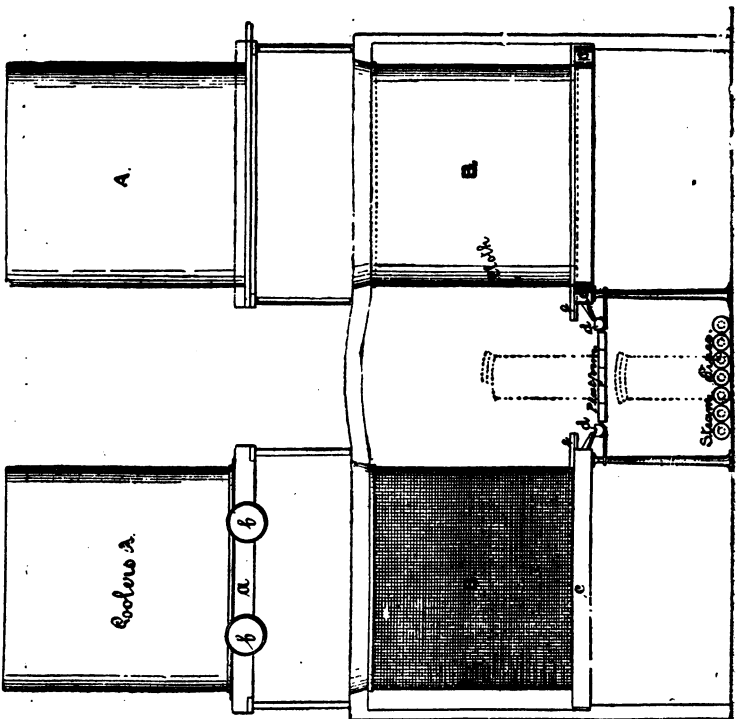
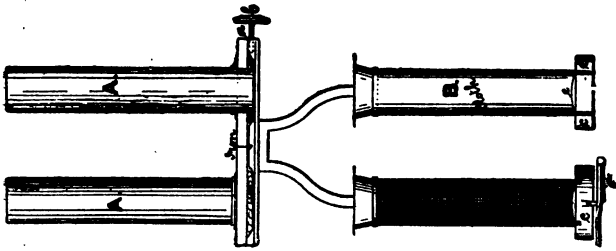
In some refineries† the following method, called the *sweating process*, is adopted:—The crude paraffin scale is melted and heated to a temperature of 170–180° F., after which it is allowed to repose until every trace of water and separable impurity has settled out, the presence of which would hinder crystallization. It is run into cooling pans, which hold from 1 to 2 gallons; these pans are generally furnished with overflows, and are arranged as shown at A (Fig. 39, p. 223). A stream of melted paraffin is directed into the top pans by the taps *b*, and is continued until the whole vertical series is full. They are then left to cool very slowly in order to promote crystallization. When cold, the solid cakes of paraffin are taken out of the pans and placed in the ovens, which are fitted with shelves, the latter having a slight inclination to the one corner, on which is laid a coarse mat of cocoa-nut fibre to prevent the paraffin from being in direct contact with the metal surface. The cakes are exposed to heat until the desired degree of purity and melting point is attained, the source of heat being a course of steam pipes laid on the floor of the ovens. The portion that has been fused out of the paraffin in the course of sweating is again treated in the same manner, only at a lower temperature suited to its mean melting point. The drainings from this latter cannot again be very profitably sweated, as they contain the whole of the oil originally present in the scale,

* R. TERVET, "Journ. Soc. Chem. Ind." 1887, p. 356.

† "Journ. Soc. Chem. Ind." 1887, p. 356.

DIAGRAM A.





and also the greater proportion of paraffins of low melting points. It is therefore cooled in a separate series of pans, and then hydraulic-pressed to get rid of the oil. The solid pressed paraffin obtained is either returned to the next make of crude scale, or it may be finished off separately as a low melting-point wax (mean melting point 102° F.). It is usual to allow a certain proportion of the paraffin of intermediate melting points to pass to this stage, in order to give solidity and maintain a suitable melting point for the finished product.

Diagram B. (p. 225) gives in outline the several stages of this process.*

An improvement in the above method, designed to economize labour, is described by R. TERVET.† By reference to Fig. 39, it will be seen that the coolers are set directly above the cells in which the sweating is conducted. The cooling and sweating cells may be made of any convenient size—they may be 3 feet broad by 6 feet high. The way in which the coolers are sealed at the bottom is made to depend upon the shape in which the alternate strips of soft wood and iron, or soft and hard wood, are placed, and which extend right across the lower openings. It is evident when pressure is applied by screws, or otherwise, to the side A, which forms the end of the system, the strips of soft wood, which go to cover the openings of the cells, will rise slightly, while the iron strips, which cover the blank spaces between, will correspondingly fall.

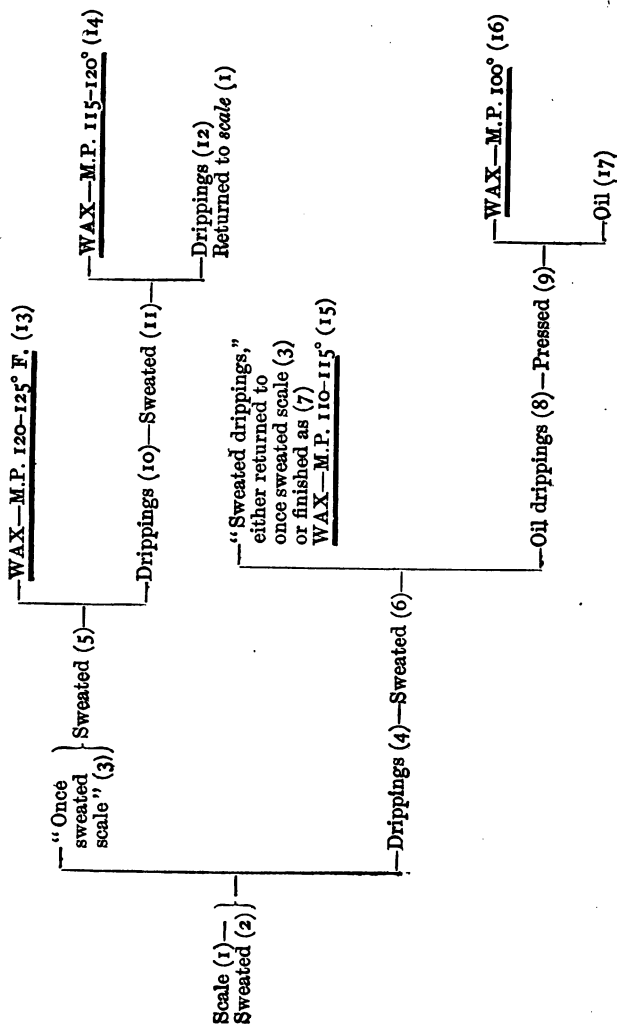
In practice this arrangement is found more than sufficient to seal the openings of the coolers.

To empty the coolers it is only necessary to relax the screws, *b*, and draw forward the strips of wood when the

* R. TERVET, "Journ. Soc. Chem. Ind." 1887, p. 357.

† *Ibid.*

DIAGRAM B.



cooled paraffin cakes are free to descend into the sweating cells, after which the strips are pushed into their place and the screws tightened, when the cells are again ready to be filled. In order to facilitate the dropping of the cakes from the coolers, a very slight taper is put upon them, which need not be more than $\frac{1}{16}$ th inch per 12 inches in height. The distance between the coolers and the sweating cells is less than the height of the cell.

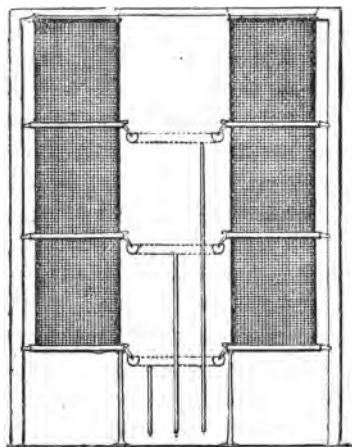
The sweating cell, *B*, is constructed of wire netting or perforated sheet metal, inside which is hung a coarse woven cloth of any description, but preferably of woollen plaiding. On the top of the cell there is set a light iron casting which forms the entrance, and assists in keeping it in position. At the bottom there is another casting, *c*, with an opening the same size as the cell, the edges of which are turned up all round, both inside and outside, forming a channel gutter, which is provided with an outlet leading to *d*. The cell is set within this channel, and, as the cloth extends to the bottom, the liquid portion fused out of the paraffin is conducted to the channel by the capillarity of the cloth. In order to prevent the solid cake from falling through the lower opening, there is provided a sliding door, *e*, of thin sheet-iron, the sides of which are turned down and overlap the inner sides, covering them like a lid. The passage for the edges of the door is therefore between the inner sides of the gutter and the cloth. This gives direction to the liquid portion, and effectually hinders any part of it from finding an outlet other than to the gutter.

In adapting this apparatus for a continuous or fractionating process, it is necessary to have two or more sweating cells in height, and a proper means of regulating the temperature. All the parts and arrangements remain the same as described, only the doors in the upper cells may, if thought proper, be dispensed with, as the paraffin, in its

plastic condition, moulds itself to the irregularities of the cell, and effectually stops any passage to the cell beneath.

Suppose such an arrangement be constructed as shown in Fig. 40, and that the temperature is under proper control. It is evident that the greater portion of the impurities will be drained away in the first or uppermost cell, and that the cake will have correspondingly diminished in bulk before passing to the second or middle cell, where

FIG. 40.



the soft, and intermediate soft, paraffin would be sweated out. Again, on passing to the third cell, the cake of paraffin will not be more than 65 per cent. of its original bulk, but containing all those hard intermediate fractions which correspond to the *once sweated scale* (No. 3) of Diagram B. (p. 225), and which, after further sweating, and when the proper melting point has been attained, may be discharged by withdrawing the bottom door. There is no danger of the partially sweated paraffin falling out, as its

descent is only gradual; indeed, one important feature of the arrangement—either as a simple or complex structure—is that the cake will not come out until it is perfectly sweated, which is only attained at a temperature which, if prolonged, would result in the complete fusion of the paraffin.

The advantages claimed by the author of this process are—(1) that the sweating, being obtained simultaneously from both sides of the paraffin, permits of the operation being carried on at a comparatively low temperature, and with greater rapidity. (2) The cakes can be made of greater thickness than by the usual method. (3) The process can be made continuous by duplicating, vertically, the cells in which the sweating is conducted. This is obtained by taking advantage of the gradual diminution in bulk which the paraffin undergoes in the course of sweating. Although this latter advantage effects no great economy, yet it makes the production of the full proportion of first-class wax obtainable from crude scale at one operation a possibility. (4) In working with an apparatus constructed of three cells, it can be charged and discharged every four hours, beginning with a scale of melting point $112-114^{\circ}$ F., and finishing with a wax melting at 126° F. As the drippings are separately fractionated out in three grades of purity, it facilitates their subsequent treatment to have them always of a uniform composition and melting point.

FORDRED* purifies crude paraffin by melting, leaving mechanical impurities to settle down, and then transferring to smaller vessels to cool. The cakes are next warmed till they become kneadable, and are then washed with a solution of 10 parts of soft soap in 90 parts of water, and heated to about 38° C. Colouring matters and any oils that

* "Monit. scien." [3], iii. 826.

may be present are transferred by this treatment to the soap water, and the solid paraffin comes out purified and bleached.

Pure Paraffin is a colourless, inodorous, tasteless solid. Its sp. gr. is from 0.870 to 0.909 at 15° C. It melts at 113–149° F. (45–65° C.). It becomes plastic much below its melting point—a disadvantage which is corrected when used for candles by admixture with bees'-wax or stearic acid. It is insoluble in water, and only slightly so in alcohol. Sulphuric and nitric acids and chlorine are without action upon it in the cold. Chlorine passed through melted paraffin slowly attacks it with evolution of hydrogen alone. This last reaction establishes its position among members of the marsh-gas family. It surpasses all other candle materials, even spermaceti, in illuminating power.

Methods of taking the Melting Point of Paraffin.—The so-called *melting point* (which is really the *setting point*) of paraffin is, in the case of the recognized American and English methods of making the test, the temperature at which the sample, after having been melted, and while in the process of cooling, *begins to solidify*.

The AMERICAN test is conducted by melting sufficient of the sample to three parts fill a hemispherical dish $3\frac{3}{4}$ inches in diameter. A thermometer with a round bulb is suspended in the fluid so that the bulb is only three-fourths immersed, and, the material being allowed to cool slowly, the temperature is noted at which the first indications of filming, extending from the sides of the vessel to the thermometer bulb, occur.

The ENGLISH test is performed by melting the sample in a test-tube about $\frac{3}{4}$ inch in diameter, and stirring it with a thermometer as it cools until a temperature is reached at which the crystallization of the material produces enough heat to arrest the cooling, and the mercury remains sta-

tionary for a short time. The results afforded by this test are usually $2\frac{1}{2}^{\circ}$ to 3° F. lower than those furnished by the American method.

The melting point is also sometimes determined by observing the temperature at which a minute quantity of the sample, previously fused into a capillary tube and allowed to set, becomes transparent when the tube is slowly warmed in a beaker of water.*

OZOKERIT (FOSSIL WAX, EARTH WAX).—This remarkable mineral, which has been utilized as a candle material by Messrs. Field, of Lambeth, is found in various localities in the Tertiary strata, mostly occurring in, or in close proximity to, the coal measures. The largest and purest deposits are found at Drohobycs and Boryslaw in Galicia, on the slopes of the Carpathians, in the island of Tcheleken in the Caspian Sea, and elsewhere, but it is by no means an abundant substance. It is obtained partly on the surface and partly by mining. A body very similar to ozokerit, called *Neft-gil*, is found on the island of Swätoi-Ostrow in the Caspian Sea.

Ozokerit is usually met with as a compact brown substance, occasionally yellow, sometimes black. It melts at about 60° C. (140° F.). It can be made to yield, by proper treatment, 80 to 90 per cent. of paraffin (FIELD).

Refining Ozokerit.—To obtain products from the mineral which can be used commercially, several processes are described by FIELD:†—

1. The most largely employed method is that of treating the crude wax with Nordhausen sulphuric acid, and heat-

* BOVERTON REDWOOD, "Jour. Soc. Arts," 1886, p. 896.

† Cantor Lectures, "Solid and Liquid Illuminating Agents," January, February, and March 1883, p. 45.

ing it till the acid has become decomposed. After proper decolorizing, the wax assumes a golden-yellow colour, and in appearance much resembles bees'-wax. It is called *cerasin* from this resemblance, and can be brought to almost a pure white. In this state it is not of much use for candle-making, as it has a strong and unconquerable tendency to smoke.

