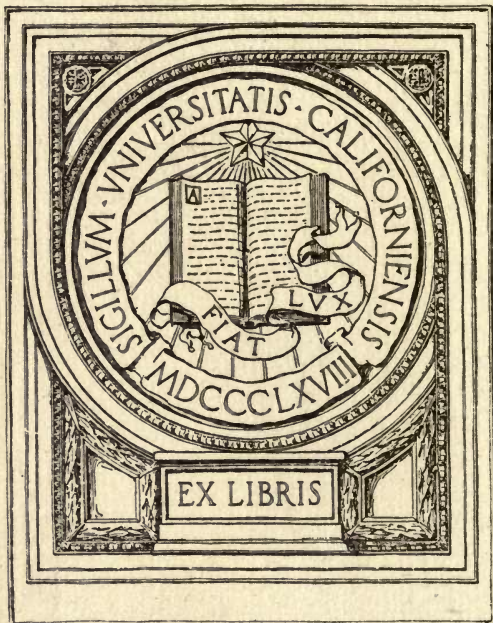


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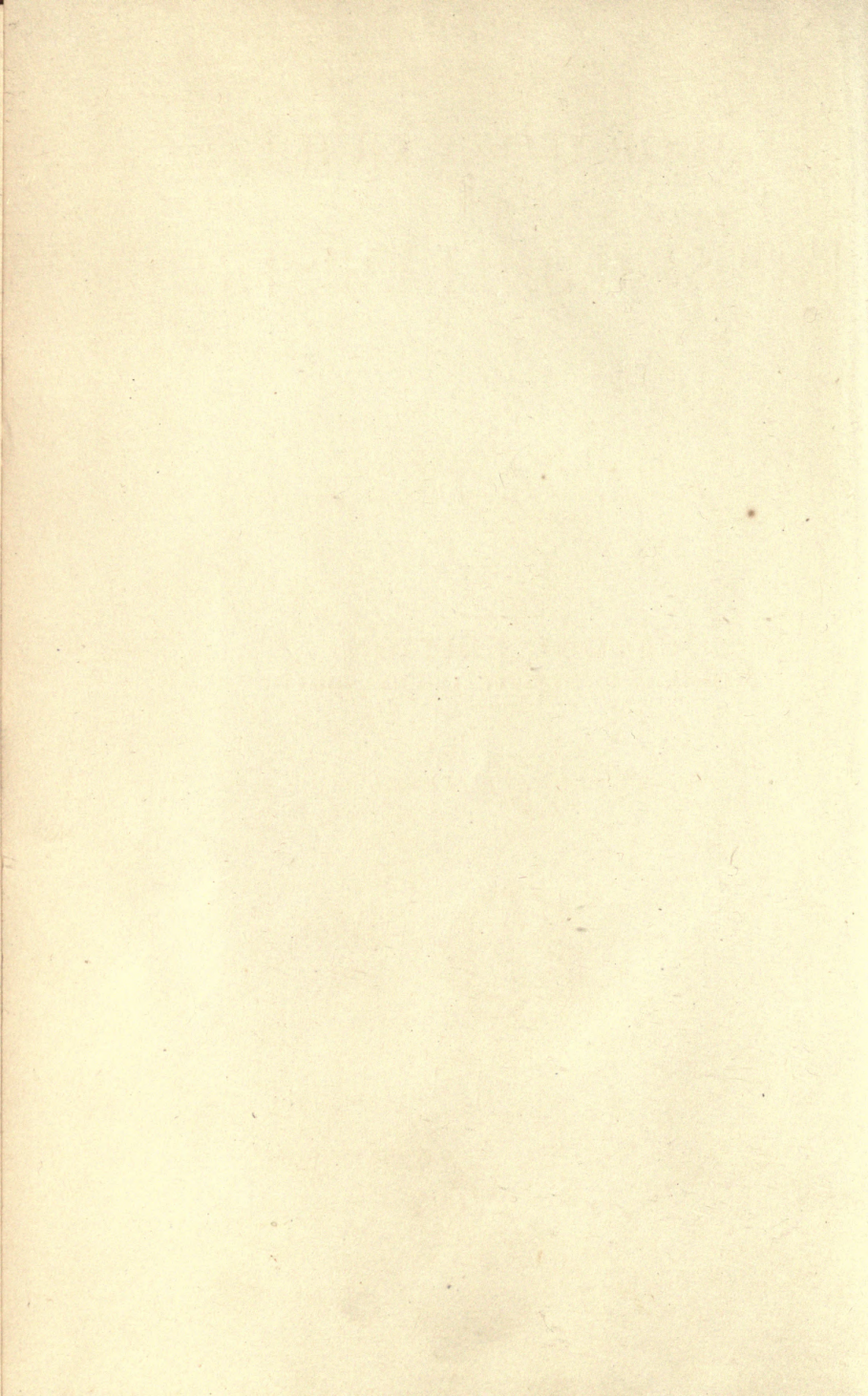


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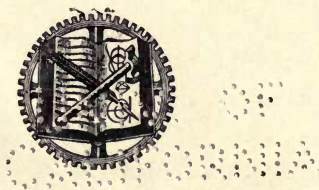
LABORATORY GUIDE
OF
INDUSTRIAL CHEMISTRY

BY
ALLEN ROGERS

IN CHARGE OF INDUSTRIAL CHEMISTRY, PRATT INSTITUTE,
BROOKLYN, N. Y.

SECOND EDITION
ENTIRELY REWRITTEN AND ENLARGED

33 ILLUSTRATIONS



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PREFACE TO SECOND EDITION

IN presenting this thoroughly revised edition, the author has endeavored to make the laboratory experiments touch more closely upon present-day problems than was possible in the first edition. It has been the aim to treat the subject in such a manner that the processes described for application on a small scale can also be adopted to the larger commercial basis. Thus, in many instances, it will be noticed that the methods are those in common use at the present time, and, whenever possible, actual factory practice has been followed. The object, therefore, is to acquaint the student of chemistry with actual commercial problems by bringing to his experience practical methods of handling materials on a large scale; the care and use of machinery; the cost of raw materials; transportation; wage system; the handling of men; and shop discipline.

This idea is being carried out at Pratt Institute through a foremanship system of instruction, in which groups of students are assigned to work in model plants, one member of each group acting as foreman. The foreman is held responsible for the quality as well as the quantity of the output of his plant, must see that the machinery is left in perfect condition, and that the factory is kept clean.

In these days, when manufacturers are turning to technically trained men to fill positions of respon-

sibility in their plants, it behooves the technical schools to make the training of young men as broad as possible along applied lines, for the man with a knowledge of manufacturing operations and factory equipment will have a decided advantage over the one with a purely scientific education.

Inasmuch as this book is a plea for a more widespread introduction of Industrial Chemistry into technical schools, the author would greatly appreciate any suggestions that might make the book more completely serve its purpose. The text presupposes a certain knowledge of chemistry and purposely leaves many of the minor details to be worked out by the student. To the instructor also is given great latitude that he may exercise his own originality the more freely.

ALLEN ROGERS.

PRATT INSTITUTE,
BROOKLYN, AUGUST, 1917.

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LABORATORY GUIDE OF INDUSTRIAL CHEMISTRY

CHAPTER I

GENERAL PROCESSES

1. Crushing. In the preparation of chemical products, one of the most important features is the converting of raw material to a state of fine division. This may be accomplished in several ways, depending upon the nature of the raw material.

For fairly hard material the laboratory crusher (Fig. 1) is usually employed. In its simplest form the crusher consists of a steel plate against which a corresponding steel jaw works on a cam, giving a rolling motion. This form of crusher will be found in practically any well equipped laboratory and can be purchased at a nominal cost. In the manufacture of such chemicals as magnesium sulphate, barium chloride, or similar products, the ore should first be brought to a state of fine division in this form of crusher.

2. Grinding. It is often necessary to reduce an ore or other raw material to a much finer state of division than is possible with the jaw crusher; for such material some other form of grinding machine must be used.

The simplest and most common form of grinding apparatus consists of two hardened steel plates, one of which rotates rapidly over the other in a manner somewhat similar to

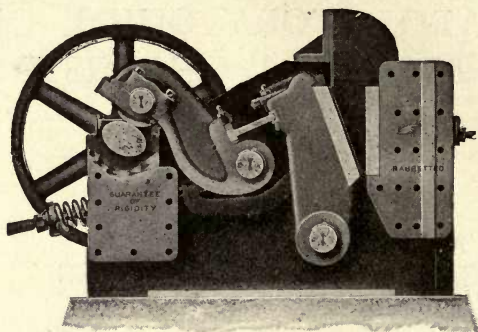


FIG. 1.

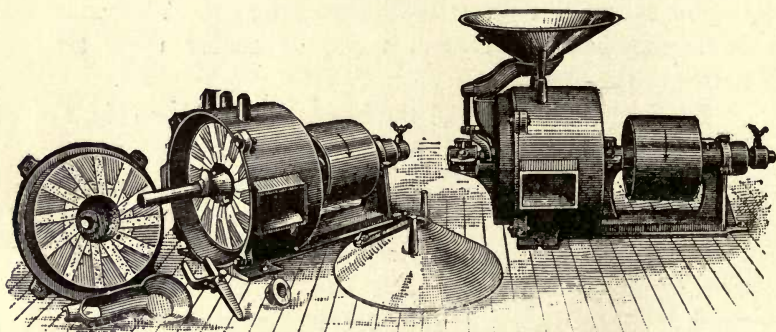


FIG. 2.

the ordinary grinding in a mortar. There are several types of such machines on the market, but, as they all work on the same principle, no special recommendation is needed for any particular one. A common form of laboratory mill is shown in Fig. 2. It should

always be borne in mind that the finer the material is ground, the more rapid and complete will be the action when chemical treatment takes place.

3. Lixiviation. This process effects a separation of water-soluble material from insoluble or less soluble material. The material to be lixiviated is placed in open tanks provided with perforated false bottoms, and the necessary amount of water added. The tanks are sometimes equipped with closed steam coils for heating. They

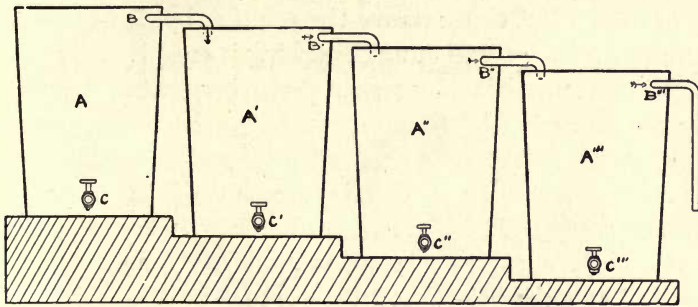


FIG. 3.

are usually arranged in series, constituting what is called a battery (Fig. 3). The solution containing the soluble material is drawn off from the false bottom of one tank into the top of the next, thus coming into contact each time with fresher material and increasing in strength, while fresh water, added to the tank after each successive drawing off, comes in contact with the partly exhausted material, dissolving out still more of the soluble portion.

4. Decantation. This process is commonly used when the preparation of chemical products is accompanied by

the formation of a certain amount of insoluble material. The precipitate, or insoluble residue, is allowed to settle and the clear, supernatant liquid removed either by means of a siphon or by pouring over the side of the vessel.

5. Washing. In many instances, it is necessary to remove the excess of impurities from a product, and, where these impurities are of a soluble nature, care must be taken to remove them as completely as possible. The simplest method of washing large quantities of material is to stir the insoluble residue with sufficient water, allow it to settle, and then remove the solution by decantation, continuing the operation until washing is complete.

6. Concentration. By concentration is meant the process by which a material is converted into a higher degree of compactness. For most chemical purposes, this is brought about by removing a certain amount of the solvent, usually water. There are cases, however, where concentration may apply to mixtures of solids, the more highly purified material being known as concentrates.

7. Evaporation. This process consists in removing a solvent from a soluble material. The solvent in most cases is water, and evaporation may be brought about in a number of ways.

(a) *Spontaneous Evaporation.* The liquid is exposed in shallow pans to the direct action of the wind and sun, as, for instance, in the production of common salt from brine.

(b) *Evaporation by Direct Heat.* This takes place when the hot gases from a fire or burner are allowed to play upon the bottom of the containing vessel.

(c) *Evaporation by Indirect Heat.* This process is carried out either by conducting steam through closed coils

contained in a suitable vessel, or by means of the steam-jacketed kettle, where the temperature is regulated by the steam pressure. The steam-jacketed kettle (Fig. 4) is generally used. The solution is placed in the kettle and the drain opened. Then the outlet is also opened, and the inlet valve given a half-turn. As the steam comes in contact with the cold surface, it condenses and passes off through the drain. As soon as live steam appears, the drip is closed and more steam admitted until the desired tem-

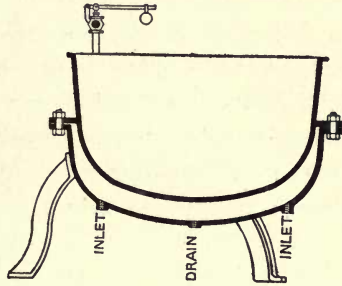


FIG. 4.

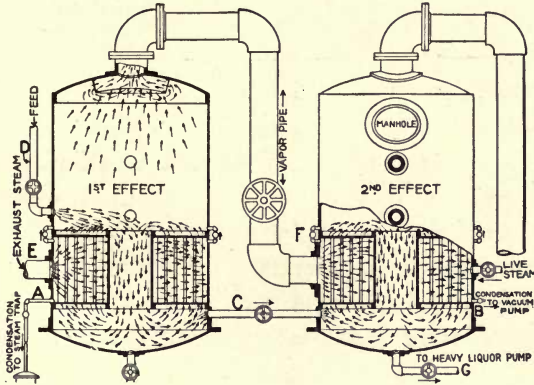


FIG. 5.

perature is reached. The temperature of the steam-jacketed kettle can be raised by partly closing the exhaust.

(d) *Evaporation under Reduced Pressure.* By means

of any of the various forms (Fig. 5) of vacuum pans now on the market, the time necessary for evaporation may be shortened and a saving in heat effected.

8. Roasting. In the manufacture of certain products, an elevated temperature becomes necessary. This is furnished by several forms of furnaces, most common among which are the reverberatory furnace, muffle furnace, revolving furnace (Fig. 6), and kiln. The process is also sometimes called calcination. The object of roasting

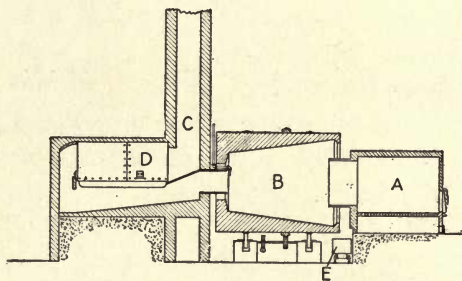


FIG. 6.

or calcination is usually to remove a volatile constituent in the product.

9. Crystallization. In the preparation of chemical salts, crystallization is resorted to in order that the material may be separated in a pure condition. When a salt is dissolved in water, a point is reached where no more of the material will go into solution. If, at this point, the solution is concentrated, until a slight scum appears on the surface, and then allowed to cool, it will be noticed that more or less regular crystalline bodies form as the temperature falls, care having been taken not to disturb the vessel during this time. The crystals thus separated

are usually a very pure form of the salt. By further evaporation or concentration of the mother liquor, more crystals will result, less pure, however, than the first crop. By means of the process of crystallization it is usually possible to separate a mixture of two or more soluble compounds.

10. Filtration. An insoluble substance, suspended in

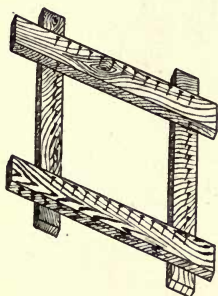


FIG. 7.

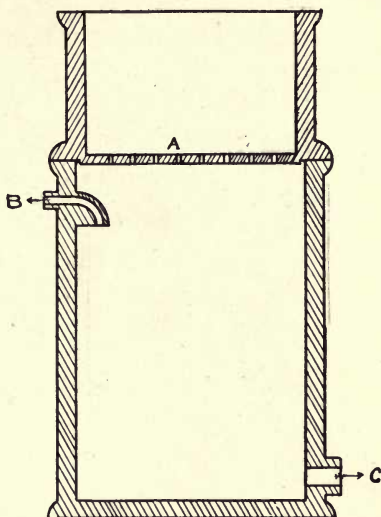


FIG. 8.

a liquid, can be removed by filtration, i.e., by passing the liquid through a more or less porous material. On a very small scale, the ordinary filter paper and funnel may be used, in which case the insoluble portion remains on the paper in the funnel, while the soluble portion passes through. On a larger scale, the same principle operates with the bag filter. This can be readily made by tacking a fairly heavy muslin over a wooden frame (Fig. 7),

the size depending upon the requirements of the operator.