2. UJHELY dissolves the crude material in benzine, or some other spirit, in which condition it can be readily filtered through charcoal. The spirit is then distilled off in an air-tight apparatus, leaving the white paraffin behind.

3. At Messrs. Field's works, Lambeth, the crude ozokerit from Galicia is distilled in a current of superheated steam, and the following distillates are obtained :—

(1) A *gaseous hydrocarbon*, to the extent of about 5 per cent.

(2) A *volatile naphtha*, about 3 per cent.

(3) A product resembling *vaseline*, termed *ozokerine*, about 6 per cent.

(4) A *soft paraffin*, melting at $112.1-115^{\circ}$ F. ($44.5-46.1^{\circ}$ C.).

(5) A *white paraffin* (*ozokerit*), about 70 per cent. Melting point, 141.8° F. (61° C.).

(6) A *black residue*, melting at 170.6° F. (77° C.).

No use has yet been found for products (1) and (2).

(3) is used as a substitute for vaseline.

(4) is used for cheap candles.

(6) is used by electrical engineers as an insulating material.

STEARIN.—We have seen (p. 210) that tallow consists mainly of a mixture of the glycerides called *stearin* and *olein*, the former solid and the latter liquid at common temperatures. Common tallow melts at between 99° and

104° F. (37-40° C.), while the melting point of stearin is 144° F. (62° C.). Hence, by the removal of a considerable portion of the olein from the tallow, the fusing point of the latter is considerably raised, and its character as a candle material much improved. In the laboratory experiments of CHEVREUL, this separation was effected by means of solvents, but it was soon found that, by attending to the temperature of the fat, it might, for all practical purposes, be produced equally well by pressure. If tallow is melted, and allowed to cool as gradually as possible, with constant agitation, the mass becomes pasty, and by slow pressure in cloths the olein is squeezed out. By repeating the operation, the stearin is obtained gradually of greater purity.

Candles made of this pressed tallow fairly deserve to be called *stearin candles*, but stearin is now seldom thus prepared, and the so-called *stearin* candles consist really of more or less pure *stearic* or *palmitic acid*.

COCO-STEARIN.—A patent was taken out by SOAMES in 1829* for making *stearin* and *olein* by the following process:—

Cocoa-nut oil as imported is submitted to strong hydraulic pressure, having been made up into small packages 3 or 4 inches wide, 2 feet long, and 1 or 1½ inch thick. These packages are formed by first wrapping up the cocoa-nut oil in a strong linen cloth of close texture, and then in an external wrapper of strong sail-cloth. The packages are then placed side by side, in single rows, between the plates of the press, allowing a small space between the packages for the escape of the olein. The temperature at which the pressure is begun should be from about 50° to 55° F., or, in summer, as nearly as this can be obtained, and the packages to be

* No. 5842.

pressed should be kept for several hours previously at about the same temperature. When the packages will no longer yield their olein freely, the temperature is to be gradually raised, but it must at no time exceed 65° F., and the lower the temperature at which the separation can be effected the better will be the quality of the expressed oil.

When the packages have been sufficiently pressed—that is, when they will give out no more oil, or yield it only in drops at long intervals—the residuum in them is to be taken out and purified. This is done by melting it in a well-tinned copper vessel, which is fixed in an outer jacket, so as to leave a vacant space closed at the top between them, into which steam is admitted, and a moderate heat is kept up for a sufficient time to allow the impurities to subside. If a still higher degree of purity is required, it is necessary to pass it through filters of thick flannel lined with blotting-paper.

Thus cleansed, the coco-stearin is fit to be used in the ordinary process for making mould tallow candles.

The second product of this operation, or olein, is purified as follows:—It is mixed with 1 or 2 per cent. by weight, according to the degree of its apparent foulness, of the sulphuric acid of commerce, of about sp. gr. 1.80, diluted with six times its weight of water. The whole is then subjected to violent agitation by mechanical means, conveniently in a vessel constructed on the principle of a common barrel churn. When sufficiently agitated, it will have a dirty-whitish appearance, and is then drawn off into another vessel, in which it is allowed to settle, and any scum that afterwards rises is carefully removed. In a day or two the impurities will subside, and the clear oil is then filtered through thick woollen cloth, and will be suitable for burning in ordinary lamps, and other purposes.

4. Fatty Acids.

History.—The obstacles which stood in the way of the employment of tallow stearin might possibly have been removed, but the researches into the nature of the process of the saponification of fats, resulting in the separation of solid acids from the fatty bodies, directed the inquiry into another channel. When it was found that stearic acid fusing at 158° F. (70° C.) could be obtained from stearin fusing at 144° F. (62° C.), while the oleic acid remained as fluid as the olein from which it was derived, it became evident that, as the difference in the fusing points of the solid and liquid acids is so much greater than that between the stearin and the olein, their separation might be effected with less difficulty. Thus the transition from the tallow candle to the stearic candle was effected.

Though CHEVREUL's researches were published in 1823, it was not till two years afterwards that the idea of making candles from the isolated *fatty acids* was matured. In 1825 CHEVREUL and GAY-LUSSAC took out a patent in France for the manufacture of fatty acids and their application to the manufacture of candles. On the 9th of June 1825, GAY-LUSSAC, in the name of his agent, MOSES POOLE, also took out a patent in England. These patents are remarkable as specifying the distillation of fatty acids with the aid of steam, and the use of lime for the saponification of the fat. The distillation by steam was not practically applied until sixteen years after this date, and, instead of lime, the alkalis potash and soda were employed by the patentees for accomplishing the saponification, and hydrochloric acid was used to decompose the soap, producing alkaline salts which were never completely separable from the fatty acids.

It is well known that CHEVREUL and GAY-LUSSAC's patent was not commercially successful; the processes which they

employed resembled too closely laboratory experiments, and the industrial execution proved too costly.

Where these illustrious chemists failed, DE MILLY succeeded by introducing the cheaper material, *lime*, as the saponifying agent, and decomposing the lime soap formed by dilute sulphuric acid. The lime saponification process, on a commercial scale, dates from the year 1831.

Preparation.—We may consider the modes of preparing the fatty acids under the five following heads:—

- I. **Lime Saponification.**
- II. **Acidification.**
- III. **Dissociation by Heat.**
- IV. **"Autoclave" Process**—a combination of the first and third methods.
- V. **Bock's Process**—a modification of the second method.

I. Lime Saponification Process.—1. *Melting.*—Into a large wooden vat (under lime tub, A, Fig. 41, p. 238), containing a coil of steam-pipes pierced with small holes, a quantity of tallow, or of tallow and palm oil (about 3 parts of the former to 1 or $1\frac{1}{2}$ of the latter*), is emptied from the original casks, together with a quantity of water. The steam, when turned on, enters through the holes into the water, raises its temperature, and melts the fat.

2. *Saponification.*—As soon as the water has entered into ebullition, a quantity of slaked lime, equal to from 10 to 15 parts of dry quicklime for every 100 parts of fat, according to the nature of the fat used, is added.† It is important

* The acids from tallow alone are often not sufficiently crystalline to admit of thorough pressing. The products from a mixture of tallow and palm oil are superior to those from either fat alone.

† According to theory, 100 parts of fat would require only 8.7 parts of caustic lime, but the excess of lime renders saponification easier.

that the lime should be *caustic*, and as pure as possible. If not entirely caustic, a larger proportion will be necessary to thoroughly decompose the fat, and more acid will be afterwards required to remove it. If the impurities are considerable, they may become insoluble, and difficult to separate afterwards from the mixed acids. The vat having been tightly closed, the boiling is continued for about six hours, or until complete saponification is effected, which is ascertained by drawing out a small portion of the boiling mixture in a ladle. This, when cold, should appear perfectly smooth and solid, and should be very brittle, powdering finely in a mortar. During the boiling, the mixture is kept in constant agitation by means of a wooden shaft, furnished with horizontal arms, and worked by steam. At the end of the operation, the fatty acids will have combined with the lime to form a lime soap, called *rock*, which is, chemically, a mixture of stearate, palmitate, and oleate of lime. The whole is allowed to cool in the same vessel, and the liquid portion, containing 5 to 15 per cent. of glycerin, and termed *sweet-water*, is run off, and may then be evaporated down to about one-fifth of its bulk, yielding crude glycerin of about sp. gr. 1.26.

3. *Decomposition*.—The lime soap, or *rock*, is next dug out from the saponifying tank and removed to the lead-lined *separating* vat, *B*, which is also furnished with a perforated steam coil. Here the rock is *separated* into calcium sulphate and fatty acids. When the boiling point is reached, sulphuric acid (the ordinary brown acid of commerce), previously diluted, is added in the proportion of about 25 parts of the strong acid to every 100 parts of fat, and the boiling and agitation are continued. The acid may be added in successive portions till the workman sees, by the appearance of the mass, that a sufficient quantity has been introduced. It rapidly combines with the lime, forming insoluble calcium

sulphate, and liberating the oily acids, which float on the surface, and are called *yellow matter*. When partially cool, this yellow matter is either run off by cocks, placed at the proper level, or pumped into the washing vat, *C*.

4. *Washing*.—In this vat the acids are washed at a high temperature by means of steam and water, first mixed with very dilute sulphuric acid, and afterwards with water only.

5. *Caking*.—The washed fatty acids are then removed to flat tin pans, or *caking tins*, *D*, and left at a temperature of 68° to 86° F. (20° to 30° C.), for two or three days, or until they have solidified with a granular or crystalline structure.

6. *Cold-pressing*.—The cakes are next placed in bags of cocoa-nut matting or horsehair, and introduced into the hydraulic press, *E*, which at first is worked very gently. The bulk of the oleic acid is thus removed, and the cakes assume a light-yellow, instead of their original dark-brown, colour.

7. *First Refining*.—The *cold-pressed cakes*, as they are now called, which still contain about 10 per cent. of oil, are remelted by steam in a lead-lined wooden vat, *G*, with a little dilute sulphuric acid to remove the last traces of lime, oxide of iron, or other impurity. The melted material is then placed in flat tin trays, and again allowed to cool (at a slightly higher temperature than in the previous cooling) and solidify.

8. *Hot-pressing*.—The cakes, thus further purified, are now placed in stronger bags, conveniently made of goats' hair, introduced into the horizontal hot press, *H*, and subjected to great pressure at a high temperature for about two hours. By this operation the remainder of the oleic acid, holding a little of the solid acid in solution, is removed. The pressed cakes retain a small quantity of oleic acid at the edges; these are therefore scraped off, melted, and again pressed.

9. *Second Refining.*—The refined cakes are now placed in the melting vat, *I*, and heated by steam, a little wax being sometimes added at this stage to destroy the crystalline

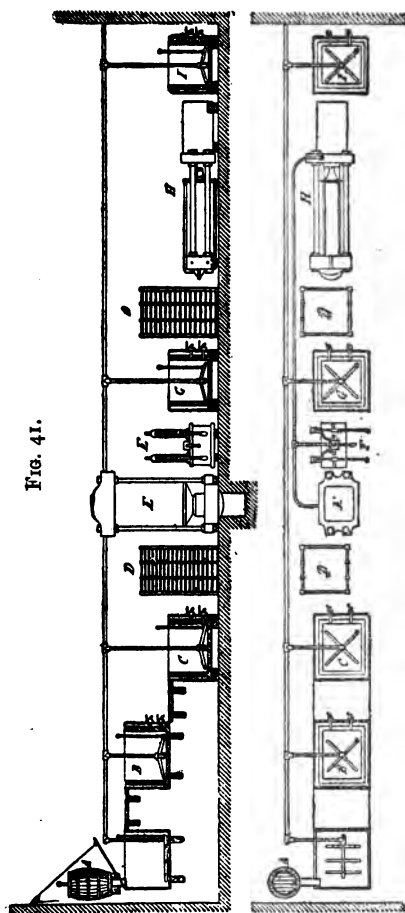


FIG. 41.

Apparatus for the preparation of stearin (stearic acid) by the lime saponification process.

A, Lime tub, from which lime is emitted. Under *A*, Saponifying tank, lead-lined, with steam pipes for boiling fat and lime. *B*, Separating vat, lead-lined, in which rock is boiled with sulphuric acid. *C*, Washing vat. *D*, Racks with caking tins. *E*, Cold press. *F*, Hydraulic-press pumps for working *E* or *H*. *G*, Melting vat. *H*, Hot press. *I*, Melting vat for refined stearin previous to casting into blocks.

texture of the stearic acid. The material is afterwards cast into blocks.

The final product is a mixture of impure stearic and palmitic acids, having a melting point of 132–135° F.

The lime process admits of the use of very impure fatty materials.

MODIFICATION OF THE LIME PROCESS BY MOINIER AND BOUTIGNY.*—2 tons of tallow are introduced with 900 gallons of water into a rectangular vat of about 270 cubic feet capacity.

1. *Melting*.—The tallow is melted by means of steam admitted through a pipe coiled round the bottom of the vat, and the whole kept at the boiling point for an hour, during which a current of sulphurous acid is forced in.

2. *Saponification*.—At the end of this period 6 cwt. of lime, made into a milk with 350 gallons of water, are added. The mixture soon acquires some consistence, and becomes frothy and very viscid. The whole is now agitated in order to regulate the ebullition and prevent the sudden swelling up of the soapy materials. The pasty appearance of the lime soap succeeds, and it then agglomerates into small nodular masses. The admission of sulphurous acid is now stopped, but the injection of the steam is continued until the small masses become hard and homogeneous. The whole period occupies eight hours, but the admission of the sulphurous acid is discontinued at the end of about three hours. The water containing the glycerin is run off from below by a tube to a large underground cistern.

To prepare the sulphurous acid, sulphuric acid and pieces of wood are introduced into retorts, which are heated by fire. The sulphurous acid which passes off is conveyed by leaden pipes to the vessels containing the tallow, where the

* RONALDS and RICHARDSON, "Technology," vol. i. pt. ii. p. 437.

saponification is effected under the joint influence of the acid and steam.

3. *Decomposition.*—The lime soap formed is moistened with 12 cwt. of sulphuric acid at 152° F., diluted with 50 gallons of water. The whole is thoroughly agitated and the steam cautiously admitted, so as not to dilute the acid too much until the decomposition is general at all points. This occupies about three hours, and in two or three hours more the calcium sulphate has collected at the bottom, while the fatty acids float on the surface of the solution of bisulphate of lime.

4. *Washing.*—Washing with steam and water is afterwards necessary to remove the adhering portions of calcium sulphate, &c., and, after settling for four hours, the fatty acids are forced through a fixed siphon into a vat, where they are again washed with water. They are then a third time washed with water, and siphoned at last into a trough lined with lead, on the bottom of which are placed leaden gutters pierced below by long pegs of wood.

5. *First, or Cold, Pressing.*—The cakes of fatty acids are inclosed in bags of flannel, and pressed in the cold in a hydraulic press. The oleic acid, squeezed out, is conveyed into a washing cistern. It is important to allow the fatty acids to cool slowly, so as to prevent a too confused crystallization, and to facilitate the expulsion of the oleic acid.

6. *Second, or Hot, Pressing.*—The cakes are now placed between horsehair sacks, and submitted to a second pressure at a high temperature. The whole is covered with oil-skin, and the temperature raised to 158.5° F. (70° C.) when the pressure is applied. The heat slowly falls to 113° F. (45° C.), and ultimately to $95-86^{\circ}$ F. ($35-30^{\circ}$ C.). This second pressing occupies about an hour. The oleic acid obtained contains large quantities of stearic and palmitic acids.

7. *Sorting*.—The cakes of the stearic acid are sorted according to colour and translucency.

8. *Refining*.—20 cwt. are introduced into a vat, constructed of wood lined with sheet-iron. The materials are boiled by steam admitted through a leaden pipe. Water acidulated with sulphuric acid is first employed, and afterwards water alone. When the materials are boiling, the white of twenty-two eggs is introduced, and the albumen is intimately mixed by the violent ebullition. As soon as the albumen is coagulated, the mass is allowed to cool, but is constantly agitated so as to prevent the formation of crystals.

MOINIER and BOUTIGNY considered that the use of sulphurous acid increased the yield of acid by about 4 per cent., the calcium sulphite formed, when treated with sulphuric acid, yielding sulphurous acid, which destroyed the nitrous acid contained in the sulphuric acid employed, which, otherwise, acted upon the fatty acids, and lessened their amount.

II. Acidification and Distillation Process.—*History*.—It was known to ACHARD in the year 1777 that neutral fats are decomposed by concentrated sulphuric acid in a manner similar to the decomposition effected by caustic alkalies. This fact was again brought forward in 1821 by CAVENTON, and in 1824 by CHEVREUL, but was not scientifically investigated till 1836 by FRÉMY. Both sulphuric acid and the alkalies decompose the fats, but, while the alkalies combine with the fatty acids and liberate the glycerin, the sulphuric acid, it was thought, combined with both, producing from the acids of the fat, sulpho-stearic, sulpho-palmitic, and sulph-oleic acids, and from the glycerin, sulpho-glyceric acid.*

* It will be seen on p. 250 that Dr. Bock does not agree with this view of the reaction.



GEORGE GWYNNE, in March 1840, appears to have described for the first time a method of obtaining fatty acids by the treatment of neutral fats with sulphuric acid and subsequent distillation of the resulting products. The proposal was to distil *in vacuo* by means of an apparatus similar to that used in sugar-refining, but the working was not found practicable owing to the difficulty of maintaining a good vacuum on the large scale.

In November 1840, GEORGE CLARK took out a patent for utilizing this property of sulphuric acid in decomposing fats, but without subsequent distillation. This also was found unworkable, owing to the great cost of purifying the fat after decomposition.

In August 1841, DUBRUNFAUT obtained a patent in England, and, about the same time, another in France, for the purification of fatty bodies and their distillation. The chief object of this patent was the purification of the commoner oils by heating them to a high temperature and then passing steam through them. In this way their disagreeable odours were to be removed. But the distillation of fatty bodies was also claimed.

In 1842, Price & Co., under the name of WILLIAM COLEY JONES, patented the process of distillation of acids from cocoa-nut oil alone, and also after saponification with lime. The candles made from the first product were objectionable on account of the unpleasant vapours evolved, while the candles made of the product of the distillation of the cocoa-nut lime soap, though satisfactory, were too costly.

On December 8, 1842, a patent was obtained by WILLIAM C. JONES and GEORGE WILSON for decomposing fats with sulphuric acid, aided by heat, and distilling the fat, thus decomposed, by means of steam. This is *the first successful application of the combined processes* of acidification and steam-distillation.

A patent, dated December 28, 1843, by GWYNNE and WILSON, describes a method of reducing the quantity of sulphuric acid employed for decomposing the fats to from 10 lb. to 6 lb. for every cwt. of fat. This saving was effected by heating the fat to 350° F. (177° C.). Another improvement was the heating of the steam in a series of pipes after it had left the boiler, instead of depending on the temperature of the fat to effect it.

By a subsequent patent, dated October 30, 1844, GWYNNE and WILSON proposed to use a jet of superheated steam to heat the fats previous to sulphuric saponification.

The following are the details of the process as now ordinarily practised:—

1. *Melting*.—The fat to be operated upon is melted from the casks by means of a steam-jet inserted in the bung-hole, and is then run into the underground tank, *A*, Fig. 42 (p. 245).

2. *Boiling*.—The lead-lined tank, *B*, is one of a series into which, after settling in the tank *A* for some hours to separate the condensed water and grosser impurities of the fat, the melted fat is raised by means of the force-pump, *C*. In these vats, which are fitted with steam coils, the material is boiled.

3. *Acidifying*.—The fat is next pumped into the vessel *D*, called the *acidifier*. This is constructed of stout copper, and supported either on wrought-iron girders or brickwork. It has the following fittings—viz., A valve with pipe for the admission of superheated steam; a copper pipe, fitted with a water shower-pipe, *d*, for condensing the generated vapours; a thermometer for the guidance of the operator; and a gun-metal cover at the lower side, for cleaning out, to which is affixed a cock by means of which the acidified materials are drawn off. After its introduction into this vessel, the fat is heated by the admission of superheated

steam at 350° F. (176° C.)* from the superheater, *F* (the design of Mr. EDWARD FIELD, C.E.), and then sulphuric acid, in the proportion of about 1 to $1\frac{1}{2}$ cwt. per ton of fat, is run in from the acid tank, *E*, above.† The fat is decomposed and becomes much blackened, the glycerin being converted into sulpho-glyceric acid, with evolution of sulphurous acid, and at the same time any foreign organic matter in the fat is carbonized, with evolution also of sulphurous fumes. The neutral fat is converted into a mixture of sulpho-fatty acids and sulpho-glyceric acid. The whole operation may take from fifteen to twenty hours, and when the acidification is complete the contents of the vessel are allowed to rest for from four to six hours.

4. *Washing*.—The materials are next discharged into a series of lead-lined washing vats, *G G*, previously filled to about one-third with water, containing a little sulphuric acid. The vats are furnished with copper steam coils, and the contents are boiled with free steam for two hours, and then left to settle for about twenty-four hours.

5. *Distillation*.—The fatty acids are then drawn off from the vats *G G* into the tank *A*, from which they are pumped through the tap *c* into the lead-lined charge tank, *H*, above the still. Inside this tank is a steam coil, which is charged with steam at the time the acids are admitted, in order to keep them liquid. From this tank the material is run into the still, *I*, of which the body is made of iron and the dome of copper. The distillation requires several precautions;

* This is the temperature employed at Price's works, Battersea. At the works at Gentilly, near Paris, the heat is seldom higher than from 110° to 115° F.

† The proportion of sulphuric acid depends upon the nature of the fatty materials employed. Kitchen-stuff, slaughter-house fat, and the like require about 12 per cent. of their weight; palm oil, from 5 to 9 per cent., according to quality.

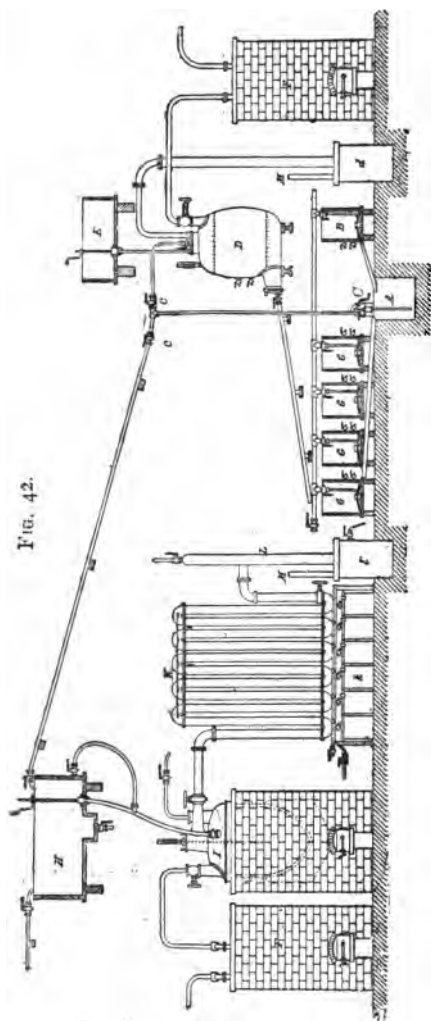


Fig. 42.

Apparatus for the acidification and distillation of stearin.

with an open fire the fatty acids are apt to be converted into oil, tar, and a carbonaceous residue, if the heat is too high. Air should be also completely excluded from the apparatus. The contents of the still are heated by the fire underneath to about 240° F. (116° C.) and then low-pressure superheated steam, at about 560° F. (293.3° C.), is admitted by a pipe from the superheater (shown on the left of the still in the illustration). The process of distillation then begins. The current of steam carries with it the vapour of the fatty acids, and thus facilitates the process. The mixed vapours pass to a series of vertical refrigerating pipes, *K*. These are of copper, connected at top and bottom by gun-metal bends, mounted on iron frames, and set over the series of iron tanks *k*, containing copper cooling coils, through which cold water can be passed, and also furnished with steam pipes. *L* is the essence tank, fitted with a safety condenser, or shower pipe, which prevents the possibility of any vapour passing away uncondensed. *M* is a pipe for conveying gas to be burnt in the flue.

The fatty acids as they run from the still are, to a great extent, available for candle-making *without pressing*, but other portions are subjected to pressing, sometimes both cold and hot, and often to a second distillation.

Out of every 100 lb. of tallow subjected to this process it is stated by W. LANT CARPENTER* that about 78 to 80 lb. of crude stearic acid are produced, of which 60 lb., or three-fourths, are ready for making *stearin* candles without further pressing. The remaining one-fourth, after being pressed and re-distilled, yields about 15 lb. more stearic acid and 5 lb. of oleic acid.

The residue is a sort of pitch, and is transferred, before it solidifies, to a vessel of iron, where it is submitted to a much

* SPON'S "Encyclopædia," p. 582.

higher temperature and a jet of steam more strongly heated. An additional quantity of fatty acids is thus obtained, of inferior quality, but applicable to the preparation of *composite* candles. The final residue is used for many purposes in the same way as ordinary pitch.

The following is a brief description of Fig. 42 (p. 245):—

A is the melting tank. *B* is one of a series of lead-lined boiling tanks. *C* is the force-pump. *D* is the “acidifier.” *E* is the acid tank. *F* is the superheater. *G G G G* are the washing vats. *H* is the charge tank. *I* is the still. *K* is the refrigerator. *k*, one of the series of iron tanks containing the copper cooling coils. *L*, the essence tank. *M*, a pipe for conveying gas to the flue.

III. **Dissociation by Heat.**—This may be effected either by the *high-pressure process*, or by superheated steam at ordinary pressures. The first was patented in 1854 by TILGHMANN, and its object is the separation of fats into acids and glycerin by heating with water only, under pressure, by which, at the same time, the substances are, to a certain extent, bleached. The method consisted, briefly, in pumping the mixture of fat and water through a coil heated to above 800° F., and at a pressure of about 2000 lb. to the inch. This operation was attended with considerable risk.

The second method was suggested to WILSON and PAYNE by the above, and was patented by them in the same year.* It is conducted as follows:—The fatty matter is heated in a still to about 550–600° F. (290–315° C.). Superheated steam, at a temperature of 600° F., is injected in such a way that it rises up through the molten fat in numerous streams. Saponification is thus effected, and the liberated fatty acids and glycerin are volatilized, and carried over

* No. 1624—1854.

in an atmosphere of steam to the condensing arrangement.

If the temperature is too high (above 600° F.), there is a great liability (diminished, however, by a very plentiful supply of steam) that the fatty acids and glycerin will be further decomposed—gaseous hydrocarbons, acrolein, and tarry matters being produced. On the other hand, if the heat is insufficient, either the separation of the glycerin is imperfect, or proceeds too slowly.

When the refrigerating arrangement consists of a series of chambers, each provided with a cock to draw off the distillates, and each more and more distant from the still, the compartments nearest to the still are found to condense little but fatty acids, being for the most part free from water and glycerin, which chiefly accumulate in the more distant and cooler condensers. In all the receivers the fat acids quickly separate from any aqueous solution of glycerin present, when allowed to cool for a little time. The last of the condensing chambers is open to the air, as no pressure is necessary in this apparatus. By simply evaporating off the water, very pure glycerin is obtainable.

IV. The "Autoclave" Process.—The large amount of lime required in carrying out the lime saponification is attended with the disadvantage that the great quantity of sulphuric acid necessary for the decomposition of the resulting *rock* injuriously darkens the fatty acids produced. By a combination of the lime saponification and TILGHMANN'S high-pressure processes, DE MILLY, in 1856, found that the proportion of lime could be reduced to 2 or 3 per cent., while a less pressure also was sufficient to effect the decomposition. This is called the *autoclave** process. The fat is

* From αὐτός, self, and κλείς (Lat. *clavis*), a key = that which shuts itself. It is a Papin's digester with a steam-tight lid fixed perpendicularly, and is preferably furnished with a safety valve.

put into a strong boiler provided with a stirrer, and mixed with 3 per cent. of slaked lime. Superheated steam is passed in till the pressure equals 160 to 180 lb. on the inch. After some three hours at this pressure, the separation is complete, and when the exit pipe is opened the fatty acids are forced out. A very small amount of sulphuric acid is afterwards needed to free them from lime. In twenty-four hours three operations of 2 or 3 tons each may be completed. The subsequent treatment for the crystallization of the fatty acids, cold- and hot-pressing, &c., are the same as in the other methods.

The fatty materials submitted to the autoclave process should be of good quality.

V. Bock's Process.—In 1871 Prof. Bock, of Copenhagen, pointed out that the neutral fats are composed of a congeries of little globules enclosed in envelopes, probably albuminous. To the presence of these in the fat he attributed the difficulty of eliminating the fatty acids by means either of sulphuric acid, except in excess, or of alkali, except under great pressure, conceiving that both these agents, as ordinarily employed, are to a great extent expended in rupturing and destroying the albuminous envelopes.*

CARPENTER† gives the following synopsis of Dr. Bock's process, extracted from "*Dingler's Polytech. Journ.*" May 1873:—

"By the lime saponification plan, the albumen contained in the fat is dissolved, lime soap is formed, and the extraction of the glycerin is rendered possible. By acidification, the whole process is effected at once. Conducted properly, the fat, washed out with water, always remains as a neutral fat, and, by the use of concentrated sulphuric acid, not a

* COOLEY'S "*Encyclopædia*," ii. 1557.