11. Vacuum Filter. For some coarse materials the vacuum filter gives very satisfactory results. In its simplest form, this filter (Fig. 8) comprises a lower vessel arranged for attachment to a vacuum pump, and an upper vessel fitted tightly to the lower portion. The

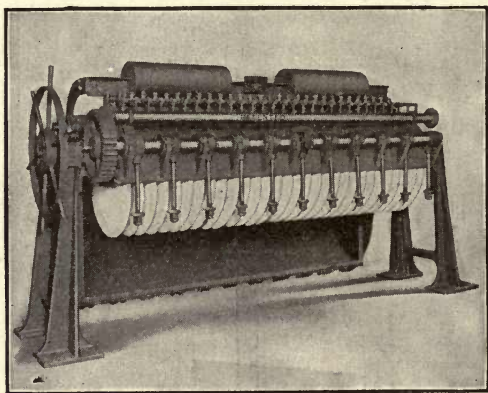


FIG. 9.

upper vessel is provided with a perforated bottom upon which may be spread filter paper, canvas, sand, or asbestos.

12. Filter Press. For handling large volumes of liquid, the filter press method is most common. Many types of filter presses are on the market, all of which work upon practically the same principle. A plate is so arranged that it may be covered with canvas or bolting cloth, and the material to be filtered is forced against this plate under pressure. A simple laboratory press is shown in Fig. 9.

13. Drying. Chemical salts, after crystallization, carry with them more or less of the mother liquor. In removing this, the centrifugal machine has been found to give the best results. This machine (Fig. 10) consists of a basket suspended on a shaft in such a manner that it may be rotated at very high speed. The crystals, being thrown against the walls of the receptacle, are retained,

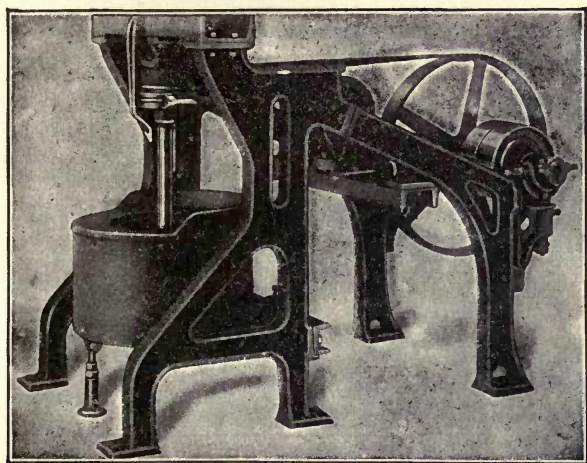


FIG. 10.

while the mother liquor passes through the perforations. For materials that cannot be dried in the centrifuge other machines must be employed. Among these should be mentioned the drying oven and the vacuum dryer. The drying oven is essentially a chamber heated by means of steam; while the vacuum dryer (Fig. 11) is provided with air-tight doors and a vacuum connection.

14. Sublimation. This occurs when a substance passes

from a solid to a vapor and then back again to a solid without passing through the intermediate liquid stage, as for instance, when crude naphthalene is heated in an iron kettle over a low flame, or, better, on asbestos. The naphthalene vaporizes and may be condensed by placing a cone over the top of the kettle, leaving a very small opening in the top for escaping gases. The cone may be made of sheet iron or even of heavy cardboard. A piece

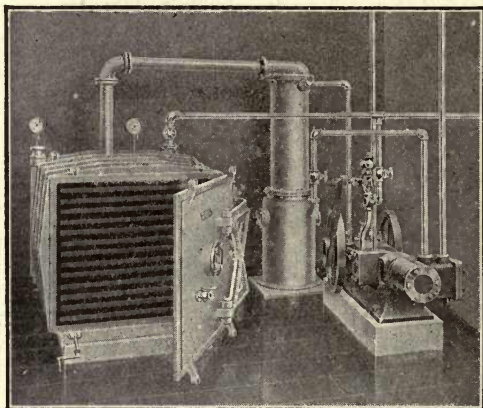


FIG. 11.

of muslin or filter paper may also be stretched over the kettle to serve as a filter and retain any liquid that may be given off.

15. Distillation. There are many forms of apparatus employed for fractional distillation. The one illustrated in Fig. 12 is very satisfactory for laboratory work. This can be obtained from any copper maker. It may be heated by steam-coils, steam-jacket, or by open fire, according to the nature of the product being distilled.

16. Density. By density or specific gravity of a liquid is meant its relative weight as compared to the weight of an equal volume of water at a definite temperature. The determination of specific gravity is one of the most frequent operations in chemical work. For all practical purposes the test may be made with the hydrom-

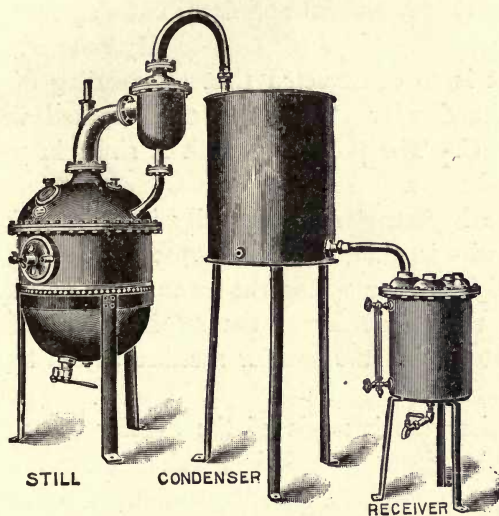


FIG. 12.

eter, but for very exact results the specific gravity bottle (pycnometer), or the Westphal balance is employed.

Three systems of hydrometer scales are in common use, as well as a great many special ones intended for determining the density of a particular substance in a liquid; the principle, however, is the same in each case. These instruments are usually of glass, having a bulb weighted at the lower end, and drawn out at the upper end into a

long slender tube carrying a scale. The graduations on the scale are at the top and read downward, the largest number being at the bottom, except in one instance—that of Baumé's scale for liquids lighter than water, where it is the reverse.

When the hydrometer is placed in a liquid it sinks sufficiently to displace a volume equal in weight to the weight of the instrument and floats in an upright position.

17. The Direct Specific Gravity Hydrometer. This instrument is so constructed that the reading of the scale shows the density of a liquid as compared with water at 15.5° C. The point to which it sinks in pure water is marked 1.000.

These hydrometers are usually in a set of four. The first spindle is graduated from .700 to 1.000 and is for liquids lighter than water, the 1.000 being at the bottom; whereas the remainder of the set is for liquids heavier than water, with the smaller number at the top starting with 1.000.

Practical use is made of the density of solutions for determining the weight of given volumes.

18. Barkometer. This is a special form of direct specific gravity hydrometer in which only the numerals to the right of the decimal are used. Thus, 1.030 specific gravity would be 30° on the Barkometer, and 1.125 specific gravity would be 125° Bk. This instrument is in common use in the tanning industry.

19. Twaddell's Hydrometer. This is also a direct-reading instrument. The set consists of a series of spindles (usually six) having graduations from 0° to 174° . The reading for water at 15.5° is taken as 0° , and each rise of 0.005 in sp. gr. is recorded on the scale as one additional

division. Thus, 10° Twaddell becomes 1.050 sp. gr. The density by this means is recorded as 10° Tw.

Twaddell readings are readily converted into specific gravity in the following manner:

Multiply the reading by .005 and add 1.000 to the product. Thus, 15° Tw. = 1.075 sp. gr.

$$(1.000 + (15 \times .005) = 1.075).$$

In the reverse order, specific gravity may be converted into Twaddell. Thus, 1.150 sp. gr. becomes 30° Tw.

$$((1.150 - 1.000) \div .005 = 30).$$

That is, from the specific gravity subtract 1.000 and divide by .005.

This is the most convenient hydrometer for factory or laboratory use. It is, however, not adapted to liquids lighter than water.

20. Baumé's Hydrometer. This is a very unscientific instrument, but is largely used in technical work. Its readings have no direct relation to specific gravity. Baumé dissolved 15 parts of pure salt in 85 parts of pure water at 12.5° C. The point to which his instrument sank was marked 15; the point to which it sank in pure water was marked 0. The distance between these points was divided into fifteen equal parts. This produced an instrument for liquids heavier than water.

For liquids lighter than water, the point to which the instrument sank in a 10 per cent solution of salt was marked 0, and that to which it sank in water was marked 10; the distance between these points was divided into ten equal parts, and this graduation continued the

entire length of the spindle. Thus the lighter the gravity of the liquid tested, the greater numerically is the reading of the scale. For instance, a liquid reading 70° Bé. is of less density than one at 50° Bé., which, in turn, is lighter than water at 10° Bé.

The conversion of American Standard Baumé readings to specific gravity is usually accomplished by reference to tables, but may be worked out as follows:

$$\text{Sp. gr.} = \frac{144.3}{144.3 - \text{Bé.}} \quad (\text{for liquids heavier than water at } 15^{\circ} \text{ C.})$$

$$\text{Sp. gr.} = \frac{140}{130 - \text{Bé.}} \quad (\text{for liquids lighter than water at } 17.5^{\circ} \text{ C.})$$

There is still another form of Baumé which is in quite common use, the so-called Rational Scale Baumé instrument (Kolb's), which indicates zero in pure water at 15° C., and 66 in sulphuric acid of sp. gr. 1.842 at 15° C.

21. The Pycnometer. This consists of a small bottle, having ground into its neck a capillary stopper provided with a reservoir. The bottle is filled with the liquid to be tested, and the stopper loosely inserted. When the bottle and contents have reached the normal temperature, the excess of liquid is removed by means of filter paper until the level of the liquid reaches the graduation mark. The stopper is tightly inserted, and, after cleaning and drying, the instrument is weighed.

The density of the liquid is ascertained by the following:

$$\text{Sp. gr.} = \frac{\text{Weight of bottle and liquid} - \text{weight of bottle}}{\text{Weight of bottle and water} - \text{weight of bottle}}$$

Or,

$$\text{Sp. gr.} = \frac{\text{Weight of liquid}}{\text{Weight of equal volume of water}}$$

22. Westphal Balance. This is a special form of balance for determining the density of liquids. A glass plummet of known weight and volume is suspended from the beam by a fine platinum wire, and is submerged in the liquid to be tested. The weight which the plummet loses is the weight of the liquid displaced. This loss can be determined by using a set of riders graduated to read directly into specific gravity.

In all determinations of density, the question of temperature is an important factor, and all corrections should be made accordingly. When using the Twaddell hydrometer for commercial purposes, however, the addition of 1° Tw. for each 20° F. above 60° will suffice.

The foregoing general processes are given in order that the student may have some idea of them previous to their use in his subsequent work. For a more detailed description of the apparatus and methods employed, he is referred to the Manual of Industrial Chemistry, or to the Elements of Industrial Chemistry, by the same author.

CHAPTER II

INORGANIC PREPARATIONS

23. Aluminium Sulphate ($\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$). This compound is prepared from the mineral bauxite ($\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$). On a small scale porcelain evaporating dishes may be used, but on a larger, laboratory scale, earthenware crocks are most satisfactory. They may be heated by means of live steam led into them through heavy glass tubes connected with the steam line.

Weigh out 1000 gms. of bauxite, previously ground to a fine powder, and mix it to a paste with water. To the paste add slowly and with constant stirring 2300 gms. of 66° Bé. sulphuric acid. Turn on the steam and boil continuously until all frothing ceases and the mass is in a state of quiet ebullition. To complete the reaction and insure the elimination of free sulphuric acid, a small amount of powdered bauxite is cautiously introduced from time to time until no more frothing occurs. Add 10 volumes of water to the paste and bring again to a boil. During the process of boiling, any iron that may be present is in the ferric condition and must be reduced to the ferrous state. This is accomplished by adding powdered zinc equal in amount to 0.5 per cent of the weight of the bauxite. At this stage of the operation considerable suspended matter is present, which is allowed to settle by standing overnight. When the solution is clear, decant it into a lead-lined evaporating pan or earthen-

ware dish, concentrate to a density of 40° Tw., and allow to cool. Any ferrous sulphate present will crystallize, and, after removing it, concentrate the liquor again until a test sample solidifies upon cooling. It is then allowed to cool and the solid mass broken into lumps.

Write the reactions involved.

Calculate the theoretical yield.

Weigh the product obtained.

Estimate the percentage yield.

Estimate the manufacturing cost.

In all manufacturing operations, the main object is to make a profit on the goods. It is necessary, therefore, to have a pretty clear idea of the cost of production. The following example will serve as a guide to this and other problems.

(In making estimates, use grams as if they were pounds.)

Bauxite, 1000 lbs. at $\frac{1}{2}$ ¢.....	\$ 5.00
Sulphuric acid, 2300 lbs. at 1 ¢.....	23.00
Incoming freight, 3300 lbs. at 6 ¢. per 100	1.98
Outgoing freight, 4970 lbs. (theoretical yield) at 6 ¢. per 100.....	2.98
Containers, 4970 lbs. at 15 ¢. per 100-lb. unit.....	7.50
	\$40.46
Fixed and overhead charges, 20 per cent of cost of production.....	8.09
Total cost of production.....	\$48.55
Cost per pound, \$48.55 ÷ 4970 lbs.....	0.98

24. Potash Alum ($K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$). This compound is produced by dissolving the molecular equiv-

alents of aluminium sulphate and potassium sulphate in the necessary quantity of water and concentrating the resulting solution until crystals appear on the edge of the evaporating dish. The liquor is then transferred to a crystallizing tank and not disturbed until a full crop of crystals has been obtained. After the removal the crystals, the mother liquor may again be concentrated for a further yield.

For crystallizing on a small scale, vessels of glass may be used, but, on a larger scale, square earthenware tanks are best fitted for the purpose. On a commercial scale, lead-lined wooden tanks are preferred. To aid crystallization, strips of lead attached to wooden sticks may be suspended in the tanks.

Write the reactions involved.

Estimate the theoretical yield.

Weigh the resulting product.

Calculate the percentage yield.

Estimate the cost on theoretical yield.

Estimate the cost on actual yield.

25. Barium Chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$). This chemical is prepared on a commercial scale from barytes (BaSO_4). The mineral is ground to a fine powder and heated with carbon to reduce it to the sulphide. The lixiviated mass is treated with sodium carbonate to convert it into the carbonate, and then with hydrochloric acid to change this carbonate into the chloride.

The necessary apparatus comprises a furnace, tanks, vacuum filter, evaporating dishes, crystallizing tanks, and a centrifugal machine.

Intimately mix 1000 gms. of powdered barytes with 300 gms. of powdered charcoal and transfer the mixture

to the floor of a muffle furnace. Heat the muffle and contents to a bright red and hold the temperature at this point until a test portion shows that practically all of the sulphate has been reduced. To derive the full benefit from the reducing action of the carbon, it is necessary, during the heating, to prevent the charge from coming into contact with the air. The door, however, must be opened from time to time and the mass well stirred.

On completion of the heating, the mass is allowed to cool and is then transferred to a 10-gallon earthenware vessel containing 5 gallons of water. Live steam is now led into the jar and the contents boiled for about one hour. After standing overnight to allow insoluble matter to settle, the clear solution is decanted into another 10-gallon jar or precipitating tank, and sufficient sodium carbonate in solution added to throw down the barium as carbonate. In order to ascertain the exact amount of sodium carbonate to add, a rapid determination of barium as sulphate should be made. The precipitated barium carbonate is allowed to settle, and the clear solution, containing sodium sulphide, is evaporated to dryness in order to obtain this chemical as a by-product.

The barium carbonate is washed by decantation until free from sulphide and then dissolved by the addition of the calculated amount of hydrochloric acid diluted with three parts of water. To the solution of barium chloride a small excess of barium carbonate is added, and the whole boiled for about half an hour to throw out any impurity of iron that may be present. After again settling, the clear solution is decanted to an evaporating dish and concentrated until crystals appear on the edges of the

dish, when it is transferred to the crystallizing tank. The residue from the above is thrown on to the vacuum filter, and the filtrate added to the solution being concentrated.

The barium chloride, on removal from the tank, is placed in the centrifugal machine and whizzed for five minutes to remove adhering liquor. The mother liquor is further concentrated for another yield.

Write the reactions involved.

Estimate the theoretical yield.

Weigh the resulting product.

Calculate the percentage yield.

Estimate the cost on theoretical yield.

Estimate the cost on actual yield.

26. Chromium Acetate ($\text{Cr}_2(\text{C}_2\text{H}_3\text{O}_2)_6$). To 1000 gms. of sodium dichromate, dissolved in 10 liters of water, add 500 gms. of concentrated sulphuric and then, very slowly, a concentrated solution of 1100 gms. of sodium bisulphite. The action of the bisulphite is to change the chromium from the acid to the basic condition, the end point being determined when the yellow color changes to green. The product of this reaction is the basic sulphate of chromium ($\text{Cr}_2(\text{OH})_2(\text{SO}_4)_2$). Next dissolve 800 gms. of soda ash in as little water as possible and add, a small portion at a time, to the chromium solution. The chromium hydroxide formed is allowed to settle, and the clear supernatant liquid decanted and evaporated for the yield of sodium sulphate as a by-product.

The chromium hydroxide, washed free from sulphates by decantation, is treated with the calculated quantity of commercial acetic acid and evaporated to a concentration of 90°Tw .

Write the reactions involved.

Estimate the theoretical yield of pure salt.

Weigh the resulting product.

Calculate actual percentage yield.

Estimate the cost of manufacture.