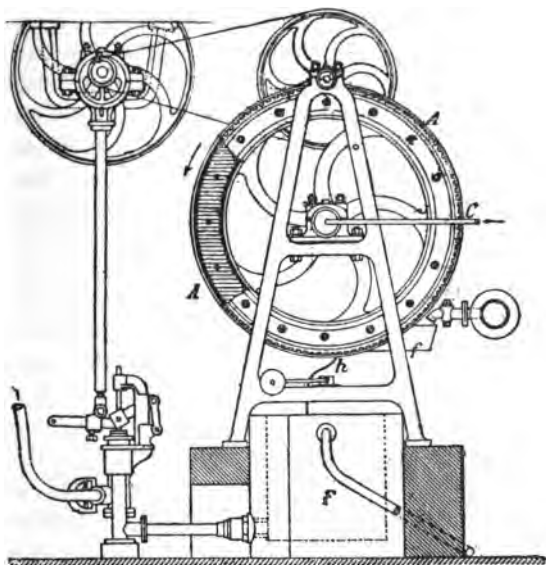
† SPON'S "*Encyclopædia*," p. 583.

trace of glycerin is left. Acidification, rationally conducted, is only a preliminary operation, intended to break up, corrode, or carbonize the albuminiferous matters. But the operation was long based on the erroneous belief that a double acid, sulpho-stearic, was formed. With due care, only the envelopes of the cells are blackened, and these are soluble neither in fat nor in fatty acids. The production of a real black solution is only an evidence that a certain part of the fat has been charred, which should be avoided under all circumstances. There is no doubt that the operation has generally been carried to excess in the matters of duration, height of temperature, or strength of acid. By proper acidification, the neutral fat is only unclothed, as it were, and freed from the cells, or, at any rate, the latter are so ruptured as to allow of the easy exit of the fat. This latter is then in a condition to be decomposed, an operation accomplished in a much shorter time by the chemical equivalent of acid—4 to 4.5 per cent.—and the necessary water. After letting out the glycerin waters, the fatty acids appear more or less black. They may now be distilled. Their melting point varies from 120° to 134° F. (49° to 57° C.).

“The real value of the new method consists in dispensing with this distillation. The object of this operation is the removal of the black colour, or rather of the black-coloured matters, by superheated steam. These black matters are the partially carbonized albumen cells, which swim about in the fatty acids, because the specific gravity of the two bodies is about the same. The difficulty is overcome by oxidizing the mass, by which the specific gravity of the cells is raised from 0.9 to 1.3. They are thus precipitated, and the fatty matters can be washed off. The subsequent cold- and hot-pressing are the same as with ordinary methods.”

Dr. BOCK's process, according to CARPENTER, consists of five stages:—(1) *Acidification*, to remove the cellular tissue of the fat. (2) *Decomposition*, by acidulated water, into dark fatty acids and glycerin. (3) *Oxidation*, to increase the specific gravity of the dark membranous matters, so that they may separate from the fatty acids. (4) *Repeated washing with water*. (5) *Pressing*, both cold and hot.

FIG. 43.



The following advantages are claimed for this process:—

1. *Freedom from danger of explosion*, as the steam is only used in open tanks.
2. *Economy*, from simplicity of plant and reduction of labour, the acidification, oxidation, and decomposition being all conducted, in rapid succession, in the same wooden tank.

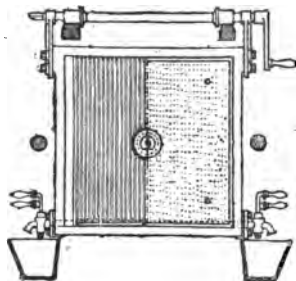
3. *Superiority of product*, the stearic acid being of great hardness, and melting at from 136° to 140° F. (58° to 60° C.).

4. *Increased product*, the stearic acid amounting to from 55 to 60 per cent. of the tallow employed.

5. The oleic acid is more suitable than that obtained by any other process for conversion into palmitic acid by RADISSON'S method, yielding a greater percentage of palmitic acid.

Separation of Stearic and Oleic Acids.*—In the ordinary method of separation from the mixture of fatty acids which is obtained by saponification of tallow or palm oil by means of lime, the solid stearic acid (so-called "stearin") is removed by passing through a filter-press at the common temperature. Under these conditions a considerable quantity of the stearic acid remains dissolved in the liquid oleic acid. By moderate cooling a further quantity of stearic acid can be obtained without solidification of the oleic acid. For this purpose a revolving drum, *A*, is employed (Fig. 43, p. 251), containing cold water, supplied by a cooling machine through the tube *C*, and carried off by another tube. The drum dips

FIG. 44.



into the trough *f*, containing the liquid fatty acids, which are carried round in a thin layer upon the surface. During the revolution the oil solidifies, and is scraped off by the scraper, *h*, into the reservoir, *F*, from which it is pumped through a Farinaux filter-press (Figs. 44 and 45). An increased

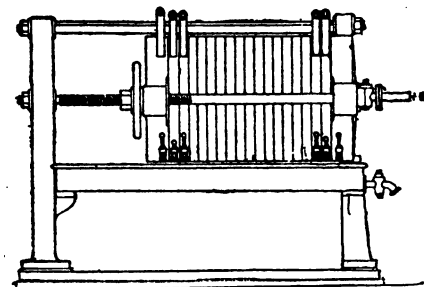
yield of 4 per cent. on the raw material is obtained, and the

* "Dingl. Polyt. J." 263, pp. 48, 49; "J. Chem. Ind." 1887, p. 372.

oleic acid has a higher value on account of its greater clearness.

Wicks.—The preparation of the wick is a very important branch of the candle manufacture. The wicks of ordinary tallow candles are made of the rovings of Turkey skeincotton, lightly twisted, the threads known in the trade as Nos. 16 * to 20 being employed. Twisted wicks are now only used for tallow† and for wax candles. The plaited or *braided* wick was introduced by CAMBACÈRES so as to do away with the necessity of snuffing. The effect of plaiting is to cause the wick to bend over during the combustion of

FIG. 45.



the candle, so that its end falls outside the flame where it is exposed to the air, and its complete combustion is thus insured. This bending over is caused either by twisting the wick with one strand shorter than the rest, which, being slightly stretched during the moulding, contracts again and bends the wick when the fat melts, or by plaiting the cotton into a *flat* wick, which naturally takes the required

* That is, 16 or 20 *hanks* of which weigh 1 lb.

† Plaited wicks are unsuitable for tallow candles because, owing to the ready fusibility of the fat, the bending over to one side would cause guttering.

curve. In 1830, DE MILLY found that boracic and phosphoric acids obviated snuffing by the formation of a bead at the end of the wick, which, by its weight, turned the end out of the flame.

Wicks should be of uniform thickness throughout, and quite free from knots and loose threads, as the presence of any of these tends to produce excrescences and *guttering*. The finer the thread of which the wick is composed, *cæteris paribus*, the more complete will be the combustion of the fatty materials.

Size of Wicks.—The size of the wick requires to be adjusted according to the diameter of the candle and the fusibility of the material (*i.e.*, there must be a sufficient number of capillary threads to carry up the melted material from the *cup* of the candle). If the wick is too large in proportion to the diameter, no cup can be formed, and *guttering* ensues; if too small, the unmelted substance forming the rim of the cup does not melt regularly with the descent of the flame, and forms little pillars round it, which are objectionable, because they not only cast a shadow, but by-and-by melt, fall into the reservoir of melted fat, and cause an overflow.

*Index to Thickness of Wicks.**

| For Tallow candles—8 to the lb.—wick (No. 16 yarn) contains 42 threads | | | | | | | |
|--|---|---|---|---|---|---|-------|
| " | " | 7 | " | " | " | " | 45 " |
| " | " | 6 | " | " | " | " | 50 " |
| " | " | 5 | " | " | " | " | 55 " |
| " | " | 4 | " | " | " | " | 60 " |
| Stearic | " | 8 | " | " | " | " | 63 " |
| " | " | 6 | " | " | " | " | 87 " |
| " | " | 5 | " | " | " | " | 96 " |
| " | " | 4 | " | " | " | " | 108 " |

Pickling.—To prevent too rapid combustion and smouldering of the wick when extinguished, wicks are dipped in various pickling solutions, such as boracic acid, 1 kilo. in

* SPON'S "Workshop Receipts," 1885, p. 355.

50 litres of water, or 5 to 8 grams boracic acid to 1 litre of water, with the addition of a little sulphuric acid (PAYEN); a solution of sal ammoniac marking 2° or 3° B. (recommended by Dr. BOLLEY), of ammonium phosphate (frequently used in Austria), or of bismuth nitrate (PALMER's patent). A solution of $2\frac{4}{10}$ oz. of boracic acid in 10 lb. (1 gallon) of water, with $\frac{1}{2}$ oz. of strong alcohol and a few drops of sulphuric acid, is also said to form a good pickle.

FIELD* treats wicks by steeping in a solution of phosphoric acid, or ammonium phosphate, or ammonium phosphate and borax, or ammonium phosphate and boracic acid.

The plaited wicks are kept for about three hours in the pickle, and are then either wrung out, or placed in a centrifugal machine, to get rid of the greater portion of the water. After this they are completely dried in a jacketed tinned-iron box, heated by steam.

* English patent No. 2061—1879.

CHAPTER III.

MANUFACTURE.

ORDINARY candles are made either by dipping or moulding. Wax candles are made chiefly by basting or pouring.

Dipping.

The commoner tallow candles are made by this process.

The purified melted tallow is placed in a trough 3 feet long, made of stout boards, lined with lead, sufficiently deep for the reception of the largest-sized candles, and furnished, on the side at which the workman stands, with a wiping board projecting upwards and outwards along the whole upper edge of the vessel. On this board the ends of the candles are, after each immersion, tapped, so that the superfluous material may be detached.

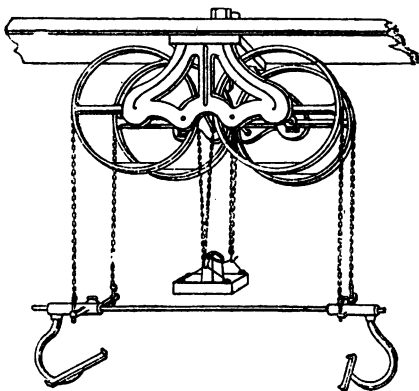
Another vessel is generally placed beside this trough, from which the melted fat is obtainable as required. In it the tallow is kept properly fluid by means of a steam- or hot-water jacket.

The operation is thus performed:—16 or 18 twisted wicks, according to the weight of candles desired, are looped, side by side, and as nearly as possible equidistant from each other, on a wooden, or thin iron, rod (*broach*, or *baguette*). Six or eight rods, or more, carrying the wicks, are then placed upon a frame, hung above the trough, and capable

of being raised or lowered at will. There are various dipping machines used by chandlers for this purpose, one of which, made by Merryweather & Sons, London, is illustrated by Fig. 46.

The advantage of this arrangement is that a perfectly

FIG. 46.



Dipping machine.

horizontal position is always secured, even under unequal pressure at either end, and candles of uniform length are more easily produced than with the ordinary machines.

The tallow should be hotter for the first than for the subsequent dippings, because hot tallow penetrates more readily into the interstices of the wick.

When the dry wicks have been saturated, they are withdrawn, care being taken to separate the ends of any that may be adhering to each other, and placed on the dripping frame, or *port*, below which is a tray to receive droppings.

A fresh batch of wicks is then treated in the same way. For the second and following dippings the fat is at a lower temperature, about 100° to 110° F., with a tendency to

solidify at the sides of the vessel. After each immersion the candles are allowed to cool sufficiently to retain a fresh coating of tallow at the next dipping. The dippings are continued till the candles have acquired the thickness and weight desired. Greater care is required for the final dippings to insure symmetry of form; if the lower ends of the candles are too thick, they are kept for a little in the molten tallow, so that the excess may be melted off and the temperature of the bath may be somewhat raised to produce a more even finish. The lower ends may finally be either cut away, or removed by placing the candles for a moment on a copper plate or sheet-iron tray heated by steam, and provided with a spout to carry away the melted portions.

A different method of dipping is practised at Messrs. Price's works, Battersea. Instead of dipping the wicks, they dip a series of steel skewers into the melted candle material, and, after the candles have been formed and cooled, these are removed, and the wicks, specially prepared and cut to the required length, are inserted. This method entirely prevents the waste of wick by the old method, and the saving thus effected is said to cover the whole cost of the candle-maker's labour.

Moulding.

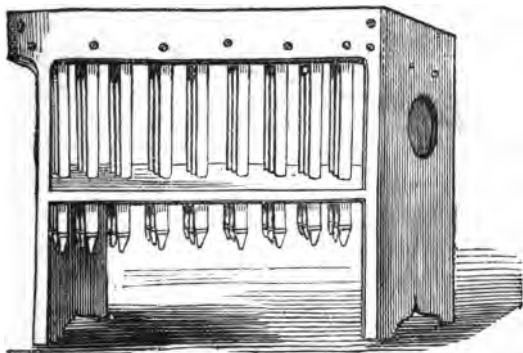
This operation is performed on the small scale by hand-frames, and in large works by some of the various moulding machines.

Hand-frames.—Fig. 47 exhibits the form of the hand-frames. They are made in all sizes, and suitable for all materials and shapes. They are convenient for small manufacturers, as an assortment of all sizes is less costly than one moulding machine. They are now, however, comparatively little used.

Moulding Machines.—Candle machines, or continuous

wick machines, manufactured by Biertumpfel & Son, Albany Street, London, N.W., and by E. Cowles, Novelty Works, Hounslow, are shown in Figs. 48, 49, and 52. They are modifications of the machines introduced into this country from America about 1849.