27. Sodium Dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$). Mix intimately 980 gms. of lime, 960 gms. of soda ash, and 1000 gms. of very finely powdered chromite. Convey the mixture to the floor of a reverberatory furnace and heat with the oxidizing flame at a bright red heat for a period of from three to four hours. The resulting mass, when cooled, is pulverized and extracted with hot water until practically no more color is shown in the wash water. The solution and washings are then concentrated to 35° Bé. To this hot concentrated solution, sulphuric acid is added until the starch iodine paper test shows the presence of free chromic acid. The solution is then further concentrated until crystals of sodium sulphate separate. These are removed by rapid filtration in the centrifugal machine and washed with a small quantity of water. The mother liquor and wash water from the sodium sulphate is then slowly evaporated with constant stirring. The crystals of sodium dichromate obtained are whizzed to remove adhering liquor.

Write reactions involved.

Estimate theoretical yield.

Weigh the resulting product.

Calculate the percentage yield.

Estimate cost of theoretical yield.

Estimate the cost of actual yield.

28. Hydrogen Peroxide (H_2O_2). Mix 500 gms. of barium peroxide to a thin cream with water and slowly

add the calculated quantity of phosphoric acid. The temperature should not rise above 15° C. When all of the barium has been precipitated as phosphates, it is allowed to settle and the solution of H_2O_2 removed by decantation.

29. Ferrous Sulphate ($FeSO_4 \cdot 7H_2O$). This chemical, on a commercial scale, is usually manufactured in connection with foundry practice. It is obtained from the pickle liquor used for cleaning castings. In the laboratory, the most satisfactory method is to use a wooden tub or oak barrel, placing therein a quantity of scrap iron or iron turnings. Cover the iron with water and add a small quantity of commercial sulphuric acid, diluting from time to time with more water when crystallization is indicated. When the liquor is sufficiently concentrated, it is removed, filtered, and transferred to a shallow crystallizing vessel. To aid crystallization, suspend narrow strips of clean iron in the solution. Immediately upon separation from solution, the crystals should be dried in the centrifuge, otherwise rapid oxidation will take place.

Write the reactions involved and test the purity of the product by chemical methods.

30. Ferrous Ammonium Sulphate ($FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$). Dissolve 1400 gms. of ferrous sulphate and 664 gms. of ammonium sulphate in 2000 cc. of water separately, heating to 60° C. Filter, if necessary. Mix the two solutions, concentrate to 36° Bé., add 36 gms. of sulphuric acid, and allow to crystallize. If, on cooling, crystals do not form, concentrate the liquor until a scum appears on the edge of the evaporating dish. Cool without disturbing; when crystals form, remove them, and dry in the centrifugal machine. The mother liquor from

the first crop of crystals is evaporated for a further yield.

Test the purity of the product so obtained.

31. Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$). Place in a large evaporating dish about 1600 cc. of water, add 330 cc. of concentrated sulphuric acid, and heat to about 90°C . To the diluted acid add 320 cc. of strong nitric acid and stir well. Now pulverize 3200 gms. of ferrous sulphate and add this slowly, a small portion at a time, to the hot mixed acid. After each addition of ferrous sulphate, stir the solution well and add the subsequent portions only after effervescence has ceased. When all of the ferrous sulphate has been used up and no more fumes appear, a small portion should be tested by adding a small quantity of nitric acid. If, by an evolution of red fumes, the test portion indicates that oxidation has not been complete, more acid should be added to the reaction product, until the evolution of fumes ceases. The solution thus formed is heated to boiling until all fumes of nitric oxide have been eliminated and the solution has assumed a deep, reddish brown color. Concentrate the liquor to the density of 130°Tw . at normal temperature. Preserve this liquid in well-stoppered bottles.

Write reactions involved.

Test strength of material by analytical means.

Estimate cost of finished product.

32. Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$). Place 1000 gms. of litharge in an earthenware evaporating dish and add sufficient water to form a thin cream. To this cream, add the calculated amount of nitric acid and heat to boiling. When all of the litharge has dissolved, filter off the insoluble matter and concentrate on a steam-bath

until crystals appear at the edge. Remove the dish from the steam-bath and set aside overnight, being careful not to disturb the vessel after crystals start to form. Remove the crystals and evaporate the mother liquor for a further yield.

Write the reactions involved.

Calculate the theoretical yield.

Determine actual yield.

Test salt for purity.

33. Magnesium Sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$). Having determined the presence of magnesium in magnesite and the strength of the sulphuric acid, estimate the quantity of each necessary to produce 4000 gms. of salt.

Write equations and calculations.

Mix the magnesite with about twice its quantity by weight of water; slowly add the sulphuric acid, and heat on a steam-bath until action ceases. If acid is in excess neutralize with powdered magnesite, heat the contents of the vessel for about an hour, then allow the suspended matter to settle, and filter while still hot. Evaporate the filtrate on a steam-bath until crystals appear along the edges, then allow to remain in an undisturbed condition to cool and crystallize. The crystals, on removal, should be dried in the centrifuge, and the mother liquor evaporated for a further yield.

Estimate theoretical yield.

Determine the actual yield.

Estimate cost per pound.

34. Potassium Permanganate (KMnO_4). Ignite a quantity of powdered pyrolusite at red heat on an iron plate, to burn out any carbonaceous matter it may contain. Dissolve 840 gms. of caustic potash in 1200 cc. of water, stir in 420 gms. of potassium chlorate, and heat until

dissolved. Then mix in 720 gms. of the ignited pyrolusite, evaporate to a smooth, thick paste, and heat to redness in an iron crucible, adding small portions at a time. When thoroughly hard, let the crucible cool, and break out the mass with a chisel. Then boil with a large excess of water, passing a stream of carbon dioxide into the boiling solution to convert any hydroxide into carbonate. The color of the liquid changes from green to violet and a dense brown precipitate forms. Filter through asbestos or glass wool and evaporate the filtrate until needle-like crystals appear on the surface. Allow to cool. Separate the crystals and evaporate the mother liquor for further yield.

35. Sodium Silicate or Water Glass (Na_2SiO_3). This compound is prepared by heating a mixture of sand and sodium carbonate in a fire-clay retort or iron crucible until the product, when drawn from the heating vessel, gives the appearance of a transparent solid. If the test sample, when cool, gives no effervescence with dilute acid, the completion of the process is indicated, and the contents of the crucible are then allowed to cool. Dissolve in water, filter, and evaporate to a syrupy consistency.

Write reactions involved.

Determine practical yield.

36. Soda Ash (Na_2CO_3). Prepare a saturated brine solution by dissolving 880 gms. of salt in 3000 cc. of water at 15°C . To this brine add 1000 cc. of ammonia (sp. gr. .910) and pass in carbon dioxide as long as the gas is absorbed and a precipitate formed. Care should be taken to keep the vessel cool and to use a large delivery tube in order to prevent clogging.

The precipitate produced is sodium bicarbonate and

should be filtered by suction and washed with a little cold water to remove adhering salt. It is then dried and heated strongly (do not fuse) to convert the bicarbonate to the carbonate.

the ammonia. This ammonia may be absorbed in brine

The liquor may be heated with milk of lime to recover and the process repeated.

37. Disodium Hydrogen Phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$). Into a lead-lined tank or procelain dish introduce 5000 gms. of bone-ash and mix it with 3880 gms. of sulphuric acid (sp. gr. 1.80). The mixing is conducted in the open air or hood. Stir well and allow to stand overnight. Lixivate the mass with twice its volume of water and boil by blowing in steam. Filter off the soluble portion and wash the residue several times with fresh water. Evaporate the filtrate and washings to a convenient bulk and neutralize the boiling solution with a concentrated solution of 2750 gms. of soda ash. Filter off the precipitate of calcium carbonate and evaporate the filtrate until the solution has a density of 25° Tw. at 60° F. The crystals, which separate on cooling, should be dried in the centrifugal and bottled as soon as possible.

Write reactions.

Estimate theoretical yield.

Determine practical yield.

38. Sodium Bisulphite (NaHSO_3). This compound is usually placed on the market as a liquid containing 33.3 per cent of the salt. It is prepared by passing sulphur dioxide into a solution of sodium carbonate until completely saturated. The SO_2 may be easily obtained by burning sulphur in a small furnace (an ordinary muffle furnace with a hole bored in it) and drawing the gas by

suction through the soda solution. The liquid may be placed in flasks or small casks.

Estimate the quantity of soda ash, water, and sulphur required to produce 10,000 gms. of the product, and also the cost per pound. Write the reactions.

39. Sodium Sulphite ($\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$). This compound is prepared in the same manner as sodium bisulphite, except that the solution resulting from the saturation with sulphur dioxide of the sodium carbonate solution is converted into the normal salt by adding the same amount of sodium carbonate to produce the original sodium bisulphite solution and then evaporating on a steam-bath until a scum appears. It is then set aside to crystallize, the mother liquor being evaporated for a further yield.

Write the reactions involved.

Calculate the theoretical yield.

Determine the practical yield.

40. Sodium Thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). Dissolve 1000 gms. of sodium sulphite in 5 liters of water. Place in an earthenware vessel provided with a live-steam connection. To the solution add an excess of flowers of sulphur and boil until no more sulphur is dissolved. Filter and evaporate to the point of crystallization. Separate the crystals and concentrate the mother liquor for a further yield.

Write the reactions involved.

Estimate the theoretical yield.

Determine the practical yield.

41. Sulphur Monochloride (S_2Cl_2). Into a 1000-cc. tubular retort place 500 gms. of sulphur and connect the retort with a Liebig condenser and receiver. Heat the sulphur until melted and pass in a current of chlorine

in such a manner that the delivery tube comes just to the top of the melted sulphur. The chlorine may be generated by means of manganese dioxide and hydrochloric acid, or by the electrolysis of a salt solution. If manganese dioxide and hydrochloric acid are used, the solution of manganese chloride should be concentrated to recover the manganese chloride as a by-product. In the preparation of sulphur monochloride, all vessels should be perfectly dry; also the chlorine gas should be dried by passing it over sulphuric acid.

Write the reactions involved.

Calculate the theoretical yield.

Determine the actual yield.

42. Ammonium Paratungstate $((\text{NH}_4)_6\text{W}_7\text{O}_{24}\cdot 6\text{H}_2\text{O})$.

The finely ground mineral, Wolframite, is mixed to a thin cream with nitric acid and digested on a steam-bath for several hours. The product of the reaction is then evaporated to dryness and the resulting solid treated with ammonium hydroxide. When all of the yellow tungstic oxide has dissolved, the insoluble residue is filtered off and the filtrate evaporated until crystallization is indicated. The solution is then set aside overnight. The white crystals of ammonium paratungstate being separated, the mother liquor is concentrated for a further yield.

Write reactions involved.

Determine the practical yield.

43. Zinc Sulphate $(\text{ZnSO}_4\cdot 7\text{H}_2\text{O})$. Having determined the strength of the sulphuric acid and zinc oxide, estimate the quantity necessary to produce 5000 gms. of zinc sulphate and work out the method of procedure.

CHAPTER III

ORGANIC PREPARATIONS

IN all of the following preparations the student is required to write the reactions involved, estimate the theoretical yield, determine the percentage yield, and test the product for purity.

44. Alcohol (C_2H_5OH). Dissolve 500 gms. of glucose syrup in 3 liters of water at $33^\circ C.$, or $90^\circ F.$, and add a yeast cake that has been made into a cream with water. Place the flask in a warm place and allow it to stand for three days, or until fermentation is complete. Then heat the contents to boiling and distill off about 25 per cent. Determine the specific gravity of the distillate, refer to table, and subject to redistillation on a sand-bath, repeating the operation, if necessary, until a 95 per cent alcohol is obtained.

45. Ethyl Bromide (C_2H_5Br). Into a 1-liter, round-bottom distilling flask (Fig. 13) introduce 20 gms. of amorphous phosphorus and 120 gms. of absolute alcohol. From a tap funnel slowly add 120 gms. of bromine, keeping the flask cool by immersion in cold water. Allow the flask and contents to rest for two hours and then heat gently on the water-bath at a slowly rising temperature. Shake the distillate, consisting of alcohol and ethyl bromide, with double its volume of water, to which a small amount of sodium carbonate has been added. The ethyl bromide,

which sinks to the bottom, is drawn off and shaken several times with a fresh quantity of water. It is then completely separated from the water, dehydrated over calcium chloride, and distilled on the water-bath. That portion passing over between 36° – 40° C. is pure ethyl bromide.

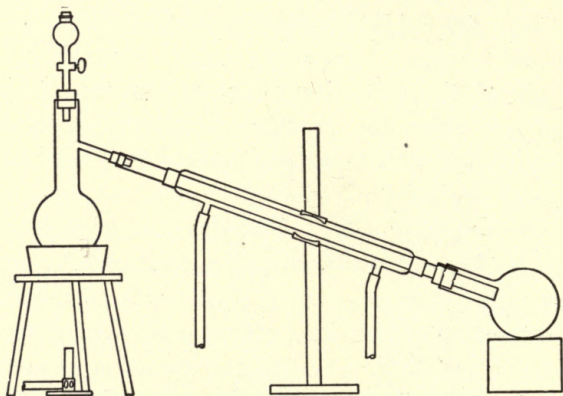


FIG. 13.

46. Ether ($(C_2H_5)_2O$). Ether is extremely inflammable. Take care that no flame is brought near it. The reagents used are

150 gms. (80 cc.) conc. sulphuric acid,
85 gms. (110 cc.) absolute alcohol.

A distilling flask ($\frac{1}{2}$ -liter) is equipped with a thermometer with bulb below liquid (Fig. 14), and a separatory funnel. The side tube of the distilling flask is fitted by a cork into the upper end of a long condenser; and an adapter at the lower end passes through the neck of a flask surrounded by ice. The sulphuric acid and alcohol

are cautiously mixed in the distilling flask, which is then placed upon a sand-bath and attached to the condenser. The mixture is heated at 140° C. and more alcohol run in from the funnel at the same speed the liquid distills (about 3 drops a second). The temperature must be kept constant, 140° – 145° C. When about 150 cc. of alcohol has been added and converted into ether, the distillation is stopped. In addition to ether, the receiver now contains alcohol, water, and sul-

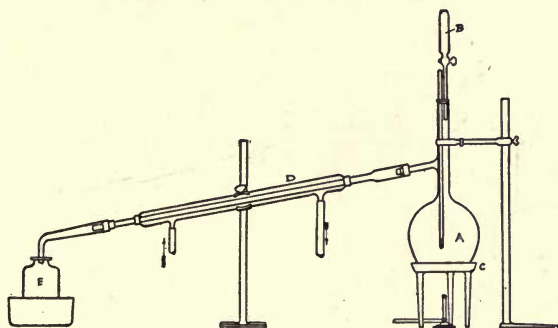


FIG. 14.

phurous acid. The liquid is poured into a large separating funnel and a small quantity (30–40 cc.) of dilute caustic soda added and well shaken. After settling, the caustic soda solution is drawn off below, about the same quantity of a strong solution of common salt added, and the process of shaking and drawing off repeated. The ether, which is now free from sulphuric acid and from most of the alcohol, still contains water. It is, therefore, poured into a large dry distilling flask and some pieces of solid calcium chloride added. It is allowed to stand loosely corked overnight.

The distilling flask is finally attached to a long condenser and heated on the water-bath, the ether passing over at about 40° C.

47. Acetone (CH_3COCH_3). Place 200 gms. of calcium acetate, known commercially as *gray lime*, in an iron retort (Fig. 15), connected to a Liebig condenser. Heat the retort and contents over a strong flame until no more distillate is obtained. The product of the distillation is impure and must be purified by treatment with a small amount of oxalic acid and redistilled. That portion

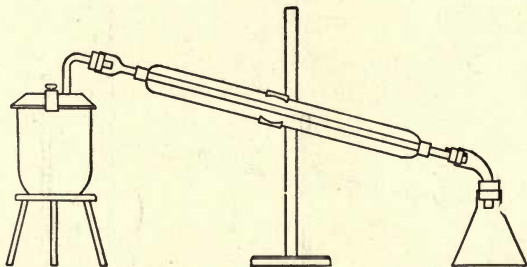


FIG. 15.

passing over at 56° C. is collected separately as pure acetone.

48. Iodoform (CHI_3). Dissolve 25 gms. of potassium iodide in 500 cc. of water and add 5 gms. of acetone. To this mixture add through a drop funnel with constant shaking a dilute solution of sodium hypochlorite as long as a precipitate forms. Allow the precipitate to settle. Decant off the liquid, wash with water two or three times, filter, drain thoroughly, dry on filter paper, and recrystallize from alcohol. The solution of sodium hypochlorite used in this experiment may be made by precipitating all the calcium in a solution of bleaching powder by a

solution of sodium carbonate. A slight excess of sodium carbonate will not interfere with the reaction.

49. Chloroform (CHCl_3). (*Trichlormethane*). A large round flask (4 liters) is fitted with a cork, through which passes a bent tube connecting the flask with a long condenser and receiver. The flask is placed upon a large sand-bath. Grind 200 gms. of fresh bleaching powder into a paste with 400 cc. of water and rinse it into the flask with 400 cc. more. Add 40 gms. (50 cc.) of acetone and attach the flask to the condenser. Heat cautiously until a reaction sets in, indicated by the frothing of the liquid. Remove the flame for a time, until the reaction has moderated; finally boil the contents until no more chloroform distills. This is easily determined by collecting the distillate in a test tube and observing if any drops of heavy liquid are present. The distillate is shaken with dilute caustic soda solution in a separating funnel, and the lower layer of chloroform run into a distilling flask. A few pieces of solid calcium chloride are added and left until the liquid is clear, when it is distilled from the water-bath with the thermometer inserted into the neck of the flask. The portion passing over at 62°C . is pure chloroform.