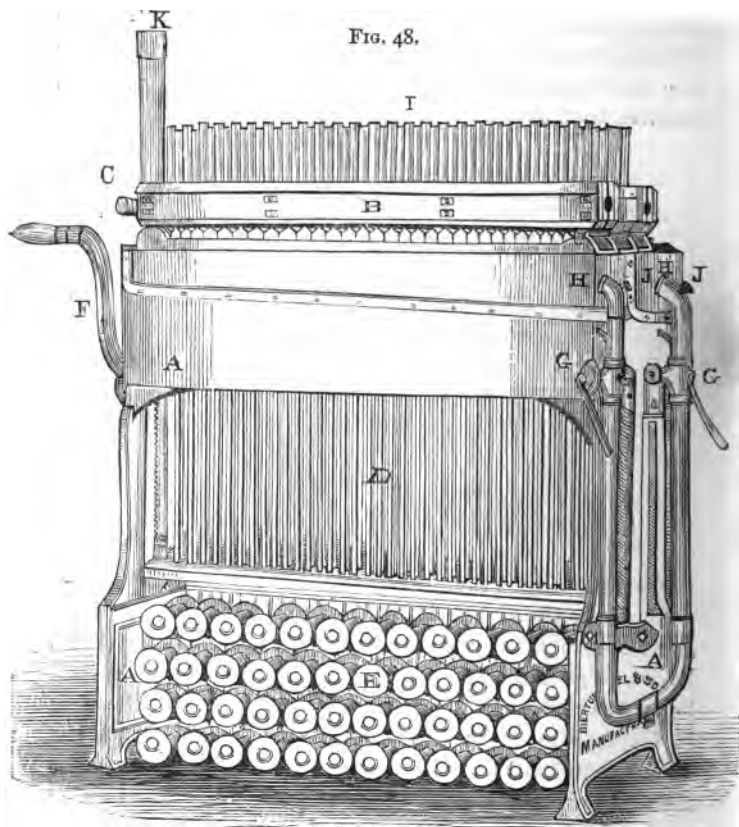
FIG. 47.



The following is the method of using these machines:—

1°. Raise the tip moulds to the top of the main moulds.

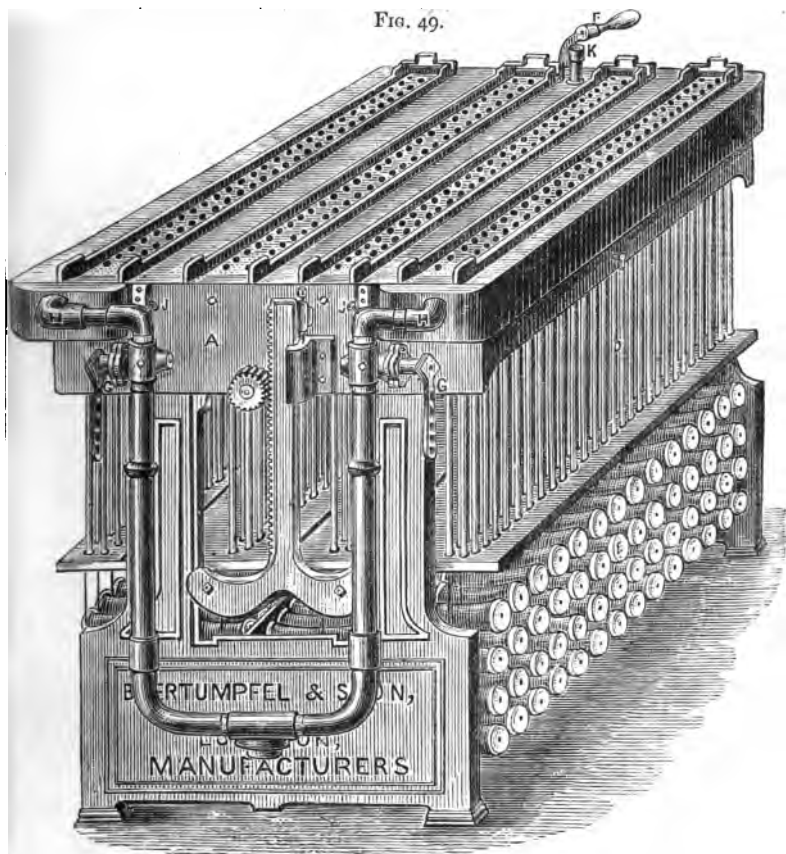
2°. Insert a very fine wire doubled, and of sufficient length to go through the tip mould and piston, and extend below the piston about 6 inches; insert the end of the wick in the loop made by the doubled wire, and draw up the wick through the tip mould, and secure it in any convenient manner for the first pouring; then lower the pistons as far as they will go, pour in the material by means of the *jack* (Fig. 51), and when cold shave off the butts with the *scoop* (Fig. 50); then place the racks B in a vertical position, with the tip bars thrown out; the crank, F, is then turned, and the candles ejected into the racks; the racks are then closed by turning the handle of C, and the tip of each candle is held precisely over the centre of its mould; now, the piston-



A is the main body of the stand. B, Movable racks with tip bars. C, Handle of the eccentric wedge. D, Pistons, having the tip moulds at the upper ends. E, Spools, with pins on which they revolve. F, Crank for raising the pistons. G, Handle of cock for emptying water box. H, Overflow pipe. I, Newly made candles. J, Clearing pin. K, Pipe for admission of hot or cold water.

block with pistons is let down, and the wicks are held by the candles above and the spools below; passing through

FIG. 49.

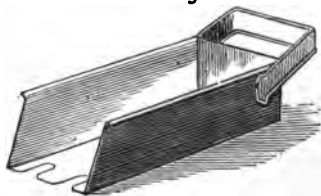


the pistons, and through a small aperture in the centre of the tip mould, they are all strained exactly in the centre of the moulds, and all is ready for the melted material again, and, when this is cold, the wicks are severed below the tip

bars, and the racks with the candles are then removed to any desirable place.

The machine represented by Fig. 46 is for making a

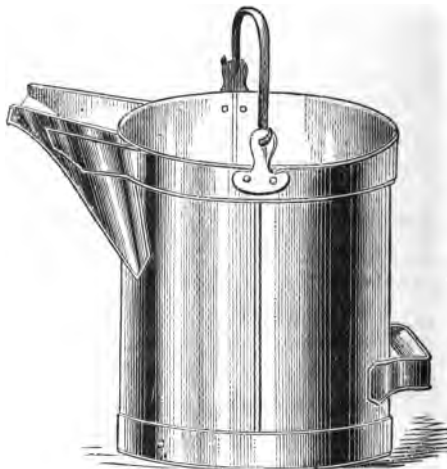
FIG. 50.



Scoop.

large number of small-sized candles, from 24 to 100 to the lb. It contains four trays of moulds for 224 candles,

FIG. 51.

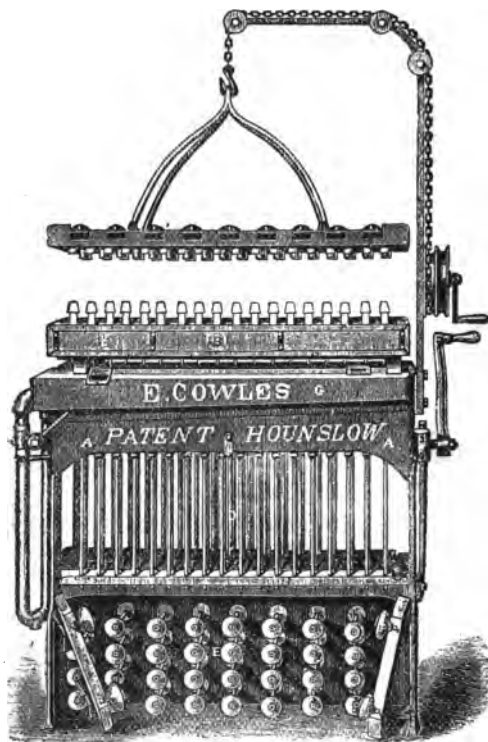


Filling can.

and may be arranged to produce at each operation four distinct sizes—say, 72, 60, 36, and 24 to the lb.

A great improvement in the candle was made in 1861,* when Mr. J. LYON FIELD patented the conical *butt*, by which a candle can be adapted to any candlestick, without paper

FIG. 52.



or scraping. This invention required special machinery for effecting its object, as the tapering butt is larger at the

* FIELD, Cantor Lectures on "Solid and Liquid Illuminating Agents," 1883, p. 48.

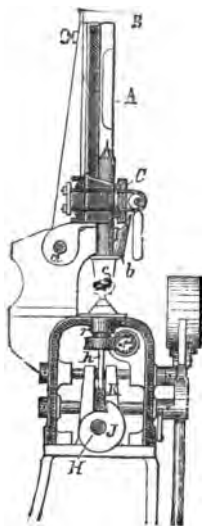
point of junction with the candle than the diameter of the latter, and could not, therefore, be extracted from the ordinary mould. Fig. 52 exemplifies how this difficulty is overcome. The moulds for the butts are cast in a separate frame, which is removed, when the candles are finished, by a chain and pulley, and the candles are then pushed out of the stem moulds in the ordinary manner.

Machine for Cutting the Conical Ends of Candles.*

—The conical end may also be made by the *cutting machine* shown in Fig. 53.

The plate *A*, which is capable of being turned round the

FIG. 53.



axle *a*, is furnished with grooves for holding the candles. When the plate is in an inclined position, the candles are put in the grooves, in which they are kept by the guard *B*, which can be adjusted to suit the length of the candles. On moving *A* into a vertical position, the candles slide downwards, the guard *b* keeping them from falling out, and at the same time forcing them to glide into the conical cutters, *c*. By pressing the board *C* against *A*, the candles are kept in position. The cutters, *c*, consist of conical bushes, on the inner wall of which several knives are fixed; they are attached to the square rods, *h*, which loosely move up and down in the collars, *r*. The cutters, *c*, are set in motion by the screw *G*; at the

same time the shaft, *H*, turns, and by the thumb, *J*, lifts the bearing, *K*, of the square shafts of the cutters, thus

* German patent 19,656, January 10, 1882, Motard & Co., Berlin; "J. Soc. Chem. Ind." 1882, p. 509.

causing the latter gradually to cut the ends of the candles. After one turn of the shaft, *H*, the bearing, *K*, goes down again, and restores the cutters to their original position. At this point the machine stops automatically.

Among the advantages of the moulding machines may be mentioned the following:—1. Rapidity of the process and beauty of finish. 2. Candles can be made as well in summer as in winter. 3. They can be arranged to turn out candles of different diameters and lengths in one machine. 4. By simply raising the driving plate, the length of the candles may be shortened at will.

Moulding Tallow Candles.—The moulds are generally made of pewter, carefully polished inside. The wick is inserted, after saturation with melted fat, through the opening at the smaller end, where it serves as a stopper. It is fastened at the upper orifice either to the movable top, or by means of a peg put through the looped end of the wick, and resting upon the end of the mould, while the wick is pulled tight from below. The melted fat is poured in, generally by a small can, or *jack*, Fig. 50, and it is essential that the tallow should completely fill the mould, which is of course maintained in an upright position. The candle must remain entire on cooling, without any cracks, and should readily be removable from the mould. These results can only be attained when the fat at the sides cools more rapidly than that in the interior, and a rapid cooling is always necessary to prevent contraction of the candle. Hence, cool weather is the most suitable for the operation. The proper consistence of the melted tallow to be used is known by the appearance of a scum on the surface, which in hot weather forms between 111° and 119° F. (44° and 48° C.), in mild weather at 108° F. (42° C.), and in cold weather at about 104° F. (40° C.). If the tallow is too hot when poured in, the candles are apt to *stick*, and are difficult

to draw ; if too cold, the candles are not uniform in appearance, but become granular-looking. The candles are ready to be taken out of the moulds on the day after casting, and then only require cutting and trimming at the base.

Moulding "Stearin" Candles.—The blocks of the stearic acid are melted, and, to *break the grain* or prevent crystallization, there is added 3 to 5 per cent. of wax, or 10 to 20 per cent. of paraffin, the whole is kept well stirred till the solidifying point is nearly reached, and then poured into the moulds, previously heated to about 120° F. to 125° F. It may be noted as a rule, when fatty acids are the material to be moulded, that the moulds should be heated to a temperature about 10° F. *under* the solidifying point of the material used, and the fat should be cooled down as near to its *setting* point as possible without the production of any actually solid portions.

By alternately admitting hot and cold water to the trough, a polished appearance may be communicated to the candles, but the method of doing this can only be acquired by actual experience. The fusing point of stearic candles is 131-132° F., and the produce of various makers in different countries is remarkably uniform in this respect.

Moulding "Sperm" Candles.—The moulding of sperm candles can be done in almost any of the ordinary machines. The spermaceti is heated to about the boiling point of water, run into heated moulds, and, to maintain transparency, is cooled as rapidly as possible.

To destroy its highly crystalline structure, spermaceti is usually mixed with 3 per cent. of wax. Sometimes it is tinted with gamboge, and denominated *transparent wax*.

Sperm candles, when properly made, are remarkable for the regularity of their flame, a result of the uniformity of the constitution of the material. Hence the choice of the sperm candle, burning 120 grains per hour, as the *standard*

for photometric purposes. On account of their high fusing point, spermaceti candles are very suitable for use in hot climates.

Moulding Paraffin Candles.—The same moulds may be used as for stearic and spermaceti candles. The principal difference in the operation is as regards the regulation of the heat. The moulds are heated to about 150.8° F. (66° C.), or a little *above* the melting point of the paraffin, and, when filled, they are left at rest for a few moments, and then *suddenly* cooled by cold water. This is intended to prevent crystallization, and consequent opaqueness.

The tendency of the paraffin candle to soften and bend at temperatures below its melting point is met by the addition of 5 to 15 per cent. of stearic acid.

FIELD and HUMFREY have patented* the following method of procedure:—The paraffin, having been melted at about 140° F., is run into moulds heated to the same temperature, or rather higher. After standing for a few minutes to allow bubbles to escape, the moulds are surrounded by cold water. This sudden cooling of the paraffin prevents the formation of crystals, and candles nearly transparent, and which draw freely, are thus obtained.

For paraffins of good quality, a wick of ordinary plaited cotton can be used, and, by dipping it in a weak solution of boracic acid, the ash of the wick will be fluxed, and the candles will burn with a bright and clear end.

Moulding Composite Candles.—J. P. WILSON patented the *composite* candle in 1840. The material was a mixture of coco-stearin and stearic acid. This candle is somewhat greasy, but is comparatively cheap, and gives a good light.

Another method is to melt together, over a water bath, 100 parts of stearic acid and 10 to 11 parts of bleached

* Patent No. 454, February 22, 1856.

bees'-wax, but, to insure success, the mixture must remain over the bath from twenty to thirty minutes without being stirred. At the end of that time the fire is extinguished, and the mixture allowed to cool until a slight pellicle is formed on the surface, when it is cast direct into the moulds, previously heated to about the same temperature.*

Cutting and Polishing.—The candles taken from the moulds have the ends cut by a circular saw, and have the length adjusted. The machinery allows them afterwards to fall upon an endless woollen cloth belt, supported by rollers, which carries them under other similarly covered cylinders, revolving in the opposite direction, by which means they receive a polish. Some of the higher class candles are hand-polished by rubbing with a woollen cloth moistened with ammoniated alcohol.