50. Acetic Acid (CH_3COOH). Dissolve 200 gms. of calcium acetate in the least possible quantity of water, using a 1500-cc. round-bottom flask. Add the necessary quantity of commercial hydrochloric acid to combine with the calcium present and distill on a sand-bath, connecting the flask with a Liebig condenser as in Fig. 16. Redistill the distillate and collect separately that portion passing over at about 118°C .

51. Acetyl Chloride (CH_3COCl). To 100 gms. of glacial acetic acid in a 250-cc. distilling flask add, by

means of a dropping funnel, 80 gms. of phosphorus trichloride. The distilling flask must be connected with a return condenser and be immersed in ice water. Shake

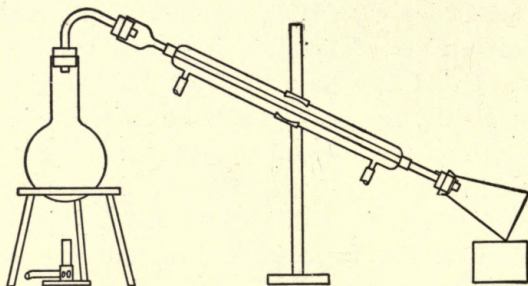


FIG. 16.

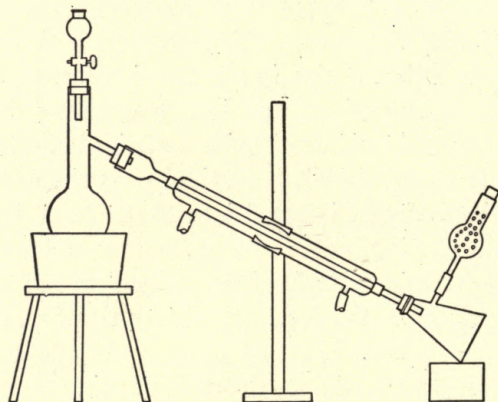


FIG. 17.

the flask constantly while the phosphorus trichloride is being added. After ten minutes the ice-water may be replaced with warm water and heated until almost all the hydrochloric acid gas has been evolved. The con-

denser may then be turned down (Fig. 17) and the acetyl chloride rapidly distilled off. The acetyl chloride is very easily decomposed by moisture, therefore the receiver must be protected by a calcium chloride tube. The acetyl chloride boiling at 55°C . may be purified by re-distillation.

52. Acetic Anhydride $((\text{CH}_3\text{CO})_2\text{O})$. Place 140 gms. of fused sodium acetate in a tubular retort, as illustrated in Fig. 18, and slowly add through a tap funnel 50 gms. of acetyl chloride. Thoroughly mix the mass with a rod

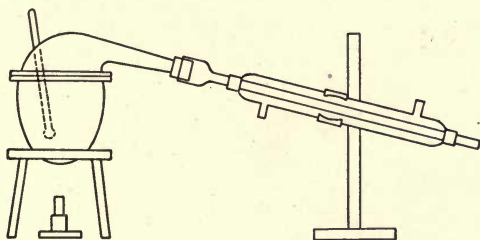


FIG. 18.

and introduce another 50 cc. Heat slowly on a sand-bath until all of the anhydride has passed over. Then purify the distillate by redistillation in an apparatus like that shown under acetyl chloride. Add about 10 gms. of fused sodium acetate to the contents of the flask so as to take up any uncombined acetyl chloride. The portion distilling at a temperature of about 138°C . is pure acetic anhydride.

53. Amyl Acetate $(\text{CH}_3\text{COOC}_5\text{H}_{11})$. Into a 1-liter, side-neck distilling flask place 200 gms. of fused sodium acetate and to it slowly add a mixture of 215 gms. of amyl alcohol with 250 gms. of sulphuric acid. Then con-

nect the distilling flask with a condenser and heat the contents on the sand-bath until no more distillate is obtained. The impure amyl acetate being transferred to a separating funnel is washed with water and then with dilute sodium carbonate, dehydrated, and redistilled. The portion passing over between 145° – 155° C. is pure amyl acetate.

54. Monochloroacetic Acid (CH_2ClCOOH). Place 200 gms. of glacial acetic acid in a half-liter retort, provided with reflux condenser. Heat to boiling over a wire gauze and pass in dry chlorine gas through a tube that just dips below the surface of the acid. This operation should take place in direct sunlight and will require about five hours for complete conversion. Provision should be made for absorbing the excess of chlorine, and this is accomplished by connecting the end of the condenser with a drying tower containing moist soda lime. When the reaction is complete, the resulting product is distilled with thermometer dipping in the liquid. That portion passing off up to 130° C. consists of unchanged acetic acid and is rejected. That portion boiling between 130° – 190° C. consists of practically pure monochloroacetic acid, which quickly solidifies on cooling. The colorless, crystalline plates are separated at the filter pump and redistilled. The portion passing over between 185° – 190° C. is collected.

55. Acetamide (CH_3CONH_2). Half fill a number of heavy-walled pressure tubes with ammonium acetate. After properly sealing, place in a bomb furnace and heat for five hours at a temperature of 230° C. During the heating, care should be taken not to open the furnace or even look in at the tubes. After heating the tubes for five hours, the furnace is allowed to cool, and the tubes

removed, carefully opened, and distilled, using an air condenser. That portion passing over at above 180° C. is nearly pure acetamide, which on cooling gives almost colorless rhombohedral crystals.

56. Ethylene Bromide ($C_2H_4Br_2$). A mixture of 50 gms. of absolute alcohol and 300 gms. of concentrated sulphuric acid is heated in a 2-liter round-bottom flask on a sand-bath until a steady stream of ethylene is evolved. A mixture of 1 part of alcohol with 2 parts of sulphuric

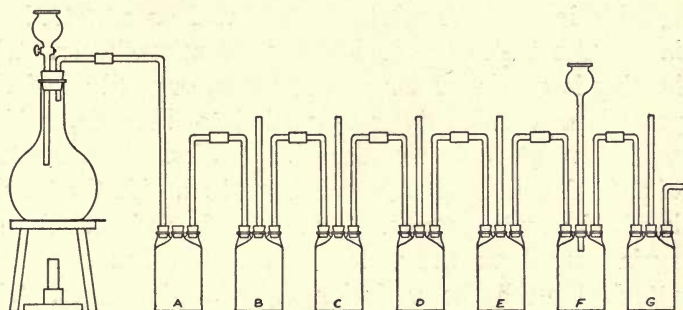


FIG. 19.

acid is then run into the flask through a tap funnel at such a rate that a stream of ethylene is produced, care being taken that the flask does not froth too violently. The gas thus generated is purified by passing it through a series of wash bottles (Fig. 19), the first of which is empty; the second contains concentrated sulphuric acid; while the third and fourth are provided with a solution of caustic soda. The purified gas is now conducted through two Woulff bottles containing bromine covered with a layer of water. The last bottle in the series holds broken glass and soda-lime. As soon as the bromine takes on a

pale yellow color, it is removed, and more bromine introduced. After the desired quantity of crude ethylene bromide has been obtained, the evolution of ethylene is interrupted and the product washed with water containing caustic soda, then with water alone, and finally dehydrated over calcium chloride and distilled. That portion boiling at about 130° C. is collected as pure ethylene bromide.

57. Succinic Acid ($\text{COOHCH}_2\text{CH}_2\text{COOH}$). Two hundred gms. of ethylene bromide and 150 gms. of potassium cyanide in alcoholic solution are heated on a water-bath in a 2-liter flask connected with a reflux condenser. The heating is continued for several hours, or until all of the potassium cyanide is converted into bromide, which separates out from the alcoholic solution. The crystals of potassium bromide are then filtered off. To the filtrate is added about 150 gms. of caustic soda and the mixture again heated on the water-bath, with reflux condenser attached, until the evolution of ammonia ceases. The contents of the flask, on cooling, are acidified with dilute hydrochloric acid and evaporated to dryness on the water-bath. The dry powder is next treated with absolute alcohol, which dissolves the succinic acid and is separated from it on the water-bath by distillation. The slightly colored product remaining in the flask is recrystallized from hot water to which a small amount of animal charcoal has been added. As the filtrate cools, colorless, prismatic crystals separate.

58. Monobrombenzene ($\text{C}_6\text{H}_5\text{Br}$). Two hundred gms. of benzene and 105 gms. of bromine are mixed together in a round-bottom flask provided with reflux condenser (Fig. 20), further provision being made to absorb

the escaping gases in a tower containing soda-lime. The mixture is gently heated over a wire gauze until the reaction is complete. The contents of the flask are aspirated with dry air to drive off the remaining hydrobromic acid, and the unattacked benzene distilled off on a water-bath. The residue from the benzene is now washed in a separatory funnel with water containing a small amount of caustic soda. The heavy liquid is carefully removed from the separatory funnel, dehydrated

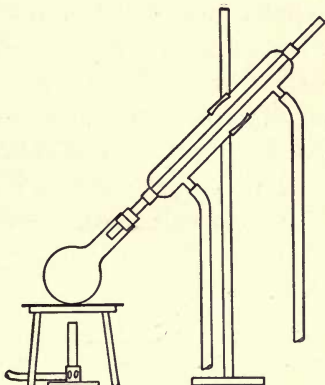


FIG. 20.

over fused calcium chloride, and distilled. That portion passing over between 150° – 155° C. is nearly pure monobromobenzene.

59. Sodium Benzene Sulphonate ($C_6H_5SO_3Na$). A mixture of 200 gms. of benzene and 400 gms. of concentrated sulphuric acid is heated at a gentle boil for twenty-four hours in a 1-liter round-bottom flask, connected with a reflux condenser. At the end of this time, about four-fifths of the benzene should have disappeared, and the lower portion should assume a deep black color. The unattacked benzene is removed by means of a separatory funnel, and the residue thrown into 2 liters of water. The liquid is then neutralized with milk of lime, and the precipitate, calcium sulphate, removed by means of a bag filter. The solution of calcium benzene sulphonate is concentrated to a small volume, when, on cooling, almost a solid mass of crystals is obtained. The salt is separated

from the mother liquor, dissolved in hot water, and exactly neutralized with a concentrated solution of sodium carbonate. The solution of sodium benzene sulphonate thus resulting is filtered from the calcium carbonate and concentrated until crystallization is noticed. The crystals are separated and dried on porous plates, the mother liquor being concentrated for further yield.

60. Phenol (C_6H_5OH). One hundred and fifty gms. of caustic soda is dissolved in the smallest possible quantity of water and heated in an iron crucible on a sand-bath. The temperature is raised to about $300^\circ C.$ and 100 gms. of sodium benzene sulphonate slowly added, the mixture being constantly stirred. The mass at first becomes thick, then finally semi-fluid. The end of the reaction is indicated when the color becomes somewhat lighter. The contents are then cooled, dissolved in water, and neutralized with dilute hydrochloric acid. The phenol separates as a light yellow oil and is removed by means of a separatory funnel. On distillation, the product passing over between $175^\circ-185^\circ$ is nearly pure phenol.

61. Para Cresol ($C_6H_4 \begin{matrix} \text{CH}_3 \\ \text{OH} \end{matrix}$). Dissolve 50 gms. of concentrated sulphuric acid in 1500 cc. of water and add to this 50 gms. of paratoluidine. Cool the solution to room temperature and slowly add 40 gms. of sodium nitrite dissolved in 100 cc. of water. The clear solution is gently heated on the water-bath until the evolution of nitrogen ceases. The dark-colored solution is now steam-distilled until a slight cloudiness appears, when bromine water is added to a portion of the distillate. The entire distillate is then extracted several times with small quantities of ether, and the ethereal solution dehydrated over

plaster of Paris. After filtering off the precipitate, the solution is heated on a water-bath to remove the ether, and the remaining liquid distilled over a naked flame, using an air condenser. The portion passing over between 195° – 200° C. is practically pure para cresol.

62. Benzyl Chloride ($C_6H_5CH_2Cl$). Boil gently 200 gms. of toluene in a weighed $\frac{1}{2}$ -liter retort, provided with reflux condenser (Fig. 21). Into the boiling liquid conduct a stream of chlorine, preferably in direct sunlight, until the

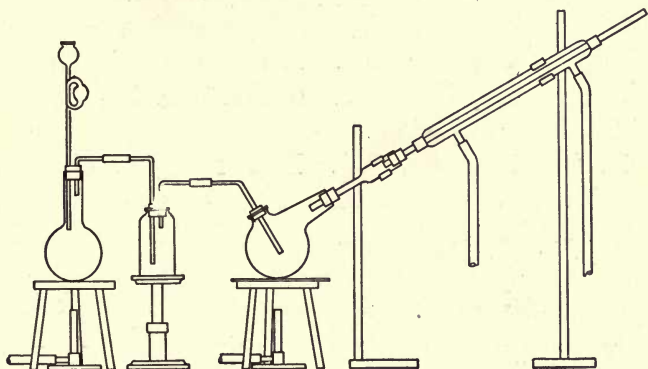


FIG. 21.

retort has gained 75 gms. in weight. When the reaction is complete distill the contents of the retort and collect separately the fraction passing over between 165° – 185° C. On redistilling this product, only that portion between 176° – 180° C. is collected as nearly pure benzyl chloride.

63. Benzaldehyde (C_6H_5COH). A mixture of 50 gms. of benzyl chloride, 40 gms. of copper nitrate, and 500 gms. of water is heated to boiling for a day (eight or nine hours) on the sand-bath, in a round-bottom flask with upright condenser. A slow current of carbon dioxide is

at the same time passed through the liquid to prevent oxidation of the benzaldehyde by absorption of oxygen from the air. During the process nitrous fumes are slowly evolved. When the reaction is complete, the contents of the flask are extracted with ether, and the yellow oil remaining after distilling off the ether is shaken with a saturated solution of sodium bisulphite and allowed to stand for some time. The colorless, crystalline mass which separates out is filtered, washed with a little alcohol and ether, and then drained in a porcelain filter. The aldehyde is regained by adding dilute sulphuric acid in excess and distilling with steam. The distillate is extracted with ether, dehydrated over calcium chloride, decanted, and the ether distilled off.

64. Anisol ($C_6H_5OCH_3$). Pour 100 gms. of methyl alcohol into a round-bottom flask connected with an upright condenser. Then add 5 gms. of sodium cut into small pieces. When the sodium has dissolved, add 20 gms. of phenol and 40 gms. of methyl iodide. The mixture is heated on the water-bath until the solution no longer has an alkaline reaction (two to four hours). As much as possible of the methyl alcohol is distilled off on the water-bath and water added to the amber-colored residue. The colorless oil which separates is extracted with ether. The ethereal solution is dehydrated over calcium chloride and distilled, first on the water-bath, until the ether has been driven off, and then over the flame. Almost all of the residue distills at 150° - 155° .

65. Benzoic Acid (C_6H_5COOH). Mix 50 gms. of benzyl chloride with 40 gms. of soda ash, dissolved in 500 cc. of water, and heat to boiling in a round-bottom flask provided with a reflux condenser. A solution of 85

gms. of potassium permanganate in 1000 cc. of water is gradually poured in through the top of the condenser. After boiling for about three hours, the pink color of the permanganate will have disappeared and a heavy precipitate of manganese dioxide formed. The solution is then filtered and the filtrate acidified with hydrochloric acid and allowed to cool; whereupon crystals of benzoic acid separate.

66. Methyl Benzoate or Niobe Oil ($C_6H_5COOCH_3$). Dissolve 50 gms. of benzoic acid in an excess of methyl alcohol and saturate with dry hydrochloric acid gas. After saturation the solution is heated for about four hours on a steam-bath and then poured into an equal volume of water. The oil which separates is purified by rectification.

67. Benzoyl Chloride (C_6H_5COCl). Treat 50 gms. of benzoic acid in a dry 500-cc. flask with 90 gms. of finely pulverized phosphorus pentachloride, under the hood. The two are shaken well together, whereupon an energetic action takes place with the evolution of hydrochloric acid; in order to prevent the cracking of the flask, place it on a wooden block and not on any stone or porcelain. After standing for a time, it is twice fractionated, using a long, wide air condenser.

68. Nitrobenzene ($C_6H_5NO_2$). Two hundred and forty gms. of nitric acid, sp. gr. 1.4, is added, a small portion at a time, to 360 gms. of sulphuric acid, sp. gr. 1.82, in a 1000-cc. flask. During this operation the flask is well cooled under the tap. The mixture is transferred to a tap funnel and added slowly to 200 gms. of benzol in a 2-liter flask. The flask is well shaken after each addition, and the temperature not allowed to rise above 25° C. until nearly

all of the acid has been added. A wide glass tube is now attached by a cork to the flask, forming an air condenser, and the mixture heated on the water-bath for an hour at 60°C ., being frequently shaken. The contents of the flask are poured into about 2 liters of water, when the nitrobenzene settles to the bottom and can be removed by means of a separatory funnel. It is then washed several times with water. The nitrobenzene is next run into a distilling flask fitted with a thermometer and connected

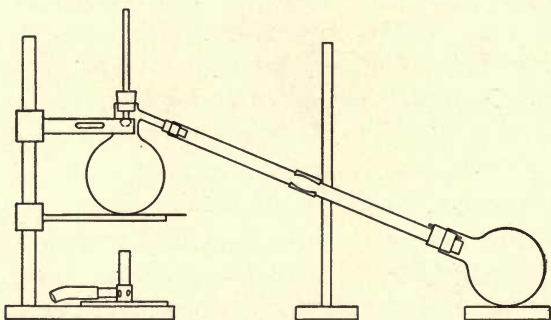


FIG. 22.

with a long glass tube about 8–10 mm. in diameter, forming an air condenser (Fig. 22). The oil is distilled over a free flame. At first, water and benzene pass over, then the temperature rises rapidly to over 200°C ., when the receiver is changed and the nitrobenzene collected at 204° – 207°C .

69. Metadinitrobenzene ($\text{C}_6\text{H}_4(\text{NO}_2)_2$). To a mixture of 70 gms. of fuming nitric acid and 70 gms. of sulphuric acid add 100 gms. of nitrobenzene, shaking the flask well after each addition and maintaining the temperature at about 75°C . When all of the nitrobenzene has been added,

heat the flask for a short time on the water-bath until a test portion, when thrown into water, forms a yellow solid. While the entire reaction product is still warm, throw into a large quantity of water. The metadinitrobenzene, mixed with a small amount of ortho and para compounds, separates, on standing, as a yellow crystalline mass, which is filtered at the pump and washed with cold water. The mixed compounds are dissolved in hot dilute alcohol, from which, on cooling, the pure metadinitrobenzene crystallizes in light yellow needles.

70. Ortho- and Para-Nitrophenol $\left(\text{C}_6\text{H}_4 \begin{array}{l} \text{OH} \\ \text{NO}_2 \end{array}\right)$. One

hundred gms. of phenol is melted and slowly added to a mixture of 175 gms. of nitric acid in 400 cc. of water and well shaken after each addition. When all of the phenol has been added, the mixture is allowed to stand overnight. The heavy, oily layer in the bottom of the flask is washed several times by decantation. The mixture of the ortho and para compounds is then subjected to steam distillation, when the ortho compound passes over as a yellow liquid that becomes solid upon cooling and is separated at the pump.

The residue in the flask consists of the para compound mixed with resinous matter from which it is separated by repeatedly extracting with boiling water. To purify the product, make it alkaline with caustic soda and boil again. Filter and concentrate the filtrate until crystals of the sodium salt separate. The latter are dissolved in hot water, neutralized with hydrochloric acid, separated by filtration, and recrystallized from hot water.

71. Picric Acid $(\text{C}_6\text{H}_2(\text{NO}_2)_3\text{OH})$. Heat together on a water-bath to about 100°C . a mixture of 20 gms. of

phenol and 20 gms. conc. sulphuric acid until complete solution takes place. Dilute with twice the volume of water and transfer to a separatory funnel. Now add this mixture *gradually, carefully*, and with *constant shaking*, to 100 gms. of conc. nitric acid. Warm the mixture on a water-bath until the red color changes to yellow, then pour into a liter of water, filter off the crystals, drain thoroughly, wash with water, and recrystallize from hot water containing 10 per cent sulphuric acid.

Dye a skein of worsted and silk in a solution of the acid as given in dye-test Nos. 124 and 129. Be careful with the salts of this acid, as they explode when heated or struck.

72. Cinnamic Acid ($C_6H_5CH:CHCOOH$). A mixture of 20 gms. benzaldehyde, 30 gms. acetic anhydride, and 10 gms. anhydrous pulverized sodium acetate is heated in a flask provided with a wide vertical air condenser for eight hours in an oil-bath at $180^\circ C$. If the experiment cannot be made at one time, a calcium chloride tube may be placed over the end of the condenser. After the reaction is complete, pour the hot reaction product into a large flask, add water, and distill with steam (see Fig. 23), until no more benzaldehyde passes over. The quantity of water used here is large enough to dissolve all the cinnamic acid except a small oily impurity. The solution is then boiled a short time with some animal charcoal and filtered. On cooling, the cinnamic acid separates out in lustrous leaves. Drain and dry. Determine melting-point.

73. Salicylic Acid ($C_6H_4 \begin{matrix} \langle OH \\ COOH \end{matrix}$). Dissolve 100 gms. of caustic soda in 100 cc. of water in an iron crucible and

add 230 gms. of phenol. Heat the retort gently and stir well with a small iron rod. As soon as the mass becomes stiff, the flame should be removed and the stirring continued until the lumps are broken up. The mass, while yet warm, is powdered in a mortar, transferred to a retort, and heated in an oil-bath to a temperature of about 130° C., the apparatus being aspirated by means of dry air until moisture is removed. The mass is then again transferred to the mortar, quickly powdered, and returned to the retort. A stream of dry carbon dioxide is now passed over the surface of the dry mixture and the temperature of the oil-bath raised to 190° C. After heating for four hours, the temperature is again raised to 200° , the heating continued for another hour and then stopped. The mass is now shaken out into a suitable vessel and sufficient water added to dissolve it. The solution is next made acid with hydrochloric acid, allowed to cool, and filtered. The dark-colored precipitate of impure salicylic acid is washed from the filter, boiled with water containing a small amount of animal charcoal, filtered again, and the filtrate allowed to cool, when needle-shaped crystals of salicylic acid separate.

74. Aniline ($C_6H_5NH_2$). Place 240 gms. of iron powder with 320 cc. of water in a large flask (2-3 liters) and shake well. Warm the flask slightly and add a few drops of nitrobenzene. Then pour 20 gms. of hydrochloric acid into the flask and introduce the remainder of the nitrobenzene (200 gms. in all). Add the latter in small portions and after each addition shake the flask thoroughly and cool under the tap. The addition of the nitrobenzene is so regulated as to keep the temperature at about 80° - 90° C. When no further rise in temperature

on shaking indicates that the reaction is finished, neutralize excess of acid with hydrated lime and distill the contents of the flask with steam (Fig. 23) until the distillate is no longer milky, then transfer it to a separatory funnel and draw off the aniline. The water is now saturated

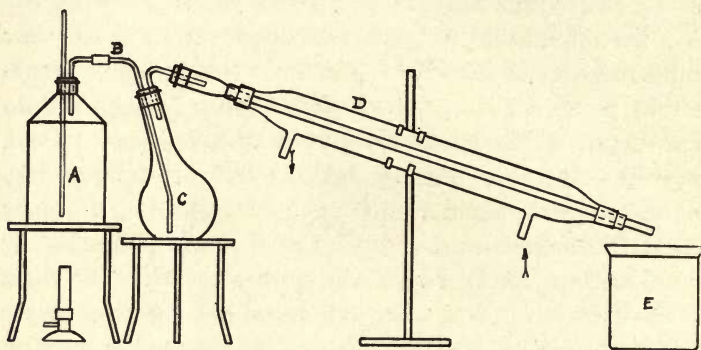


FIG. 23.

with salt, and the oil which rises to the top is added to the first portion, and the whole distilled. A little water passes over at first and is collected separately; then aniline passes over at 182° .

75. Dimethyl Aniline ($C_6H_5N(CH_3)_2$). A mixture of 150 gms. of aniline, 50 gms. of aniline hydrochloride, and 150 gms. of methyl alcohol is heated in an iron autoclave for seven or eight hours at 230° – 240° C. The product is then made alkaline with NaOH, distilled with steam, and the oil separated. The product is dried over solid caustic potash, and the fraction boiling at 190° – 200° collected.

76. *p*-Sulphanilic Acid ($C_6H_4\begin{matrix} \langle NH_2 \\ HSO_3 \end{matrix}$). Two hundred gms. of aniline is stirred into 220 gms. of conc. sulphuric

acid in a shallow porcelain dish, and the acid sulphate thus obtained ($C_6H_5NH_2H_2SO_4$) is heated in an oven until the temperature reaches $205^\circ C$. This must take four hours. The oven is kept at this temperature for six hours or more. The product is broken up and dissolved in hot water with the addition of 80 gms. of caustic soda (alkaline reaction must be obtained). The solution of sodium sulphanilate is boiled for a few minutes with a little animal charcoal and filtered hot. On acidifying with hydrochloric acid (Congo paper must be turned blue), the sulphanilic acid crystallizes out. After standing overnight it is filtered at the pump and dried at $100^\circ C$.

77. Acetanilide ($C_6H_5NHC_2H_3O$). Two hundred gms. of aniline and 150 gms. of glacial acetic acid are mixed in a round-bottom flask of 1 liter capacity, fitted with a reflux air condenser, and boiled on a sand-bath for ten or twelve hours. The hot liquid is poured into hot water containing 30 gms. of hydrochloric acid, and the whole well stirred. After cooling, the acetanilid is filtered at the pump, washed with cold water, recrystallized from hot water, and dried in the centrifugal.

78. *p*-Nitracetanilide ($C_6H_4NH(CH_3CO)NO_2$). Dissolve 200 gms. of acetanilide in 600 gms. of sulphuric acid in a round-bottom flask, the temperature not being allowed to rise above 40° . The solution is cooled to $5^\circ-10^\circ$ by immersion in ice-water, and a cool mixture of 125 gms. nitric acid and 100 gms. of sulphuric acid added very slowly. After each addition the flask is well shaken and cooled in ice-water, the temperature being kept below 15° . After standing a short time, the nitration mixture is poured into about 20 liters of water containing several pieces of ice, when the *p*-nitracetanilide separates.

It is filtered, washed free from acid, and dried on a porous plate.

79. *p*-Nitraniline ($\text{C}_6\text{H}_4 \begin{matrix} \text{NH}_2 \\ \text{NO}_2 \end{matrix}$). Two hundred gms. of *p*-nitracetanilide is boiled with 500 cc. of dilute sulphuric acid (25 per cent) in a round-bottom flask, connected with a reflux condenser, until the whole dissolves. The clear solution is poured into a beaker, and the free base precipitated by adding dilute caustic soda solution until alkaline. After cooling, the *p*-nitraniline is filtered, re-crystallized from hot water, and dried on a porous plate.

80. Benzidine ($\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4 \cdot \text{NH}_2$). One hundred gms. of caustic soda is dissolved in 300 cc. of water in a 2-liter flask and a mixture of 50 cc. of ethyl alcohol and 100 gms. of nitrobenzene added. The flask is then connected with a Liebig's reflux condenser. Through the top of the condenser add 170 gms. of zinc dust in small portions, shaking well and allowing any foaming to subside before introducing a fresh quantity. The brown color noticed at first disappears as the reduction progresses, until the contents appear grayish white, usually accomplished by heating on the sand-bath toward the end of the reaction. The contents of the flask on cooling are diluted with water; ice is added; and then the whole is acidified with hydrochloric acid, care being taken that the temperature does not exceed 15°C . The precipitate, consisting of hydrazo-benzene and undissolved zinc, is filtered through glass wool. It is then removed from the funnel and boiled with 500 cc. of water to which strong hydrochloric acid is added as long as absorbed. The solution, on being filtered, is treated with sulphuric acid, which precipitates the benzidine as sulphate. This is

filtered off, washed with cold water, and then boiled with dilute caustic soda solution. The pure benzidine now separates and, on cooling, crystallizes in large silky plates.

81. Naphthalene β -Sulphonic Acid ($C_{10}H_7HSO_3$). Two hundred and forty gms. of concentrated sulphuric acid is warmed to $100^\circ C$. and 200 gms. of powdered naphthalene gradually added. The mixture is then heated at $170^\circ C$. for twelve hours. The sulphonation mixture, on cooling, is poured into 3 liters of water, brought to a boil, and sufficient milk of lime added to produce an alkaline reaction. The precipitate of calcium sulphate is filtered at the suction pump and washed with boiling water. The filtrate and washings are evaporated to a small volume and allowed to stand overnight, when the calcium salt of naphthalene β -sulphonic acid separates. The calcium salt is now dissolved in hot water and sufficient sodium carbonate added to combine with all of the calcium as carbonates—care should be taken not to add an excess. The precipitate of calcium carbonate is filtered off and the filtrate evaporated until crystals appear. It is then set aside overnight and, the crystals being separated, the mother liquor is evaporated for a further yield.

82. β -Naphthol ($C_{10}H_7OH$). Six hundred gms. of caustic soda and 60 gms. of water are placed in an iron crucible and heated to $280^\circ C$. Care must be taken to inclose the bulb of the thermometer in copper foil; the hands should be protected with thick gloves, and the eyes with goggles. To the heated caustic soda, 200 gms. of sodium naphthalene β -sulphonic acid is cautiously added, care being taken that the temperature does not fall below $260^\circ C$. The temperature is now raised to $320^\circ C$. When the mass froths and becomes liquid, the reaction is com-

plete. The liquid is now thrown on shallow iron pans and, when cool, is broken up and dissolved in hot water. To the solution of sodium β -naphtholate, concentrated hydrochloric acid is added, and the impure β -naphthol is filtered at the pump. To further purify the product, it is distilled in a vacuum.

83. β -Naphthol-6-Sulphonic Acid (Schaeffer's Salt)
 $(C_{10}H_6 \begin{matrix} \text{OH} \\ \text{HSO}_3 \end{matrix})$. Two hundred gms. of finely divided β -naphthol is gradually added to 400 gms. of sulphuric acid previously warmed to 35° C. The mixture is then heated on the water-bath to 100° C. for eight hours. The resulting product is dissolved in 2 liters of water and saturated with common salt. The sodium salt of the naphthol sulphonic acid separates on standing and is filtered off, washed with saturated salt solution until free from acid, and then dried.

84. β -Naphthol-3 : 6-Disulphonic Acid ("R Salt")
 $(C_{10}H_5 \begin{matrix} \text{OH} \\ (\text{SO}_3\text{H})_2 \end{matrix})$. Eight hundred gms. of sulphuric acid is heated to 125° C., 200 gms. of finely powdered β -naphthol added, and the temperature kept at 125° C. for six hours. The reaction product is now thrown into 3 liters of water and neutralized with milk of lime. The calcium sulphate is filtered off and the precipitate washed with boiling water. To the filtrate and washing is added sodium carbonate, and the resulting calcium carbonate removed on the suction filter. The "R Salt" is precipitated from the filtrate by the addition of common salt, the mixture being allowed to stand overnight.

85. α -Nitronaphthalene ($C_{10}H_7NO_2$). To a mixture of 400 gms. of nitric acid and 500 gms. of sulphuric acid

slowly add 500 gms. of finely powdered naphthalene. Shake the flask well and cool it after each addition, keeping the temperature at about 50° C. After adding all of the naphthalene heat the mixture for two hours at 60° C. and pour into cold water. The solid cake which settles to the bottom is washed several times with cold water, then with boiling water. The melted product is now subjected to steam distillation to remove excess of naphthalene. The residue in the flask is poured into cold water when the nitronaphthalene separates out as a granular solid.

86. α -Naphthylamine ($C_{10}H_7NH_2$). One hundred and sixty gms. of iron filings are mixed with 80 cc. of water and warmed to about 50° C. To this is added 25 cc. of concentrated hydrochloric acid, and then 120 gms. of α -nitronaphthalene is slowly introduced, temperature being maintained at about 70° throughout the reduction. When the reaction is complete—indicated by no further rise in temperature—a small amount of milk of lime is added until an alkaline reaction is obtained, and the mass allowed to cool. The precipitate, which is separated at the pump, is dried in the air and distilled under reduced pressure.

87. Anthraquinone ($C_6H_4(CO)_2C_6H_4$). Twenty gms. of anthracene is dissolved in 240 gms. glacial acetic acid by boiling them together in a round-bottom flask (1-liter) with upright condenser over wire gauze. A solution of 40 gms. of chromium trioxide in 30 cc. of water and 150 cc. of glacial acetic acid is then dropped in from a tap funnel pushed into the top end of the condenser, while the liquid is kept cooling. This operation should consume about one hour. The solution becomes a deep green. It is allowed to cool and is poured into water (1 liter)

which precipitates the anthraquinone in the form of a brown powder. After standing for one hour it is filtered through a large folded filter, washed with a little hot water, then with warm dilute caustic soda, and again with water. It may be purified by sublimation.

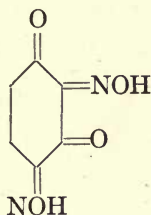
88. Anthraquinone Sulphonic Acid ($C_6H_4(CO)_2C_6H_3 \cdot HSO_3$). Add 100 gms. finely powdered anthraquinone to 100 gms. of sulphuric acid and heat the mixture gradually in an oil-bath until a temperature of 160° is obtained. The mixture is slowly and carefully poured into hot water (2 liters), the solution boiled for some time, and the unchanged anthraquinone filtered off at the pump. The filtrate is neutralized with caustic soda and allowed to cool, when the greater part of the sodium anthraquinone sulphonate crystallizes out. This is the so-called "silver salt." A second crop of crystals may be obtained by further concentration of the filtrate, but is apt to be contaminated with sodium sulphate.

89. Fluorescein ($C_{20}H_{12}O_5$). Grind together and heat in an oil-bath to 180° C. 30 gms. phthalic anhydride and 44 gms. resorcinol. For this purpose a nickel dish may be used. As soon as the temperature has reached 180° C., 14 gms. of zinc chloride is added gradually during ten minutes, the melt being stirred with a glass rod. After the zinc chloride has been added the temperature is raised to 210° C. and maintained until the mass becomes solid (one to two hours). The cold melt is broken out of the crucible with a chisel or knife, powdered, and dissolved in dilute caustic soda. After filtering, hydrochloric acid is added, which precipitates the fluorescein; this is filtered, washed and dried.