Night-lights.—These have taken the place generally of the old rushlight. Formerly they were called *mortars*.† As intensity of light is not required, a very thin wick is used, with a disproportionate thickness of fatty matter, so that a very deep and full reservoir is formed, containing an excess of melted fat, which is prevented from flowing over by the case of cardboard or wood shaving, or by a small glass vessel.

They were first made of wax or spermaceti, or a mixture of wax and spermaceti, but now generally from stearin, and coco-stearin, or from cocoa-nut oil and palmitic acid, in varying proportions.

The wick is fastened to a little square of tin-foil—the *sustainer*—and secured in the centre of the little case by a drop of wax. The cases, placed in rows, are filled by pouring the melted material into each from a jack.

* SPON'S "Workshop Receipts," 1875, p. 358.

† *Mors* (Lat.), death—from their use in death-chambers.

In another kind of night-light made of harder material, largely consisting of palmitic acid, the case is dispensed with, and, during the burning, the light is placed in a small glass. This description is made by running the melted fat into a special moulding frame. When cold, the night-lights are turned out ready punctured for the wick, which is afterwards inserted by hand.

Wax Candles.

The wicks for wax candles are made of *twisted* unbleached Turkey cotton. Plaited wicks are not so suitable, as the plaiting, by retarding the capillary action, necessitates the employment of a larger wick, which is apt to curl round in the flame and obscure the light.

Wax is not well adapted for moulding, on account of its tendency to adhere to the mould, and its great contraction on cooling.

The process of making wax candles is analogous to that of dipping, but, instead of dipping the wicks into the material, the melted wax is *poured* upon the wicks.

The wicks, having been warmed in a stove, are suspended on a hoop of wood or metal, which hangs over the cauldron of melted wax. The operator causes the hoop to revolve, and, taking a ladleful of the fluid material, pours it over each wick in succession, taking the precaution to keep turning the wick quickly on its axis by the fingers at the same time, so that the wax may not accumulate more on one side of the wick than the other. After three or four revolutions of the hoop, or when the candles are coated to about one-third of their proper size, the first hoop is laid aside, and, while its load is cooling, another hoop is taken in hand. The candles on the first hoop are afterwards again *basted* till they are half the required size. They are next, while still warm, rolled, upon a marble slab sprinkled with water,

with a rolling board, so as to make the cylinders smooth and of a uniform thickness. After this they are suspended again on the hoop, but in a reversed position, and the basting is continued till they are of the required size. When this is attained they are once more rolled on the slab, cut to a certain length, and have their tops trimmed with a piece of wood. The operation throughout is one requiring much skill and experience. A section of a well-made wax candle shows rings, resembling the annular layers of a tree, and corresponding to the number of *bastings*.

Large Wax Candles for ecclesiastical use are made by placing the wick on a layer of wax, bending the wax over it, and then rolling, as in the ordinary wax candles. Other layers of wax may, if necessary, be rolled on up to the required thickness.

Wax Tapers.—The materials—wax, with stearic acid, paraffin, &c.—are melted in a jacketed pan (Fig. 54); and

FIG. 54.



Silver-plated bougie or draw-wick pan, with winding drum, to heat by steam.

the wick, usually of several fine yarns of cotton, twisted to suit the thickness of the taper to be made, is wound on a drum, and drawn through the pan.

CHAPTER IV.

SPECIALITIES.

Belmont Sperm Candles.—The body of this description of candle is said to be a mixture of stearic and cocinic acids, with a portion of paraffin.

Belmont Wax Candles consist of stearic acid with a small proportion of wax. They are tinted with gamboge.

Ozokerit Candles.—These are a speciality of Field, of Lambeth. They have a remarkably high melting point and great illuminating power. They burn with a dry cup, are not liable to gutter, are free from smell, and not greasy to the touch. They do not bend or soften in a warm atmosphere like ordinary paraffin candles. The hardness and high melting give rise to one drawback—the wick is apt to smoulder on extinction. The cause of this is the fact that the cup of the candle dries and solidifies as soon as the flame is blown out, so that there is no liquid matter left to extinguish the spark. This difficulty, however, is overcome by special attention to the preparation of the wick.

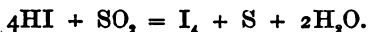
Double- and Treble-wick Candles of large diameter are made for police and nautical use.

Hydraulic-pressed Candles.—E. L. BROWN, of Chicago, has patented* a process to prevent unnecessary waste in

* United States patent No. 345,272, July 13, 1886.

the use of candles, by so treating them in the process of manufacture that they will melt very slowly. This is accomplished by forming the body of the candle under extreme pressure. The candle cylinder is first moulded in the usual way, and is then compressed by means of a hydraulic press.

Hygiénic Candles.—WATSON and FULTON* prepare these by incorporating iodine and a small quantity of sulphur with the candle material, and they consider that during the combustion the iodine and sulphur are both eliminated in the free state, according to the equation—



Wright's Pulmonic Candles.—These are impregnated with anti-asthmatic remedies, and are made on Messrs. FIELD's patent for securing perfect combustion and freedom from guttering by means of three or more air channels running parallel to the wick throughout the length of the candle.

SWEETSER, BELL, and BOHM have taken out a patent† for moulding and pressing candles direct from the candle material, whilst in a solid or plastic state, in continuous lengths. The material is kept under pressure, and, being forced through a tube, carries the wick along with it *in situ*. The coated wick has then only to be pointed, by being projected against a rotary cutter, or by other means, and cut into lengths to form candles.

Ornamental Candles.

Decorated Candles.—The materials for candles intended to be decorated should be of the best quality, and should have a high melting point. They may be varnished by gum

* English patent No. 10,876—1885.

† " " No. 13,417—1885.

dammar, dissolved in turpentine or alcohol, or by mastic varnish, and the design painted on by hand or otherwise.

Cable, Twisted, or Spiral Candles.—These are moulded in the ordinary way, and then turned by means of a special lathe; or they may be cast in rifled moulds, from which, on cooling, they are wound out.

Coloured Candles.—Among the colouring matters used for candles are the following:—

Blue: Prussian blue, indigo, ultramarine, copper sulphate, aniline blue.

Red: Carmine, Brazil wood, alkanet root, minium, vermilion, aniline reds.

Yellow: Gamboge, chrome yellow, naphthaline yellow.

Green: Mixture of blue and yellow colours.

Purple or Violet: Mixture of blue and red colours.

Neutral Tints: Oxides of iron, yellow ochre, Frankfort black.

Black: Fruit of *Anacardium occidentale*, aniline blacks.

In order to dye paraffin candles with an aniline base, such as magenta, the dye is first dissolved in stearin, and a little of the resulting stearate is added to the paraffin.

There are two ways in which candles may be coloured black:*

(1) *Anacardium Method.*—Paraffin, or whatever material is desired for the candles, is heated to from 200° to 210° C. with 25 per cent. of its weight of the chopped fruit of *Anacardium occidentale*. Candles prepared in this way are equally black throughout, and yield no irritating vapours when burnt.

(2) *Aniline Method.*—The material to be dyed is heated a few degrees above its melting point with 1 to 2 per cent. of nigrosine fat colour (prepared by Destrée, Wiescher, &

* "Chemist and Druggist," 1884, p. 290.

Co., of Brussels). Paraffin and spermaceti require 1 per cent.; stearin and wax require from $1\frac{1}{2}$ to 2 per cent. The candles thus prepared are said to be of a sombre hue throughout, and of a jet-black appearance.

Quality of Candles.

In judging of the quality of candles, the following points should be considered :*

(1) Nature of the fatty materials. (2) Whiteness. (3) Transparency. (4) Hardness. (5) Dryness to the touch. (6) Fusing point. (7) Form and moulding. (8) Character of wick. (9) Nature of the flame—is it uniform, long or short, well supplied, brilliant, without smoke? (10) Does the *cup* burn dry, or is it filled with melted fat? (11) Is the fatty matter free from mineral ingredients?

Bending Point.—Candles may be compared, as to their tendency to *bend* in warm atmospheres, by observing their behaviour when kept, for an hour or more, in a cupboard, or oven, heated to 100° F.

Illuminating Value.—The illuminating value of candles may be determined by the photometer, as described in the fourth volume of this series of Handbooks, pp. 310–315.

* CRISTIANI, "Treatise on Soap and Candles," p. 488.

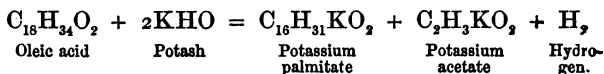
CHAPTER V.

BYE-PRODUCTS.

Oleic Acid.—The oleic acid may be used for soap-making, and is specially valuable for the production of soap for the use of textile manufacturers.

Oleic acid from the lime process is the best for this purpose, because it is free from hydrocarbons. If a soap is made from oleic acid containing hydrocarbons, when dissolved in water these separate and adhere to the fabric. Oleic acid free from hydrocarbons, when saponified by heating in a test-tube with twice its bulk of alcoholic soda, forms a soap which gives a *clear* solution when dissolved in water.

It may be also converted into palmitic acid by RADISSON's method, which is founded on the discovery of VARENTRAPP, in 1841, that when oleic acid is heated with a great excess of caustic potash it is decomposed into palmitic and acetic acids, and hydrogen, according to the equation—



The following is an outline of the method followed by RADISSON,* and described by CARPENTER:†—

* English patent 1782—1869.

† SPON'S "Encyclopædia," pp. 584—586; "Journ. Soc. Chem-Ind." 1883, p. 98.

About $1\frac{1}{2}$ ton of oleic acid and $2\frac{1}{2}$ tons of caustic potash lye (43° B.) are pumped into a cylindrical cast-iron vessel, about 12 feet in diameter and 5 feet high, provided with a sheet-iron cover. The vessel is heated from below by a fire, sufficiently far off to avoid burning. The steam evolved passes off by a large man-hole on the top. This is closed when the soap gets dry, and the gases afterwards disengaged are conveyed through pipes, first to a condensing tower, and thence to a gas-holder. The materials are kept constantly stirred by a mechanical agitator, in order that the heat may be equally distributed, and that the froth, which rises abundantly, may be beaten down. Eventually, the soap becomes fused, and at 554° F. begins to give off hydrogen. The temperature is slowly raised to 608° F., and the gases then given off have a characteristic odour. If the heat were longer continued the materials would enter on the stage of destructive distillation. The operation at this stage is therefore suddenly stopped by the introduction of steam and water through a GIFFARD injector, and, at the same time, a door in the bottom of the cylinder is opened, through which potassium palmitate falls into an open tank. Here the soap, with a sufficient quantity of water, is melted by means of a jet of steam. After subsidence the contents of the tank become separated into two layers, the upper of neutral potassium palmitate, and the lower of potash lye (usually about 18° B.). The palmitate is removed to another vessel, decomposed by sulphuric acid, and the liberated palmitic acid is washed with water to free it from potassium sulphate.

The palmitic acid thus obtained is of a clear chocolate colour, and crystallizes in large tables. Its melting or solidification point ranges from 122° to 127° F., according to the character of the oleic acid employed. Distilled in the usual apparatus, it leaves only 3 per cent. of pitch. After

distillation, it is very white, and burns with a clear smokeless flame. Moulded into candles, it compares very favourably with the best stearic acid, and, when mixed with ordinary stearic acid, *breaks the grain* of the latter, and gives it a semi-transparency very valuable in the eyes of the candle-manufacturer.

RADISSON has experimented with the object of replacing potash by soda, but experienced at first a difficulty in heating the materials uniformly. This difficulty he successfully overcame by introducing paraffin. When paraffin is present with sodium oleate and excess of soda, the mass becomes fluid on heating, and a uniform temperature throughout is speedily established. There is no fear of decomposing the sodium palmitate, since the point at which this would occur is above the temperature at which paraffin distils. The small quantities of paraffin which are unavoidably volatilized are caught in a condenser, and the hydrogen evolved is so charged with hydrocarbons as to form a good illuminant.

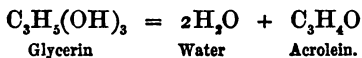
At the end of the reaction the whole is allowed to fall into water, as in the former process, and after a time three layers are formed—the bottom layer of soda lye and sodium acetate, the middle of neutral sodium palmitate, and the uppermost of paraffin. The top and bottom layers are removed, and serve for succeeding operations, and the sodium palmitate is decomposed by sulphuric acid. The palmitic acid obtained has, according to the author of the process, a solidifying point varying from 140° to 154° F., according to the kind of oleic acid operated upon.

A ton of palmitic acid by the first process costs about £13, by the second only about £7 10s.

The candle-maker gains, according to the inventor, the following advantages by adopting this process :—(1) Utilization of the olein, a troublesome bye-product of variable value. (2) The floating capital necessary for the purchase of raw

material is diminished by about 30 per cent., the proportion of hard candle material being increased by nearly the amount of olein produced. (3) Low-priced grease, whose value varies in inverse proportion to its richness in olein, can be employed. (4) The candle material produced is little, if at all, inferior to that produced by any other method.

Glycerin, $C_3H_5(OH)_3$.—*Syn.* GLYCEROL—the base of the ordinary fats, is a colourless, odourless, syrupy liquid of intensely sweet taste, and miscible in all proportions with water. It was discovered in 1779 by SCHEELÉ, who obtained it, in the preparation of lead-plaster, by saponifying lard with lead oxide. CHEVREUL afterwards showed that it is a constant product of the saponification of the ordinary fats. It is not susceptible of the alcoholic fermentation, but an aqueous solution of glycerin, if kept in a warm place, is slowly converted by the action of brewers' yeast into propionic acid ($C_3H_5O_2$). It has no action on vegetable colours. When heated in air at the ordinary pressure, it decomposes—one of the products being *acrolein* (C_3H_4O), which has a well-known peculiarly irritating odour:—



In presence of aqueous vapour under pressure in air and *in vacuo*, it can be distilled unchanged. Its specific gravity is 1.27–1.28. It boils *in vacuo* at 179.5° C., and at 755.55 mm. pressure at 200.08° C. According to F. NITZSCHE,* a method of obtaining glycerin in crystals was discovered by KRAUT in 1870. This method is applied in the works of Sarg & Co. at Liesing, near Vienna, to the production of glycerin, the crystals being freed from

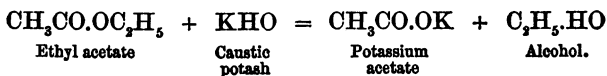
* "Dingl. Polyt. J." ccix. 145; WATTS' "Dictionary of Chem." vol. viii. pt. ii. suppl. 3, p. 871.

adhering mother liquor in a centrifugal machine, then dried, and melted.

When quite pure and anhydrous, it crystallizes* on exposure to a very low temperature, especially if agitated. The crystals so obtained are mono-clinic, perfectly colourless, and melt at 60° F.

According to WERNER,† commercial glycerin may be made to crystallize by passing a few bubbles of chlorine into it.

Glycerin does not, for the most part, exist in the free state, or ready formed, in natural fats, but, when the fat is saponified, glycerin is formed by the addition of the elements of water to the radical *glyceryl* (see p. 52). The reaction is similar to that by which common alcohol may be produced from ethyl acetate (acetic ether):—



In fact, glycerin is an alcohol, bearing the same relation to the fats stearin, palmitin, olein, &c., that ordinary alcohol bears to the compound ethers.

BERTHELOT's researches on the synthesis of fats, by the direct action of acids on glycerin, have shown that glycerin is a tri-atomic alcohol, in which one, two, or three atoms of hydrogen may be replaced by acid radicals, producing fatty or oily compounds, some of which are identical in composition and properties with the natural fats.‡

The following table shows the specific gravities and freezing points of aqueous solutions containing different percentages by weight of glycerin:—

* Roos, "Journ. Chem. Soc." 1876, i. 651.

† "Zeitschr. f. Chem." [2], iv. 413.

‡ See also "Oils and Varnishes," p. 11.

| Percentage. | Specific Gravity. | Freezing Point (C.). |
|-------------|-------------------|----------------------|
| 10 | 1.024 | - 1° |
| 20 | 1.051 | - 2.5° |
| 30 | 1.075 | - 6° |
| 40 | 1.105 | - 17.5° |
| 50 | 1.127 | - 31.34° |
| 60 | 1.159 | } Below - 35° |
| 70 | 1.179 | |
| 80 | 1.120 | |
| 90 | 1.232 | |
| 94 | 1.241 | |

Some of the chief methods proposed for the recovery of glycerin from soap lyes have been given in Part I. p. 175.

The glycerin separated in the various processes for the preparation of fatty acids to be used in the manufacture of candles is now of great commercial importance. The crude or raw glycerin is obtained by concentrating the sweet water by evaporation to about 44° Tw. (sp. gr. 1.22). Some candle-makers carry the operation no farther, but dispose of the raw article to those who make its purification a branch of their business.

The purification may be effected by superheated steam in the manner already described (pp. 247 and 248).

Removal of Glycerin from Fats before Saponification.—A patent has been taken out in the name of IMRAY* with this object. The fatty matter is mixed with about one-third of its weight of water, and from $\frac{1}{8}$ to $1\frac{1}{2}$ per cent. of its weight of zinc oxide. It is then subjected in a close vessel to the action of steam at a pressure of from 100 to 130 lb. per square inch from three to four hours. The product thus saponified is treated as in calcareous saponification, but the very small proportion of mineral substance used enables the acid treatment for decomposition of the soap to be dispensed with, and the acid fat can be at once employed in the manufacture of soap or candles.

* English patent 5112, October 27, 1882.

Testing Glycerin.*—Oxide of lead, lime, and butyric acid, the result of incomplete purification, are the impurities most frequently met with in commercial glycerin.

Lime and *lead* are indicated when, on the addition of a few drops of dilute sulphuric acid to a portion of the sample diluted with its own volume of water and with a little alcohol, a white precipitate is obtained. If the precipitate is blackened by sulphuretted hydrogen, lead is present.

Butyric acid is detected by mixing strong alcohol and sulphuric acid with the sample, and heating slightly, when, if this impurity be present, the agreeable odour of butyric ether becomes manifest.

Formic acid, if present, gives the odour of formic ethyl (peach-flower smell) when the glycerin is heated with alcohol of 40° and a drop of sulphuric acid.

Oxalic acid would be shown by a white precipitate on the addition to equal quantities of glycerin and water of 2 drops of a solution of calcium chloride containing a little ammonia (free from carbonate).

Glucose would reduce FEHLING's copper solution ; and *cane sugar*, after inversion by a mineral acid, would be detected by the same reagent. Or, the presence of either would be detected by the polariscope, as glycerin itself has no optical activity.

The *chloroform* test consists in mixing equal parts of chloroform and glycerin, stirring, and then leaving the mixture to settle. Of the two layers which form, the upper one consists of pure glycerin, the lower of chloroform with the impurities. If the glycerin is pure, the chloroform remains clear ; if not, a greyish belt is observed at the line of separation.

Perfumers test glycerin with silver nitrate ; if pure, there

* F. JEAN, "Journ. de Pharm. d'Alsace-Lorraine," ix, 136 ; "Year Book of Pharmacy," 1883, p. 258.

is no sensible coloration produced at the end of twenty-four hours.

Sulman and Berry on the Examination of Commercial Glycerin.*—Colour.—SULMAN and BERRY state that the colour of commercial glycerin does not necessarily indicate whether a sample is crude or once distilled, for, although crude samples are usually highly coloured, pale samples are often obtained by the lime process, while once distilled samples from soap lyes are sometimes very dark.

Mineral Matter.—On incineration, distilled glycerin never yields more than 0.2 per cent. of mineral matter. Crude glycerin from soap lyes gives from 6 to 14 per cent. of ash. The crude product obtained in candle factories, either by the lime, magnesia, zinc, or other processes, contains a smaller proportion of mineral matter than that from soap lyes.

“Crude glycerin invariably contains albuminous matters, derived from the nitrogenous envelope of the fat globules, often to the extent of several per cent. Here, as usual, it is the soap lyes which yield the most heavily contaminated samples owing to the ready solvency of the proteid matters contained in the fats by the alkalies employed. They are chiefly objectionable on account of the mechanical difficulties to which they give rise in the subsequent distillation, and on account of the contamination of the distillate with empyreumatic and coloured products. In glycerin from soap lyes a frequent and very objectionable impurity is rosin, which often imparts a characteristic fluorescence to the distillate. Rosin oils may be detected in the distilled samples by shaking with ether, the bulk of which rises to the surface on standing, and contains most of the oil present, which may be recognized on evaporation of the de-

* “Analyst,” 1886, p. 12.

canted ether by its physical character, its odour on warming, and its characteristic taste. Glycerin from candle factories contains no rosin.

“On acidifying crude glycerin from soap lyes, a milky white precipitate is frequently obtained, the quantity of which depends upon the process of extraction adopted, and whether acidification has previously taken place. The precipitate consists mainly of resinous acids and free sulphur (the latter being due to the decomposition of the sulphur compounds introduced with the caustic soda used for the saponification of the fats); the sulphur has been found at times to constitute 40 to 60 per cent. of the whole precipitate. The sulphur is hardly less objectionable than the rosin, as it gives rise to volatile sulphur compounds on distillation.

“The albuminous matters cannot be completely removed from the crude glycerin except by distillation, and for analytical purposes it is not necessary to separate them from the other organic impurities, which are separable by basic acetate of lead, and of which they form the bulk.

“Crude glycerin obtained by the sulphuric acid processes of fat saponification is always charged with sulphates, and generally sulphites; occasionally appreciable quantities of sulphide are found. Both the latter are injurious in distillation. Glycerin from candle factories frequently contains some free fatty acid, which is usually oleic.

“With regard to the impurities in distilled glycerin the traces of mineral matter present may consist of sodic chloride and salts of lime, copper, and iron, the two latter being derived from the still and fittings, the presence of the copper being due to formic acid. This acid is produced either by the action of traces of mineral acids upon the glycerin in the still (oxalic acid being first formed and again immediately decomposed with liberation of carbonic

acid and the volatile fatty acid), or as one of the final products of the action of the small quantity of alkali remaining in the crude glycerin upon the albuminous impurities. The chief organic impurities of first distillates from soap lyes are formic, butyric, and oleic acids, rosin oils, colouring and empyreumatic products, and occasionally organic sulphuric compounds. In the samples from candle factories butyric acid sometimes (according to PERUTZ) reaches the amount of 0.5 per cent. The glycerin obtained by the WILSON-PAYNE process generally contained considerable amounts of the higher fatty acids.

“ For the determination of the *total mineral matter* present in a sample, two separate ignitions are requisite, as it is impossible to burn off all the carbonaceous portion of the residue without volatilizing some of the salt which is almost invariably present. The first portion taken is warmed, the vapours ignited, &c., and the charred mass so obtained is exhausted with hot water. The solution is filtered, and the chlorides determined by titration with standard silver solution. A second portion is burnt in the same way, and the residue strongly ignited, using the blowpipe, if necessary, till no more carbon remains, and the ash is fairly white; the weight is taken, the residue dissolved, and the chlorides determined in it. The difference between the two determinations gives the amount of chloride volatilized, which is calculated as sodic chloride and added to the weight of the second ash.

“ *Chlorides* cannot be directly determined in glycerin by precipitation or titration with silver, owing to the solubility of argentic chloride in this liquid, and to the reduction of the nitrate in the cold by the contained impurities. They are therefore determined in the ash, as before directed. Crude soap lye glycerin usually contains from 5 to 10 per cent. of salt.

"*Alkalinity*, due almost entirely to sodic carbonate, is most readily estimated by titration of the diluted sample with standard acid. Litmus is the best indicator, phenolphthalein and methyl orange giving indistinct end reactions. Crude soap glycerins are usually alkaline, and purposely so, owing to the risk of concentrating them in presence of acid. We have found them to contain, as a rule, from 0.5 to 2 per cent., the amount present depending to some extent upon the process adopted in their preparation. In a case, cited by Dr. FLEMING,* where the soap had been separated from the lyes by excess of alkali instead of salt, the resulting glycerin contained 31 per cent. of sodic carbonate.

"*Calcium, Zinc, Iron, Magnesia* are determined as usual in the ash.

"CAP'S test for *lime* in the original glycerin consists in the addition to the sample of an equal volume of alcohol containing 1 per cent. of sulphuric acid, the alcohol largely diminishing the solubility of the calcic sulphate: we have found, however, that the ordinary ammonium oxalate test gives quite as delicate results.

"*Organic Impurities*, consisting of albuminous and resinous compounds, colouring matters, and the higher fatty acids, are largely precipitable by basic lead acetate, and may be estimated with considerable accuracy by a modification of CHAMPION and PELLET'S process. The glycerin is sufficiently diluted with water, carefully neutralized with acetic acid, and warmed to expel carbonic acid: when cool, the basic acetate is added in slight but distinct excess in the cold, and the mixture well agitated. The precipitate is collected upon a tared, or, better, a double counterpoised filter, well washed (the first washings may be effected by

* "Seifenfabrikant," i. 110.

decantation in the beaker), dried at $100^{\circ}\text{C}.$ to $105^{\circ}\text{C}.$, and weighed. The precipitate and filter papers are ignited separately, each with a few drops of nitric acid. The weight of the lead oxide (and perhaps sulphate) obtained, when deducted from the weight of the dried precipitate, gives the organic matter contained by the latter. The nitric acid prevents the reduction of the plumbic sulphate, when present, to sulphide or metallic lead; to control the result, and with the above view, the precipitate, instead of being ignited, may be treated with hot nitric acid, diluted, and filtered, the lead being determined in the filtrate by any convenient method. In this way any sulphate of lead present in the precipitate is left undissolved. It has been recommended to ignite the precipitate obtained by the basic acetate of lead, with sulphuric acid, to multiply the weight of the sulphate obtained by 0.736, and to deduct the weight of lead oxide obtained as before directed; but this method is obviously inaccurate, as it takes no account of the sulphates almost invariably present, to some extent, in crude glycerin, and thus includes the sulphuric acid in terms of 'organic impurity.' No average figures can be given as to the varying amounts of the above impurities for crude glycerin, but in distilled samples the amount present should not exceed 0.5 to 1 per cent.

"*Fatty Acids* are often present in such proportions that mere dilution with water causes their precipitation; smaller quantities may be detected by diluting and applying the elaidin test—the flocculent yellowish precipitate of elaidic acid obtained by passing peroxide of nitrogen through the solution being less soluble in glycerin than the original oleic acid.

"*The Nitrate of Silver Test*, used by perfumers, depends upon the production of a black precipitate of metallic silver on standing for some time. This reduction is principally

due to the presence of small quantities of acrolein and of formic acid; a good distilled glycerin should give no precipitate after twenty-four hours, though nearly all commercial samples we have met with in bulk do speedily effect reduction.

"For the detection and estimation of *Sugar*, FEHLING's method is readily applicable. This substance cannot occur except as an adulteration, and hence it is only necessary to look for it in distilled samples. The same remark of course applies to *glucose*. Sucrose and dextrin, it need hardly be said, require the usual inversion by heating with dilute (5 per cent.) sulphuric acid before applying the FEHLING solution. The small amounts of other impurities present do not interfere with this test. That it is constantly necessary to examine samples for sugar is shown by the fact that a spurious glycerin has been found by a continental chemist to be composed of a saturated solution of glucose and magnesium sulphate.

"For *Sugar* (also *glucose* and *dextrin*, but not *lactose* or *arabin*, which give no reaction) MASON's test has been found fairly delicate and reliable, using 0.5 c.c. of the suspected glycerin, 15 c.c. distilled water, 2 drops of strong nitric acid, and 0.5 gram of ammonic molybdate. On boiling for two or three minutes, or longer, if the quantity present be small, a blue colour is produced by the above substances; 0.25 per cent. may be readily detected. The chief points to be observed are—that the liquid must not be too highly coloured, and the acid must not be in excess of the quantity mentioned.

"ZSIGMONDY and BENEDIKT* have recently put forward a process for the estimation of glycerin, depending upon its oxidation by alkaline permanganate solution into oxalic

* "Analyst," November 1885, p. 205.

acid ; the latter is precipitated by adding calcic acetate, and may be determined by titration with standard potassic permanganate in an acid solution. Fatty acids have been found not to interfere with this reaction."

For the **manufacture of dynamite**, which forms the great outlet at present for the large quantities of glycerin obtained from the soap and candle industries, *distilled* glycerin is alone of use ; and it must further answer the following conditions before it can be accepted as sufficiently pure for nitration :—

(1) Entire freedom from salt, iron, lead, lime, and fatty acids.

(2) Complete absence of sugar (which can be present only as an adulteration).

(3) The sample must be of good colour and practically odourless.

(4) Specific gravity must at least reach 1.26.

Olein.—The olein obtained in the preparation of stearin (p. 231),* and known technically as *oleo*, is now largely employed in the manufacture of margarine.