90. Eosine ($C_{20}H_6Br_4O_5Na_2$). Place 15 gms. of fluor-

escein in a flask, add 60 gms. of alcohol, and drop in slowly from a small separatory funnel 33 gms. of bromine. When half the bromine has been added, the dibromide formed is in solution; but on further addition of bromine the tetrabromide separates out. After standing for two hours the precipitate is filtered, washed with water, and converted into the sodium salt by mixing it with a little hot water, carefully neutralizing with caustic soda (avoiding an excess of this reagent) and then evaporating to dryness on the water-bath.

91. Fast Green O (*Dinitrosoresorcine*).

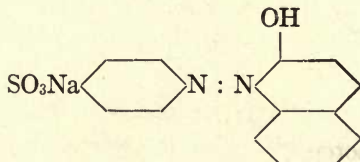


Forty gms. resorcinol is dissolved in 1600 cc. of water and 90 gms. conc. hydrochloric acid added, together with 200 gms. of common salt. Ice is added till the temperature is 4° C., and into this solution, which is stirred mechanically, 51 gms. of sodium nitrite, dissolved in 200 cc. of water, is allowed to flow very slowly, the temperature not being allowed to rise above 8° . This takes about half an hour. When all of the nitrite has been added, the liquid should show a faint acid reaction. After standing for one hour, the brownish-yellow precipitate is filtered, washed with ice-cold water, and the paste dried on a porous plate.

92. Naphthol Yellow S ($C_{10}H_4(NO_2)_2SO_4K_2$). Warm 400 gms. concentrated sulphuric acid in a round-bottom

flask to 100° C., and then add gradually 100 gms. finely powdered *a*-naphthol. Now raise the temperature to 120° and keep at this point for three or four hours. Pour the sulphonation mixture into 600 cc. of water and stir mechanically. As soon as the temperature is about 30°, add 200 gms. of conc. nitric acid very slowly, drop by drop, through a tap funnel and do not allow the temperature to exceed 45°. The dinitronaphthol sulphonic acid which separates out after standing is filtered at the pump, washed with saturated salt solution until free from acid, and mixed with boiling water. Potassium carbonate is now added until an alkaline reaction is obtained, and, after cooling, the precipitated potassium salt is filtered and dried on a porous plate.

93. Orange II.



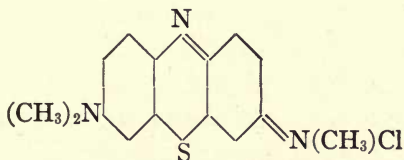
34.6 gms. of *p*-sulphanilic acid is dissolved in water by careful addition of caustic soda solution. Ice is added until the temperature is 5° C., the volume of the whole being about 1 liter; 60 cc. of conc. hydrochloric acid is poured in, and then slowly 14.48 gms. of sodium nitrite, dissolved in a small quantity of water added. Tests are made from time to time with starch iodide paper, and a slightly blue coloration should be obtained when all of the nitrite has been added. The diazo-compound separates out in fine white needles. Twenty-eight and eight-tenths gms. of *β*-naphthol is dissolved by heating it with

a solution of 9 gms. of caustic soda in 30 cc. of water, and the sodium naphtholate solution thus formed is poured into 320 cc. of cold water and cooled if necessary to about 15° . This solution is stirred and the diazo-compound (in suspension) run in gradually. When the whole of the diazo-compound has been added, the mass should show a weak alkaline reaction. The mass is stirred for an hour longer, when nearly all of the coloring-matter will have separated. A little salt solution is added to aid in the precipitation, until a test on filter paper shows only a pink orange rim.

Dye a skein of worsted and silk as under tests Nos. 124 and 127.

94. Alizarine $\left(\text{C}_6\text{H}_4(\text{CO})_2\text{C}_6\text{H}_2 \begin{matrix} \text{<OH} \\ \text{>OH} \end{matrix} \right)$. Three hundred gms. of caustic soda is dissolved in 300 cc. of water in an autoclave and 100 gms. of sodium anthraquinone sulphonate stirred in. Eighteen gms. of potassium chlorate is dissolved in 100 cc. of water (hot) and thoroughly mixed with the mass. The lid of the autoclave is then fixed on and the whole heated for 20 hours 170° . After cooling, the melt is extracted with boiling water several times, and the solution acidified with hydrochloric acid. The alizarine which separates is filtered at the pump, washed with water, pressed on a porous plate, and dried at 120° C.

95. Methylene Blue.



Twenty-four gms. of dimethyl aniline is dissolved in a mixture of 80 cc. of water and 130 gms. of concentrated hydrochloric acid, and the solution cooled with ice to 12° – 15° . This is stirred mechanically, and a solution of 14.2 gms. of sodium nitrite run in slowly (delivery tube underneath the surface of the liquid), care being taken that the temperature does not rise above 15° . The nitroso compound is reduced by carefully adding about 40 gms. of zinc dust. The reduction is complete when the solution is of a clear red color. The amount of zinc added must be sufficient to neutralize the hydrochloric acid (so that Congo paper is no longer turned blue). The solution is now diluted with water to 1000 cc. and a solution of 24 gms. of dimethyl aniline in the exact quantity of hydrochloric acid necessary to form the hydrochloride (about 20 cc.) added, and then a solution of 100 gms. of sodium thiosulphate in a little water introduced.

The mixture is oxidized by adding a concentrated solution of 50 gms. of potassium dichromate and boiling for two hours; 106 gms. of sulphuric acid diluted with 200 cc. of water is now added and the solution boiled to expel SO_2 .

The leuco-methylene blue is oxidized by adding 16 gms. of neutral sodium chromate dissolved in a little water, and the resulting dye precipitated by adding salt. The base is filtered, dissolved in a little boiling water, to which a little hydrochloric acid has been added, and the hydrochloride precipitated by common salt, filtered, and dried on a porous plate.

96. Indulin. (a) *Spirit Soluble.* Weigh off 1000 gms. of aniline in a small stoneware pitcher or a beaker and add 96 gms. 22° Bé. hydrochloric acid, stirring vigorously.

Now add slowly and with constant stirring a solution of 57.6 gms. sodium nitrite in 120 cc. of water. Allow the mixture to stand overnight and then warm to 40° C. for one hour to convert the diazoamidobenzene at first formed into aminoazobenzene. The warming is done by placing the pitcher or beaker in warm water. Next, transfer the mixture to an enameled pot with a cover (about 1 gal. capacity), or, preferably, a small enameled still of the same size provided with a detachable cover (Fig. 24) and an opening for a thermometer. Now add 240 gms. of aniline hydrochloride. Heat the container gradually, raising the temperature to 175° C. This takes from one to one and one-half hours, but the heating is continued for five hours, keeping the temperature as close to 183° C. as possible. The precaution must be observed in heating to have a sheet of asbestos under the pot large enough to project some distance beyond the rim, otherwise the escaping fumes will reach the flame and ignite the batch. In using the still this danger is avoided, because the vapors are condensed. When the aniline in the condensation is later recovered by salting out.

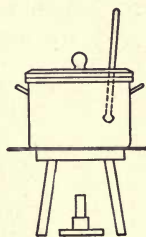


FIG. 24.

When the heating is complete, the melt is cooled sufficiently and then steam-distilled to remove excess aniline. The melt is poured into a distilling apparatus; a 3-gal. chlorinating pot (Fig. 25) serves this purpose very well. The pot is provided with a live, steam inlet at one opening and a condenser at the other. One thousand cc. of water is added and the mass steam-distilled rapidly until the distillate is clear and free from aniline oil globules. The spirit-

soluble indulin (about 240 gms.) is left as a sticky mass at the bottom of the vessel. On cooling, it becomes hard and brittle, and is easily ground to a fine powder. From the distillate the aniline is recovered by adding enough salt to give a specific gravity of about 1.026, whereupon the aniline rises to the top and is drawn off.

(b) *Water Soluble.* Eight hundred gms. of concentrated sulphuric acid is heated in a beaker, lead-lined or iron pot until the temperature reaches 100° – 130° C., when 200 gms. of coarsely powdered spirit soluble indulin is added a little at a time with constant stirring, maintain-

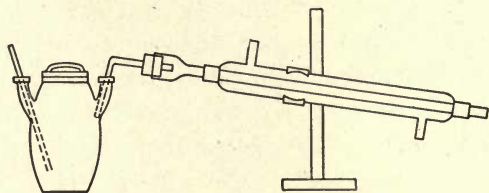


FIG. 25.

ing the temperature throughout between 100° and 130° . When all the indulin has been dissolved, as shown by the absence of lumps, dilute a drop of the sulphonated mixture in hot water in a test tube, add a few drops of dilute caustic soda solution, and warm. A complete solution of the color shows that sulphonation is accomplished. If there is still some insoluble color, continue heating the sulphonated mixture until a sample is completely soluble in the dilute alkali.

To free the sulphonated indulin mixture of excess sulphuric acid, wash by pouring into 6 liters of water. Agitate well, let settle, and filter on a suction plate. Break

up the hard cake thus obtained in 3 liters of water, agitate, and filter again.

The next step is neutralization to produce the water soluble sodium salt. To the insoluble sulphonation product of the indulin add 30 per cent caustic soda solution, a little at a time with thorough mixing, until a sample of the color is completely soluble in water. The water soluble indulin paste is then evaporated to dryness on a water-bath or in a drying oven, and ground.

97. Nigrosine. (a) *Spirit Soluble.* Into a 5-gallon,

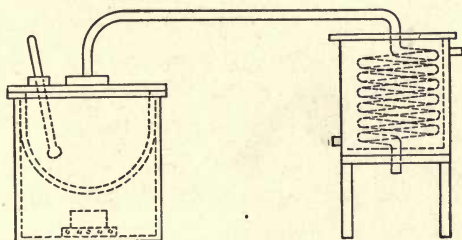


FIG. 26.

1-inch thick cast-iron kettle (Fig. 26), pour 3500 gms. of aniline, 3500 gms. of mirbane oil (nitrobenzene), and 4200 gms. of 20° Bé. hydrochloric acid, or its 22° Bé. equivalent. Add 350 gms. of iron filings and 18 gms. copper filings. Place a woven asbestos gasket on the flange, or make one by putting on a thick layer of asbestos fiber made into a stiff paste with water. Put cover in place and clamp it down tight with about 5 strong clamps. Connect to a 1-inch iron pipe condenser. Heat as follows:

Up to 110° C. in about one hour (a rise of approximately 1½° per minute);

Maintain at 110° for three to four hours to distill off the water in the hydrochloric acid (about 15 gms. should distill per minute);

From 110° – 115° in three hours, keeping the flame underneath very low. The heat adjustment will have to be very carefully made above 110° .

From 115° – 133° in three hours.

At 133° for one hour.

From 133° – 150° in three hours (about 6° per hour rise).

From 150° – 170° in one hour, and turn off the gas.

At this point a violent internal reaction will take place, vapors will be given off, and the temperature will rise above 195° . If the reaction fails to take place at 170° heat slowly above that point until the reaction starts. When the vapors cease coming over, the batch is finished. It is allowed to cool and harden. The cover is then removed and the brittle mass is chopped out. This is crude spirit-soluble nigrosine contaminated with iron, copper, and organic impurities, subsequently removed by sulphonation and washing.

Should it be impossible to complete the heating in one run, it may be discontinued at any point before the final reaction and started again at another time. It is advisable, however, to at least reach above the 110° point. In reheating, care must be taken to heat slowly.

(b) *Water Soluble*. If the heating has been properly controlled, the crude melt will sulphonate in a mixture of three times its weight of sulphuric acid (sp. gr. 1.84) and three-tenths of its weight of fuming sulphuric acid; otherwise, the sulphonation must be carried out with three times its weight of fuming acid entirely. The fuming acid costs more and is more difficult to handle.

To test for strength of sulphonating acid required, slowly stir 25 gms. of the ground nigrosine into 75 gms. of sulphuric acid and 7.5 gms. of fuming acid in a beaker, keeping at 100° - 110° . After it has completely dissolved, heating and stirring are continued for one hour. About one-fourth of the sulphonated mass is poured into 150 cc. of water, stirred, filtered, and washed. A particle of the color is added to a test tube of water, heated, and a few drops of dilute caustic soda solution added to slight alkalinity. Complete solubility of the color indicates that this strength of acid may be used. If a precipitate forms on the addition of the caustic, it shows that, although the spirit-soluble nigrosine has dissolved in this strength of acid, it has not sulphonated, and therefore, it will be necessary to use the straight fuming acid for sulphonation.

To sulphonate, heat 12.6 kgms. of sulphuric acid and 1.26 kgms. of fuming acid to 100° - 110° in a 4- to 5-gal. iron vessel, heated by gas or lead steam coil, preferably provided with an iron stirrer and hood (Fig. 27). Slowly and with constant stirring, add 4.2 kgms. of the coarsely ground nigrosine, taking care that the mass does not foam over. When all the color is in, heat and stir for about two more hours and test a sample for complete sulphonation by pouring into water, washing and neutralizing as directed in testing for the strength of sulphonating acid. If color is not entirely soluble in water, heat and agitate for two more hours, or until soluble. If the fuming sulphuric acid must be used, 4.2 kgms. of color will take 12.6 kgms. of acid.

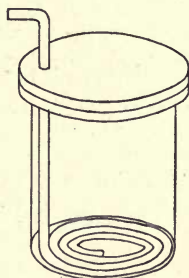


FIG. 27.

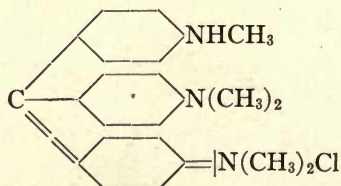
To free excess acid, wash by slowly stirring the sulphonated mass into 30 gals. of water. Agitate for one-half hour; allow to settle for about six hours or more, and siphon off the clear liquid. Add 15 gals. of water, agitate, and settle again. Siphon off. Add 15 gals. of water for the last washing and agitate while the color is being filtered on a suction-plate or through a filter press.

The next step is neutralization to form the water-soluble sodium salt. To the wet cake from the filter carefully add a 30 per cent caustic soda solution, mixing well until a sample is completely soluble in water. It is necessary to obtain a neutral or slightly alkaline condition.

The wet, water soluble nigrosine may be dried in a hot-air oven or in a vacuum drier. Water soluble nigrosine comes on the market in small lumps the size of a pea. If desired in a powdered form, it can be easily ground.

Note. This method of making nigrosine is only one of many for the preparation of this color, and together with indulin has been furnished by one of my former students, Mr. Aaron Schreiman.

98. Methyl Violet T. B.

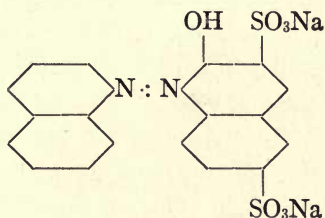


Mix well in a mortar 875 gms. of sodium chloride and 50 gms. finely powdered copper sulphate, and add

a solution of 40 gms. of phenol in 10 cc. of water. Stir well. One hundred gms. of dimethyl aniline is now added and the mixture transferred to a round-bottom flask and heated on the water-bath for eight hours at 55° C. The product is poured out into a porcelain basin and allowed to cool. In order to free it from phenol and salt, it is broken up and added gradually to 3 liters of boiling water containing milk of lime, prepared from 40 gms. of CaO and 200 cc. of water. After heating until no more lumps are present, the mixture is allowed to settle, and the clear solution of salt and calcium phenate decanted. The residue of copper oxide, methyl violet, and calcium sulphate is washed again by decantation, and finally filtered.

In order to get rid of the copper oxide, the precipitate is boiled with dilute sulphuric acid, and sodium sulphate (free from chloride) added to precipitate the dye, which is filtered and washed. The precipitated violet is subsequently dissolved in water and reprecipitated with salt.

99. Fast Red B. (*Bordeaux B*).



Seventy-one and a half gms. of α -naphthylamine is dissolved in 1500 cc. of water, 150 cc. of concentrated hydrochloric acid added, and the temperature reduced

to about 40° C. by the addition of ice. Thirty-six gms. of sodium nitrite dissolved in a small quantity of water is now poured in. The diazo solution thus obtained is poured into a solution of 175 gms. of R salt in 2000 cc. of water, previously made alkaline by the addition of 50 gms. of caustic soda, and cooled to 15° C. After standing for at least one hour, the solution is warmed to 80° C. and common salt added until the dyestuff is precipitated. The latter is then filtered off and dried.

100. Chrysamine G.



Thirty-seven gms. of benzidine is dissolved in 500 cc. of hot water, containing 100 cc. of concentrated hydrochloric acid, and then cooled with ice to 5° C. The cold solution of the benzidine chloride is next tetrazotized with 30 gms. of sodium nitrite in a small quantity of water. The solution, after addition of the sodium nitrite, should be clear and give a blue color with starch-iodide paper.

Sixty-two gms. of salicylic acid is dissolved in 500 cc. of cold water to which has been added 20 gms. of caustic soda. To this alkaline solution the above tetrazo solution is added and the whole stirred mechanically for six hours. During the stirring, 25 gms. more of caustic soda is gradually added. The coloring matter is slowly precipitated and is eventually filtered off and dried.

101. Sulphur Black T. (Constitution Unknown.) Five hundred gms. of sodium sulphide is dissolved in 750 cc. of water, and to this solution is added 180 gms. of sul-

phur and 120 gms. of orthodinitrophenol. This mixture is then boiled for eighteen hours on a sand-bath, in a flask provided with reflux condenser. The flask, after heating, is removed from the sand-bath and a stopper inserted, arranged with glass tube extending to the bottom of the liquid. Another glass tube is also placed in the cork in such a manner that it may be connected with the suction pump. A stream of air is now drawn through the liquid until a test sample on filter paper shows a colorless rim. The precipitate is then filtered off and dried.

CHAPTER IV

DYEING OF TEXTILE FIBERS

102. Dyeing is the process of precipitating coloring-matter upon or within the substance of a body by chemical action. The solution may be neutral, acid, or alkaline, according to the nature of the material and of the dyestuff employed.

103. There are two general classes of dyestuffs, namely, natural and artificial. Owing to the large number of coal-tar colors on the market, however, and the ease with which they are applied, the natural colors have been steadily replaced, until to-day logwood and fustic are practically the only ones that are of any importance in the trade.

104. For practical reasons, the coal-tar colors are divided generally into the following groups:

1. Acid dyes.
2. Basic dyes.
3. Dyes of the eosine group.
4. Substantive dyes.
5. Mordant dyes.
6. Developed dyes.
7. Sulphur dyes.
8. Spirit colors.

105. Acid Dyes. These colors are chiefly used for wool dyeing, but they are also applied to silk, and with

few exceptions the dyeing takes place in an acid bath. Although of little interest for cotton, they are of great importance for jute. The commercial dyestuffs consist of the alkali or lime salts of the color acid. Thus, in the case of wool dyeing, the wool acts as a base, combining with the color acid to form an insoluble compound.

106. Basic Dyes. These colors are chiefly used on mordanted cotton. Large quantities are also consumed in silk dyeing, and a considerable amount of a few brands for wool. They are also extensively used for jute. Basic dyes are the salts of colorless bases, that is, the color does not appear until the salt is formed. The commercial dyestuffs are usually salts of acetic, oxalic, nitric, sulphuric, or hydrochloric acid, and most of them are soluble in water. In the process of dyeing, these compounds decompose, setting free the acid, while the base combines with the acid constituent of the animal fiber, or with the acid mordant in the case of vegetable fiber, thus producing the color on the fabric.

107. Dyes of the Eosine Group. In the textile industry these colors are used for wool and silk, also to a small extent for cotton. They are derived from fluorescein by the action of halogens. To this group belong eosine, erythrosine, phloxine, rose bengal, and uranine. All of these dyestuffs possess a yellowish-green fluorescence.

108. Substantive Dyes. These are very largely consumed for dyeing cotton and unions; they are also used for wool and silk. They are sometimes called "direct dyes," owing to the fact that they dye cotton direct without mordanting. To this group belong the benzidine

dyes, primuline, Congo, diamine, benzo and amino colors.

109. Mordant Dyes. These comprise a great variety of coloring-matters, both natural and artificial, which are dyed on all fibers by the aid of metallic mordants. Many of these dyes are polygenetic, and they possess the property of forming insoluble color lakes with metallic oxides. The usual method employed is to mordant the goods before passing them into the dye-bath, which usually contains nothing but the color. In a few cases the mordant and color are introduced into the same bath, whereas, in the process known as "padding," the goods are first run in the color and subsequently in the mordant.

110. Developed Dyes. Under this heading are included two different methods of developing, viz.:

(a) The production of dyestuffs from substances which possess no dyeing quality themselves, but which can be changed into dyestuffs by a chemical reaction taking place on the fiber.

(b) The production of new products by further developing dyestuffs so as to change their shade and properties.

The first of these processes is represented by the production of aniline black; the second, by the process of diazotizing, both of which will be taken up later.

111. Spirit Colors. These dyes are insoluble in water but soluble in alcohol. They are sometimes used for dyeing silk, and, when dissolved in acetone, for printing silk and cotton, also for making colored varnish.

112. General Instructions for Dyeing. In conducting dye-tests, it will prove advantageous to make up the solutions as follows:

Natural dyestuffs and tannins . . .	10 per cent solution
Coal-tar colors	1 per cent solution
Acids	10 per cent solution
Salts	10 per cent solution
Soap	1 per cent solution

Provide yourself with a reel, a convenient form of which is represented in the illustration, Fig. 28. As all

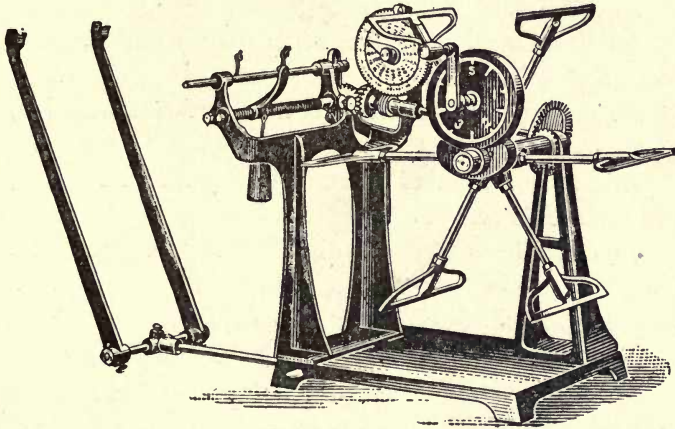


FIG. 28.

tests are conducted on the weight of the material, it is necessary to determine the number of yards required to give a skein of definite weight. The following list, however, may prove useful:

Columbia knitting worsted . . .	20 yds. weigh	5 gms.
Cotton yarn, 22-ply	100 yds. weigh	5 gms.
Spun silk, 2-ply	100 yds. weigh	5 gms.

By noting the example given below, a clear idea may be gained as to the use of the solutions mentioned and the reason for having the skeins of definite weight.

113. Example. Prepare a dye-bath with 2 per cent of Congo red, 2 per cent of sodium carbonate, and 20 per cent of Glauber's salt, using six 5-gm. skeins of cotton yarn, boil for half an hour. Remove, rinse, and dry.

Thus: $6 \times 5 = 30$ gms.

2 per cent of 30 gms. is 0.6 gm.

20 per cent of 30 gms. is 6 gms.

As the Congo red is a 1 per cent solution, it will require 60 cc.

As the sodium carbonate is a 10 per cent solution, it will require 6 cc.

And as the Glauber's salt is a 10 per cent solution, it will require 60 cc.

The dye-tests are best made in porcelain beakers placed in brine to secure a uniform temperature. The skein is suspended on a glass rod and the position changed at least once in five minutes during the time of dyeing.

On completing the tests the skeins are hung up to dry, twisted, and mounted as follows:

Place each end of the skein over the two middle fingers, twist until tight, then bring the right-hand loop under the wrist and up through the loop in the left. Now, holding the two loops, stretch the skeins out straight and allow them to curl together. In this manner a very tight and even skein is secured. These skeins should then be mounted on cardboard by sewing through from the back, catching down the threads on the under side in such a way that it does not show from the front.

114. Wool. In the study of dyes and dyeing, we will

first take up the natural mordant colors, that may be used for wool.

One of the oldest and most important dyes for wool is logwood, or Campeachy wood, which is the wood of a larch tree growing abundantly in the West Indies, Mexico, and parts of Central America. It is imported in logs weighing about 400 lbs. The fresh wood is colorless, or nearly so, and contains a glucoside, composed of hematoxyline and sugar. It is cut into chips or rasped. These chips are then digested with hot water, and the solution, on evaporation, gives the logwood extract of the market.

Formerly, for wool dyeing, the chips were allowed to oxidize before extraction, but now this is accomplished in the solution, and the product is known as hematine paste or crystals.

Logwood is a mordant color, by which we mean that the color is produced only by bringing it in contact with some metal. In this way a combination takes place with the formation of an insoluble lake.

115. The simplest method of dyeing with logwood consists in applying the coloring matter and mordant at the same time. The disadvantage of this method is that part of the lake produced is only superficially held by the fiber, and also much color is lost by being precipitated in the bath. It is used to some extent, however, on account of the cheapness of the color.

To illustrate this method, dye a 5-gm. skein of woollen yarn with 10 per cent of hematine logwood paste, 3 per cent of ferrous sulphate, and 1 per cent of oxalic acid in 400 cc. of water, keeping it at a quiet boil for one hour. In all dyeing experiments it is essential to keep the solution at a constant volume by adding hot water from time

to time. Care should also be taken that the skeins are turned several times during the dyeing operation.

116. The usual method of dyeing with logwood or other mordant color is to mordant the fiber before introducing it into the dye-bath. The wool is, therefore, boiled in the solution of a metallic salt to which has been added some acid or acid salt, the latter acting as an assistant to the mordant. The wool, being of a basic character, acts chemically upon the metallic salt, fixing the metal in such a way that it cannot be washed out.

117. Boil three 5-gm. skeins of wool for half an hour in a solution containing 5 per cent of alum, 3 per cent of oxalic acid, and 400 cc. of water. Dye one of these skeins at boiling with 5 per cent of hematine powder in 400 cc. of water. On removing the first skein, a second one should be introduced and run for half an hour to see if the color has been exhausted. The third skein is dyed with 5 per cent of fustic extract, being run for one hour.

118. The most important mordant for wool is chromium, generally applied as the dichromate, together with an acid or acid salt. The assistant in this case may be either sulphuric acid, oxalic acid, tartaric acid, potassium bitartrate, or potassium bisulphate. The color produced by logwood depends both upon the mordant and the assistant employed, and is an example of that class of dyestuffs known as *polygenetic*. *Monogenetic* colors, on the other hand, are those which give only one color, no matter what kind of mordant is used.

119. To demonstrate the application of chrome and sulphuric acid, mordant two 5-gm. skeins of worsted with 3 per cent of sodium dichromate and $1\frac{1}{4}$ per cent of sulphuric acid in 400 cc. of water, boiling for $1\frac{1}{2}$ hours. The

sulphuric acid liberates the chromic acid, which, in turn, unites with the wool, producing a chromate of wool. By this means, some of the chromic acid is reduced; the largest part, however, remains unreduced, oxidizes the coloring matter of the logwood, and combines with it to produce a blue-black lake. In addition to the above, the sulphuric acid prevents the metallic salt from decomposing too readily and thus aids in producing even and fast colors.

Introduce one of the skeins, which has been mordanted as above, into a bath containing 10 per cent of hematine paste and boil for one hour, running the second skein as an exhaust test.

120. Mordant two more skeins of worsted, as in 119, then dye with 10 per cent of hematine paste and $\frac{1}{2}$ per cent of alizarine yellow. It will be noticed that a deep black is the result as compared with the shade produced by straight hematine.

121. Mordant two 5-gm. skeins of worsted yarn with 3 per cent of sodium dichromate and 2 per cent of oxalic acid in 400 cc. of water, boiling for one-half hour. Dye one of these skeins with 10 per cent of hematine paste for one-half hour, following with the second skein as an exhaust.

122. Mordant four 5-gm. skeins of worsted yarn with 3 per cent of sodium dichromate and $2\frac{1}{2}$ per cent of cream of tartar in 400 cc. of water, boiling for one hour. Dye one of the skeins with 10 per cent of hematine paste, boiling for one hour; follow with a second skein as an exhaust. Dye a third skein with 10 per cent of hematine paste and 1 per cent of alizarine yellow, following with an exhaust.

123. Mordant four 5-gm. skeins of worsted yarn with

3 per cent of sodium dichromate and 1 per cent of sulphuric acid in 400 cc. of water, boiling for one hour. Dye one skein with each of the following dyestuffs:

Alizarine Red	Alizarine Yellow
Alizarine Orange	Alizarine Blue

The alizarine colors were originally derived from madder, but are now prepared synthetically. In working with them the mordanted material should be entered cold, the temperature slowly raised to the boil, and the boiling continued for one hour.

124. The simplest method of dyeing wool is that which employs the acid colors. This process may be considered as a lake formation in which the animal fiber acts the part of a base, while the color plays the part of an acid. In conducting these tests, the skeins are entered hot, or even at the boil, and the boiling continued for one hour, using 20 per cent of Glauber's salt, 3 per cent of sulphuric acid, and 1 per cent of color, except in the case of blacks, when as high as 7 per cent may be necessary.

Some acid colors:

Fast Red	Acid Violet (all brands)
Patent Blue	Orange (all brands)
Scarlet 6R, 2R, etc.	Naphthol Yellow S
Naphthol Brown O	Naphthylamine Black
Acid Magenta	Naphthylamine Blue
Acid Rhodamine	Naphthol Red

125. Basic colors are used to a slight extent on wool, the process being somewhat similar to that with acid colors, except that 3 per cent of acetic acid (30 per cent) is employed in place of the sulphuric acid.

Some basic dyestuffs:

Auramine	Safranine
Induline Scarlet	Phosphine
Janus Red	Janus Blue
Janus Black	Janus Brown
Bismarck Brown	Methylene Blue
Malachite Green	Methyl Violet

126. Silk. Raw silk, as it appears on the market, is heavily coated with a glue-like substance which must be removed before the fiber can be subjected to the dyeing operations. This is accomplished by "boiling off" in a strong soap solution, and is also known as "degumming" or "stripping." When boiled off, silk will lose about 25 per cent of its weight. Should the scouring be limited to a loss of only 10 to 15 per cent of its weight, it is termed "soupling"; when silk is given only a very slight scouring, the result is known as "écru." The quality of soap employed is usually about 30 per cent of the weight of the fiber, and the liquor remaining is used for dyeing purposes, being known as "boiled-off liquor."

127. Silk resembles wool in its behavior toward mordants and dyestuffs, except that it has less affinity for acid colors and greater affinity for basic dyestuffs.

128. Boil off ten 5-gm. skeins of silk with 30 per cent of Marseilles soap, boiling for one-half hour with a dilution of 1000 cc.

129. Prepare a dye-bath with 100 cc. of the boiled-off liquor (from 128) and 300 cc. of water. Make acid with sulphuric acid. Raise the temperature to 100° F. and enter the boiled-off skein. After turning the skein

several times, withdraw it and add 1 per cent of the dye-stuff. Raise to 140° F. and finally finish at the boil, turning several times during the operation.

The following dyestuffs are suitable for the above:

Naphthol Yellow	Orange
Fast Red	Acid Violet
Acid Green	Palatine Black

130. Prepare a dye-bath with 100 cc. of boiled-off liquor (from 128) and 300 cc. of water. Make acid with acetic acid. Raise the temperature to 100° F. and enter the boiled-off skein. Then add 1 per cent of the dye-stuff. Raise to 140° F., and finally finish at the boil.

Suitable dyestuffs are:

Auramine	Rhodamine
Eosine	Magenta
Methyl Violet	Victoria Green

131. Cotton. Vegetable fibers like cotton consist principally of cellulose, a very inactive substance chemically. When treated with dilute mineral acids, there is little action until the fiber becomes dry, when it will weaken or fall to a powder. This destructive action is more marked at elevated temperatures and is called carbonization, because the powder formed, hydro-cellulose, is usually black, and was thought to be carbon. For this reason, cotton cannot be dyed in an acid bath. Dilute alkalies have no action on cotton, consequently they are employed in the dyeing operations.

132. Cotton yarns or fabrics should always be "boiled off" or "wetted out" before dyeing. To do this, the

stock should be boiled in water containing 2 gms. of soda ash per liter, and, when thoroughly wet, placed in fresh water until needed. It is well to boil out at least 1 dozen skeins before starting the tests to be applied to cotton.

133. Prepare a dye-bath with 2 per cent of dyestuff (substantive color), $1\frac{1}{2}$ per cent of sodium carbonate, and 30 per cent of Glauber's salt, using six 5-gm. skeins. Boil for half an hour, remove, rinse, and dry.

Suitable dyestuffs are:

Congo Red

Primuline

Benzo Orange

Diamine Black

Benzo Purpurine

Dianil Red

Oxy Diamine Brown

Oxy Diamine Black

134. Prepare a dye-bath as in 133, leaving out the sodium carbonate, and dye in it six skeins of worsted yarn. It will be noticed that the wool has taken up more color, indicating that vegetable fibers have less than affinity for dyestuffs do animal fibers. The reason for leaving out the sodium carbonate is that wool is soluble in alkaline solutions. The object of adding the Glauber's salt is to make the dye more insoluble and cause it to unite better with the fiber.

135. In judging the value of a dyestuff, several tests are always applied to determine its quality when submitted to normal wearing conditions.

136. Into a dye-pot containing boiling water introduce a skein of the dyed cotton yarn, and, at the same time, enter a skein of undyed cotton. Boil for fifteen minutes.

137. Repeat test 136, using dyed woolen yarn with undyed woolen. It will be noticed that the color has

been removed from the cotton to a far greater extent than from the wool; and that the undyed cotton yarn has become colored. This loss of color is known as bleeding, and in washed goods would be very objectionable.

138. Prepare a soap solution with 4 gms. of soap in 400 cc. of water, and heat to about 60° C. Now work in this solution a skein of dyed cotton yarn together with an undyed skein. At the end of ten minutes note any change that may have taken place. This test represents the process of washing, so that the less a color is stripped, the better is the dyestuff.