* For further details see "Oils and Varnishes," p. 18 ; and Cantor Lectures on "Milk Supply, Butter, and Cheese-making," by R. BANNISTER (April 1888).

APPENDIX.

Composition of Black Ash.

| | 1. UNGER. | 2. STOR- MANN. | 3. BROWN and KYNASTON |
|----------------------------------|--------------|----------------------|--------------------------------|
| Sodium sulphate | 1.99 | 1.54 | 0.395 |
| „ chloride | 2.54 | 1.42 | 2.528 |
| „ carbonate | 23.57 | 44.41 | 36.879 |
| „ silicate | — | — | 1.182 |
| „ aluminate | — | — | 0.689 |
| Soda—caustic, hydrated | 11.12 | | |
| Calcium carbonate | 12.90 | 3.20 | 3.315 |
| „ sulphide | 27.61 | 30.96 | 28.681 |
| „ sulphite | — | — | 2.178 |
| Lime | 7.15 | 8.35 | 9.270 |
| Magnesia | — | 0.10 | 0.254 |
| Magnesium silicate | 4.74 | | |
| Alumina | — | 0.79 | 1.132 |
| Water | 2.10 | — | 0.219 |
| Ferric oxide | — | 1.75 | 2.658 |
| Ferrous sulphide | 2.45 | — | 0.371 |
| Silica | — | 0.89 | |
| Sand | 2.02 | 2.20 | 0.901 |
| Charcoal | 1.59 | 5.32 | 7.007 |
| Ultramarine | — | — | 0.959 |
| Total | 99.78 | 100.93 | 98.180 |

Strength of Solutions of Caustic Potash at 15° C. (59° F.)

(TUNNERMANN).

| Density. | K ₂ O in 100 Parts. | Density. | K ₂ O in 100 Parts. |
|----------|-----------------------------------|----------|-----------------------------------|
| 1.3300 | 28.290 | 1.1437 | 14.145 |
| 1.3131 | 27.158 | 1.1308 | 13.013 |
| 1.2966 | 26.027 | 1.1182 | 11.882 |
| 1.2805 | 24.895 | 1.1059 | 10.750 |
| 1.2648 | 23.764 | 1.0938 | 9.619 |
| 1.2493 | 22.632 | 1.0819 | 8.487 |
| 1.2342 | 21.500 | 1.0703 | 7.355 |
| 1.2268 | 20.935 | 1.0589 | 6.224 |
| 1.2122 | 19.803 | 1.0478 | 5.002 |
| 1.1979 | 18.671 | 1.0369 | 3.961 |
| 1.1839 | 17.540 | 1.0260 | 2.829 |
| 1.1702 | 16.408 | 1.0153 | 1.697 |
| 1.1568 | 15.277 | 1.0050 | 0.566 |

Density of Caustic-potash Solutions (SCHIFF).

| Density. | KHO in 100 Parts. | Density. | KHO in 100 Parts. |
|----------|----------------------|----------|----------------------|
| 1.036 | 5 | 1.411 | 40 |
| 1.077 | 10 | 1.475 | 45 |
| 1.124 | 15 | 1.539 | 50 |
| 1.175 | 20 | 1.604 | 55 |
| 1.230 | 25 | 1.667 | 60 |
| 1.288 | 30 | 1.729 | 65 |
| 1.349 | 35 | 1.790 | 70 |

Strength of Solutions of Caustic Soda at 15° C. (59° F.)

(TUNNERMANN).

| Density. | Na ₂ O in 100 Parts. | Density. | Na ₂ O in 100 Parts. |
|----------|------------------------------------|----------|------------------------------------|
| 1.4285 | 30.220 | 1.2392 | 15.110 |
| 1.4193 | 29.616 | 1.2280 | 14.506 |
| 1.4101 | 29.011 | 1.2178 | 13.901 |
| 1.4011 | 28.407 | 1.2058 | 13.297 |
| 1.3923 | 27.802 | 1.1948 | 12.692 |
| 1.3836 | 27.200 | 1.1841 | 12.088 |
| 1.3751 | 26.594 | 1.1734 | 11.484 |
| 1.3668 | 25.989 | 1.1630 | 10.879 |
| 1.3586 | 25.385 | 1.1528 | 10.275 |
| 1.3505 | 24.780 | 1.1428 | 9.670 |
| 1.3426 | 24.176 | 1.1330 | 9.066 |
| 1.3349 | 23.572 | 1.1233 | 8.462 |
| 1.3273 | 22.967 | 1.1137 | 7.857 |
| 1.3198 | 22.363 | 1.1042 | 7.253 |
| 1.3143 | 21.894 | 1.0948 | 6.648 |
| 1.3125 | 21.758 | 1.0855 | 6.044 |
| 1.3053 | 21.154 | 1.0764 | 5.440 |
| 1.2982 | 20.550 | 1.0675 | 4.835 |
| 1.2912 | 19.945 | 1.0587 | 4.231 |
| 1.2843 | 19.341 | 1.0500 | 3.626 |
| 1.2775 | 18.730 | 1.0414 | 3.022 |
| 1.2708 | 18.132 | 1.0330 | 2.418 |
| 1.2642 | 17.528 | 1.0246 | 1.813 |
| 1.2578 | 16.923 | 1.0163 | 1.209 |
| 1.2515 | 16.319 | 1.0081 | 0.604 |
| 1.2453 | 15.714 | | |

Soda Ash: Table of Percentages of Soda and Sodium Carbonate corresponding to "English" and DECROIZILLES' Degrees.

| Percentage of | | Degrees. | | Percentage of | | Degrees. | |
|---------------|-------------------|----------|----------------|---------------|-------------------|----------|----------------|
| Soda. | Sodium Carbonate. | English. | DECROIZILLES'. | Soda. | Sodium Carbonate. | English. | DECROIZILLES'. |
| 30.0 | 51.29 | 30.39 | 47.42 | 49.0 | 83.78 | 49.64 | 77.45 |
| 30.5 | 52.14 | 30.90 | 48.21 | 49.5 | 84.64 | 50.15 | 78.24 |
| 31.0 | 53.00 | 31.41 | 49.00 | 50.0 | 85.48 | 50.66 | 79.03 |
| 31.5 | 53.85 | 31.91 | 49.79 | 50.5 | 86.34 | 51.16 | 79.82 |
| 32.0 | 54.71 | 32.42 | 50.58 | 51.0 | 87.19 | 51.67 | 80.61 |
| 32.5 | 55.56 | 32.92 | 51.37 | 51.5 | 88.05 | 52.18 | 81.40 |
| 33.0 | 56.42 | 33.43 | 52.16 | 52.0 | 88.90 | 52.68 | 82.19 |
| 33.5 | 57.27 | 33.94 | 52.95 | 52.5 | 89.76 | 53.19 | 82.98 |
| 34.0 | 58.13 | 34.44 | 53.74 | 53.0 | 90.61 | 53.70 | 83.77 |
| 34.5 | 58.98 | 34.95 | 54.53 | 53.5 | 91.47 | 54.20 | 84.56 |
| 35.0 | 59.84 | 35.46 | 55.32 | 54.0 | 92.32 | 54.71 | 85.35 |
| 35.5 | 60.69 | 35.96 | 56.11 | 54.5 | 93.18 | 55.22 | 86.14 |
| 36.0 | 61.55 | 36.47 | 56.90 | 55.0 | 94.03 | 55.72 | 86.93 |
| 36.5 | 62.40 | 36.98 | 57.69 | 55.5 | 94.89 | 56.23 | 87.72 |
| 37.0 | 63.26 | 37.48 | 58.48 | 56.0 | 95.74 | 56.74 | 88.52 |
| 37.5 | 64.11 | 37.99 | 59.27 | 56.5 | 96.60 | 57.24 | 89.31 |
| 38.0 | 64.97 | 38.50 | 60.06 | 57.0 | 97.45 | 57.75 | 90.10 |
| 38.5 | 65.82 | 39.00 | 60.85 | 57.5 | 98.31 | 58.26 | 90.89 |
| 39.0 | 66.68 | 39.51 | 61.64 | 58.0 | 99.16 | 58.76 | 91.68 |
| 39.5 | 67.53 | 40.02 | 62.43 | 58.5 | 100.02 | 59.27 | 92.47 |
| 40.0 | 68.39 | 40.52 | 63.22 | 59.0 | 100.87 | 59.77 | 93.26 |
| 40.5 | 69.24 | 41.03 | 64.01 | 59.5 | 101.73 | 60.28 | 94.05 |
| 41.0 | 70.10 | 41.54 | 64.81 | 60.0 | 102.58 | 60.79 | 94.84 |
| 41.5 | 70.95 | 42.04 | 65.60 | 60.5 | 103.44 | 61.30 | 95.63 |
| 42.0 | 71.81 | 42.55 | 66.39 | 61.0 | 104.30 | 61.80 | 96.42 |
| 42.5 | 72.66 | 43.06 | 67.18 | 61.5 | 105.15 | 62.31 | 97.21 |
| 43.0 | 73.52 | 43.57 | 67.97 | 62.0 | 106.01 | 62.82 | 98.00 |
| 43.5 | 74.37 | 44.07 | 68.76 | 62.5 | 106.86 | 63.32 | 98.79 |
| 44.0 | 75.23 | 44.58 | 69.55 | 63.0 | 107.72 | 63.83 | 99.58 |
| 44.5 | 76.08 | 45.08 | 70.34 | 63.5 | 108.57 | 64.33 | 100.37 |
| 45.0 | 76.95 | 45.59 | 71.13 | 64.0 | 109.43 | 64.84 | 101.16 |
| 45.5 | 77.80 | 46.10 | 71.92 | 64.5 | 110.28 | 65.35 | 101.95 |
| 46.0 | 78.66 | 46.60 | 72.71 | 65.0 | 111.14 | 65.85 | 102.74 |
| 46.5 | 79.51 | 47.11 | 73.50 | 65.5 | 111.99 | 66.36 | 103.53 |
| 47.0 | 80.37 | 47.62 | 74.29 | 66.0 | 112.85 | 66.87 | 104.32 |
| 47.5 | 81.22 | 48.12 | 75.08 | 66.5 | 113.70 | 67.37 | 105.11 |
| 48.0 | 82.07 | 48.63 | 75.87 | 67.0 | 114.56 | 67.88 | 105.90 |
| 48.5 | 82.93 | 49.14 | 76.66 | 67.5 | 115.41 | 68.39 | 106.69 |

Soda Ash: Table of Percentages of Soda, &c.—(continued).

| Percentage of | | Degrees. | | Percentage of | | Degrees. | |
|---------------|-------------------|----------|------------------|---------------|-------------------|----------|------------------|
| Soda. | Sodium Carbonate. | English. | DEGREES ZILLES'. | Soda. | Sodium Carbonate. | English. | DEGREES ZILLES'. |
| 68.0 | 116.27 | 68.89 | 107.48 | 73.0 | 124.81 | 73.96 | 115.39 |
| 68.5 | 117.12 | 69.40 | 108.27 | 73.5 | 125.66 | 74.47 | 116.18 |
| 69.0 | 117.98 | 69.91 | 109.06 | 74.0 | 126.52 | 74.97 | 116.97 |
| 69.5 | 118.83 | 70.41 | 109.85 | 74.5 | 127.37 | 75.48 | 117.76 |
| 70.0 | 119.69 | 70.92 | 110.64 | 75.0 | 128.23 | 75.99 | 118.55 |
| 70.5 | 120.53 | 71.43 | 111.43 | 75.5 | 129.08 | 76.49 | 119.34 |
| 71.0 | 121.39 | 71.93 | 112.23 | 76.0 | 129.94 | 77.00 | 120.13 |
| 71.5 | 122.24 | 72.44 | 113.02 | 76.5 | 130.79 | 77.51 | 120.92 |
| 72.0 | 123.10 | 72.95 | 113.81 | 77.0 | 131.65 | 78.01 | 121.71 |
| 72.5 | 123.95 | 73.45 | 114.60 | 77.5 | 132.50 | 78.52 | 122.50 |

Exports of Soap and Candles.

| Year. | Soap. | | Candles of all Sorts. | |
|-------|---------|----------|-----------------------|----------|
| | Cwts. | Value. | Lbs. | Value. |
| 1878 | 335,592 | £405,183 | 5,345,900 | £170,161 |
| 1879 | 383,910 | 432,699 | 4,790,800 | 135,852 |
| 1880 | 391,808 | 440,286 | 5,051,800 | 143,231 |
| 1881 | 353,733 | 397,516 | 5,071,700 | 137,677 |
| 1882 | 409,907 | 458,381 | 4,992,744 | 135,051 |
| 1883 | 391,788 | 449,804 | 5,285,600 | 147,961 |
| 1884 | 476,438 | 547,613 | 7,703,400 | 213,635 |
| 1885 | 402,254 | 472,519 | 7,810,400 | 200,179 |
| 1886 | 426,904 | 446,710 | 8,967,100 | 201,919 |
| 1887 | 451,961 | 451,246 | 9,321,600 | 180,912 |

Imports of Tallow and Stearin.

| Year. | From | | | | |
|-------|------------------|---------------------------------|-------------------------|---------------------|------------------------------|
| | Russia. Cwts. | Argentine Republic. Cwts. | United States. Cwts. | Australia. Cwts. | Other Countries. Cwts. |
| 1878 | 73,646 | 66,754 | 455,991 | 216,722 | 105,820 |
| 1879 | 48,401 | 59,988 | 564,489 | 361,124 | 137,651 |
| 1880 | 25,505 | 103,665 | 516,715 | 492,527 | 178,678 |
| 1881 | 24,378 | 21,778 | 413,904 | 598,962 | 133,629 |
| 1882 | 33,497 | 128,119 | 291,641 | 434,415 | 231,167 |
| 1883 | 6,171 | 72,075 | 333,358 | 445,726 | 179,897 |
| 1884 | 14,724 | 97,703 | 332,459 | 477,680 | 187,315 |
| 1885 | 7,172 | 107,301 | 241,685 | 410,439 | 243,959 |
| 1886 | 35,579 | 55,677 | 337,443 | 388,628 | 193,069 |
| 1887 | 6,532 | 22,209 | 329,367 | 416,658 | 120,892 |

*Statistics of Soap and Candle Factories in the United States (1880).**

| | |
|---|--------------|
| No. of establishments | 629 |
| Capital | \$14,541,294 |
| Average No. of hands employed :— | |
| Males above sixteen years | 4,368 |
| Females above fifteen years | 388 |
| Children and youths | 533 |
| Total amount paid in wages during the year | \$2,219,513 |
| Value of materials | \$19,907,444 |
| ,, products | \$26,552,627 |

* "Report of the Manufactures of the United States (Tenth Census, 1880)." Published 1883.

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