139. Many colors, when exposed to sunlight, undergo a change or are said to fade. This can be readily shown by placing a skein of dyed cotton in a wooden frame so arranged as to protect half of the skein and leave the other half exposed to the action of the sun. At the end of two days open the frame and note the results.

140. Expose a skein of dyed woolen yarn at the same time and compare the two skeins.

141. Dyed goods are often subjected to acid conditions and to perspiration, so that it is necessary to know what action is to be expected in such cases. To determine this, introduce a skein of dyed cotton yarn into a 10 per cent solution of acetic acid and work for ten minutes. Wring out the skein, wash, and dry it. If the color changes, it is not fast to acids.

142. Test a skein of dyed woolen yarn in the same manner.

143. It sometimes happens that a test for alkali is necessary. To accomplish this, the dyed cotton skein is worked for ten minutes in a hot 2 per cent solution of sodium carbonate.

144. Wool is soluble in hot alkali, but, before it dissolves, a peculiar action, known as "felting," is brought about. To show this, work a skein of dyed woolen yarn in a hot 2 per cent solution of sodium carbonate for ten minutes.

145. Some colors can be used in a cold solution, which, on certain fabrics, is quite advantageous. To carry out this test, stir up the dyestuff with an equal weight of caustic soda (70° Tw.) and dissolve the whole in boiling water. Now prepare a bath with cold water to which has been added 1 per cent of soap, 1 per cent of color, and 20 per cent of Glauber's salt. Use cotton for test.

Some suitable dyestuffs are:

Cotton Yellow G

Oxamine Red

Pyramine Orange

Oxamine Fast Claret

Cotton Rubine

Oxamine Brown

146. Spirit soluble colors are sometimes used for dyeing cotton and linen fabrics such as lace or other fine materials. To illustrate, prepare a bath with Bismarck Brown base, using 1 gm. of color to 100 cc. of alcohol. Keep in a well-stoppered bottle. Into a dye pan place 200 cc. of alcohol, add 50 cc. of dye solution, and work in this a 5-gm. skein of cotton yarn. (The solution in the dye-bath may be saved for further use.)

147. Oil soluble colors are also used to some extent for laces. They are prepared by rubbing up a spirit soluble color in oleic acid and then dissolving the resulting paste in benzine. To demonstrate this method, rub up 1 gm. of Methyl Violet base with 5 gms. of oleic acid and dissolve in 100 cc. of benzine. Now make up a dye-bath with 200 cc. benzine, using 1 per cent of the color,

and dip in it a 5-gm. skein of unboiled cotton yarn, working for five minutes, remove and dry.

148. Mercerization. When cotton is treated with a concentrated solution of caustic soda it contracts in length, becoming heavier and stronger. This was first noticed by John Mercer in 1850, and hence is known as "mercerization." The chemical change taking place is due to the formation of the compound $C_{12}H_{20}O_{10} \cdot 2NaOH$. This body when washed decomposes with the production of $C_{12}H_{20}O_{10} \cdot H_2O$, cellulose hydrate, or mercerized cotton.

149. Mercerized cotton has a much stronger affinity for substantive colors than ordinary cotton, and also has a greater combining power with mordants.

150. To 200 cc. of water add 40 gms. of caustic soda and allow to cool. Cut a piece of unbleached cotton cloth about 5 inches long by 1 inch wide. Wet out and immerse in the above solution for five minutes. Remove, wash thoroughly, and dry. Note the loss in measurement due to shrinkage.

151. Work four boiled-off skeins of cotton yarn in the above caustic soda solution for five minutes. On removing the skeins, wash them well in running water, then with water containing a little acetic acid, and again with fresh water. It will be noticed that the skeins have shrunk about 25 per cent of their length. If, however, these skeins had been stretched during the process of mercerization, a high finish or gloss would have resulted. The best grade of cotton for mercerization is that known as Egyptian.

152. Dye a skein of mercerized cotton with $\frac{1}{4}$ per cent of Benzo Purpurine and 10 per cent of Glauber's Salt in 400 cc. of water, boiling for one-half hour.

153. Dye a skein of boiled-off unmercerized cotton in the same manner. It will be noticed that the mercerized skein takes up more color and that the color is faster to washing than that produced on the unmercerized yarn.

154. Mordant two skeins of mercerized cotton by boiling for thirty minutes with 4 per cent of tannic acid, using 400 cc. of water.

155. Mordant two skeins of unmercerized, boiled-off cotton in the same manner.

156. Dye a skein of mercerized cotton, which has been mordanted with tannic acid, in a bath containing $\frac{1}{2}$ per cent of Malachite Green, using 400 cc. of water at 50° C., and work for one-half hour.

157. Dye a skein of unmercerized cotton, which has been mordanted with tannic acid, in the same manner as in 156. Note any difference in the depth of shade obtained.

158. Malachite Green is an example of basic color that is a direct dye for wool but not for cotton. These dyes can be dyed on cotton only by the aid of tannic acid.

159. Mordant five 5-gm. skeins of cotton yarn with 5 per cent of sumach in 400 cc. of water, boiling for fifteen minutes, or until "wetted out," and allow to stand one hour.

160. Dye one skein, which has been mordanted as above (159), with each of the following, using 2 per cent of the dyestuff, temperature 120° - 140° F:

Auramine

Safranine T extra

Malachite Green (all brands)

Methyl Violet R

161. Mordant five 5-gm. skeins of cotton yarn with 5 per cent of cutch in 400 cc. of water in the same manner as above.

162. Dye one skein which has been mordanted as above (161), with each of the following, using 1 per cent of the dyestuff:

Auramine	Malachite Green (all brands)
Safranin T extra	Methyl Violet R

These tests with sumach and cutch illustrate the application of the numerous commercial sources of tannic acid to dyeing and printing.

The examples given under tannic acid mordants serve to illustrate a large class of basic colors applied to cotton, and, as they all act in practically the same manner, it will not be necessary to give more along this line, except to include a list for those desiring to make a more exhaustive study of the subject.

A few other basic colors are:

Rhodamine (all brands)	Magenta
Brilliant Green Cryst	Janus Red B
Cotton Green	Janus Yellow
New Fast Blue	Naphthol Blue R T
Phosphine	Bismarck Brown

163. Metallic Tannates. By subjecting cotton, which has been mordanted with tannic acid, to a metallic salt treatment, an insoluble metallic tannate is produced, possessing a greater affinity for basic colors than the tannic acid alone. The process of converting the tannic acid into this condition is known as "fixing." The salts of antimony, tin, titanium, and iron are the ones most frequently employed.

164. Mordant two skeins of cotton yarn with 5 per cent of tannic acid in 400 cc. of water. Bring to a boil and allow to stand for one hour. Fix one of the skeins by working for twenty minutes in a bath containing 2 per cent of tartar emetic in 400 cc. of water.

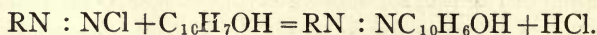
165. Prepare two dye-baths with 1 per cent of Methylene Blue in 400 cc. of water. Enter a skein of the mordanted yarn in each, raise to 70° C., and keep at this temperature for one-half hour. Squeeze out the excess of color, dye, and compare results.

166. Developed Dyes. Many of the substantive dyes may be applied to cotton in the usual manner and then changed by chemical treatment into other colors. These are usually much faster to washing and other agents than the original color. The process employed is known as "diazotizing" and "developing," and consists in subjecting the dyed skein to the action of nitrous acid, then subsequently to a developing agent. The colors which respond to this treatment are those containing the amino group NH_2 . This amino group, through the action of nitrous acid, becomes converted into the "diazo" group $\text{N} : \text{N}$. As these diazonium compounds are very unstable, the operation must be conducted in the cold and away from direct sunlight. The change which takes place may be represented by the following equation:



As soon as the cotton is diazotized, it should be immediately transferred to a developing solution. The function of the β -naphthol (or other developer) is to combine with the unstable diazo body to give the new and permanent coloring matter. This bath should also be kept cold.

The reaction of the developer may be illustrated by the following equation:



Some diazo colors are:

Sulphine	Dianil Black
Oxamine Blue	Dianil Ogene Sky Blue
Oxamine Violet	Oxy Diamine Ogene
Dianil Brown	Primuline

167. Enter five skeins of boiled-off cotton yarn into a bath containing 2 per cent of primuline, 25 per cent of salt, and 2 per cent of sodium carbonate in 400 cc. of water. Boil for one-half hour, remove, and wash.

168. To diazotize these skeins enter five of them into a bath containing 4 per cent of sodium nitrite, 50 per cent of hydrochloric acid with 400 cc. of cold water. Work for ten minutes, remove, and pass rapidly to the following solutions.

169. Hang one of the diazotized skeins in the sunlight for half an hour and develop as in (a) of 170 below.

170. Prepare three baths as follows, and enter a skein of the diazotized cotton in each. Work for ten minutes, wash, and dry.

(a) One per cent of β -naphthol and 1 per cent of sodium hydroxide in 400 cc. of cold water.

(b) One per cent of resorcin and 2 per cent of sodium hydroxide in 400 cc. of cold water.

(c) Two per cent of α -naphthol and 4 per cent of sodium hydroxide in 400 cc. of cold water.

171. Removal of Stains. Fruit or vegetable stains may be removed usually by washing with dilute oxalic acid or

vinegar. Dilute hydrochloric acid will also give the same result.

Most colors may be removed by bleaching powder and an acid. The best method is to dip the goods into the solution or emulsion of the chloride of lime and, after working for two or three minutes, ring out, then dip in a 3 per cent solution of oxalic acid and wash thoroughly. Hydrochloric or sulphuric acid, or even vinegar, may be used. This is, of course, for white goods which have been stained with colors or inks. Stains may be removed from the hands in the same manner.

Another easy method of removing stains from the hands is to wash in a 1 per cent solution of potassium permanganate, then in a 2 per cent solution of oxalic acid, and rinse thoroughly.

Stains caused by tannic acid, iron inks, and iron rust can usually be taken out with oxalic acid or potassium acid-oxalate. Many vegetable stains disappear after simply soaking in tepid water.

Mordant colors, as a rule, are more difficult to discharge from the hands or fabrics, but can be removed by the following treatment:

Place in the hand about 2 teaspoonfuls of chloride of lime and cover with about twice as much sodium carbonate. Just moisten (do not wet) with water and work into a paste. When the paste becomes warm rub over the hands or fabric like soap. Work until nearly dry, and then rinse. Follow this treatment with a strong solution of sodium bisulphite and, without rinsing, by a strong solution of oxalic acid. Wash well, and dry.

Grease spots, oils, paints, varnishes, and tar may be

removed by washing or rubbing with a cloth saturated with either naphtha, benzene, ether, chloroform, carbon bisulphide, carbon tetrachloride, turpentine, or common kerosene.

172. The Sulphur Dyestuffs on Cotton.—The colors belonging to this group are absorbed by cotton in a bath containing sodium sulphide.

The bath is prepared by boiling a solution containing from 2 to 10 per cent of dyestuff, 2 to 3 per cent of sodium carbonate, 5 to 10 per cent of sodium sulphide, and 10 to 20 per cent of sodium chloride, or Glauber's salt. The fiber is entered into the boiling bath and worked until finished, about $1\frac{1}{4}$ hours. It is very essential that, after dyeing, the cotton should be thoroughly washed before drying.

Examples. *Immedial Black*, 15 per cent; sodium sulphide, 10 per cent; soda ash, 5 per cent; Glauber's salt, 20 per cent. *Immedial Yellow*, 5 per cent; sodium sulphide, 5 per cent; soda ash, 5 per cent; Glauber's salt, 5 per cent. *Immedial Dark Blue*, 20 per cent; sodium sulphide, 20 per cent; soda ash, 10 per cent; Glauber's salt, 20 per cent. *Katigen Indigo, 2 R L extra*, 10 per cent; sodium sulphide, 20 per cent; soda ash, 6 per cent; Glauber's salt, 50 per cent. *Katigen Black T extra*, 7.5 per cent; sodium sulphide, 15 per cent; soda ash, 8 per cent; Glauber's salt, 60 per cent.

173. Vat Colors on Cotton and Wool. The most important dyestuff of this class is Indigo, produced upon the fiber by first treating with an alkaline solution of Indigo white, which, on oxidation in the air, is recon-verted into the blue. The term "vat" applies to the vessel employed for dissolving the Indigo, and also to

the solution itself, which may be prepared by the aid of various reducing agents.

174. *The Zinc Lime Vat.*

Indigo M.L.G. powder (or 25 gms. paste),	10 gms.
Zinc dust,	10 gms.
Quicklime,	30 gms.

Mix the zinc dust with 150 cc. of water at 50° C.; add the Indigo and then the lime with constant stirring. The whole is left to stand for six hours, but is stirred from time to time. The reduction is complete when a drop of the solution, placed on a sheet of glass, runs off as a yellow liquid that oxidizes in the air in the course of forty-five to fifty seconds.

The reduced solution is then diluted with 500 cc. of water and the cotton fiber dyed in it by thoroughly soaking. It is then wrung out, and dried in the air, the process being repeated until the requisite shade is obtained.

175. *The Sulphate of Iron Vat.* By following the same directions as above this bath may be prepared, using

Indigo M.L.B. powder,	10 gms.
Ferrous sulphate,	50 gms.
Quicklime,	60 gms.

176. *The Hydrosulphite Vat (for cotton).* This vat is based on the property of hydrosulphurous acid, $H_2S_2O_4$, of forming with Indigo a colorless, double compound, soluble in alkalies, and decomposed by the weakest oxidation, Indigo blue being thereby liberated.

For practical use the sodium salt of hydrosulphurous

acid, $\text{Na}_2\text{S}_2\text{O}_4$, is prepared by allowing zinc dust to act on sodium bisulphite in a tightly closed vessel, the mixture being kept stirred and cooled. The reaction is usually complete within four or five hours. The hydrosulphite solution is rendered slightly alkaline with milk of lime, in order to diminish its instability, and is then ready for use. The procedure is as follows:

Sodium bisulphite solution 71.4° Tw.,	100 parts
Cold water,	225 parts
and in the course of half an hour,	
Zinc dust stirred into this solution	$3\frac{3}{4}$ parts

The whole, which is stirred from time to time, is left to stand for four or five hours, and then mixed with

Lime, $11\frac{1}{2}$ parts, slaked in
Water, 30 parts.

After allowing the sediment to settle, the clear solution is decanted and 7 parts caustic soda solution, 36° Tw., added.

The hydrosulphite solution is now ready for use. It should stand at 19° - 20° Tw. and be kept in a tight bottle in a dark room.

177. *Preparation of Dye Vat.*

Indigo,	10 gms., mixed with
Hot water,	30 gms.; to this mixture add
Caustic soda, 76° Tw.,	85 gms. and stir well; after
heating to 50° C., add	
Hydrosulphite solution, 20° Tw.,	250 gms. and keep
the temperature at 50° C.	

In the course of the reduction which takes place, and which is tested in the usual way with a sheet of glass, more hydrosulphite solution is added in several portions. The reduced Indigo ought to run off from the sheet of glass as a yellow liquid which oxidizes in twenty to thirty seconds.

The cotton fiber is then dyed in the same manner as indicated in the case of the zinc vat.

178. *The Hydrosulphite Vat (for wool).* The process is essentially the same, except that the caustic soda must be replaced by milk of lime in sufficient amount to produce an alkaline reaction.

179. Turkey Red upon Cotton. The name "turkey-red" is applied to the color produced upon cotton by the aid of alizarine, alumina, lime, and fatty acid compounds. This fatty acid compound, known as "Turkey-red Oil," is prepared by treating olive or castor oil with concentrated sulphuric acid, usually 3 parts of acid to 10 parts of oil.

The acid is slowly poured into the oil with constant stirring, and the whole then allowed to stand until a sample of the product dissolves completely in water, whereupon it is poured into water and washed with a solution of sodium chloride until free from sulphuric acid.

During the process of mixing the temperature should not be allowed to rise above 40° C. Sometimes the last trace of acid is neutralized with ammonia.

The production of a good turkey-red upon cotton can be attained only by practice; the directions given should, however, yield fairly good results if carefully followed.

180. *Clearing.* Free the cotton fiber (5-gm. skeins)

from grease, etc., by boiling in a weak solution of sodium carbonate; wring out very thoroughly.

181. Oiling. Without drying, enter into a bath containing

Turkey-red Oil,	10 gms.
Water,	90 gms.

and work until thoroughly soaked. Wring out and dry in hot-air cupboard at 40° - 50° C.

Repeat this operation twice, drying between each immersion.

182. Alumina Mordant. Work the fiber through a bath of aluminium acetate, 9° Tw.; wring out and dry at 40° - 50° C.

Repeat this treatment.

183. Chalking. Enter the cotton into a bath containing

Chalk,	6 gms.
Water,	1 liter

at 30° - 40° C. Stir well for about half an hour, and then wash thoroughly in clean water. It is unnecessary to dry the cotton before dyeing.

184. Dyeing. Stir the dyestuff (15 per cent alizarine paste) into the water and enter the skein at 20° - 25° C. Work the skeins at this temperature for about twenty minutes, and then heat up so that in about half an hour the temperature rises to 60° C. Keep at this point for about an hour, then wring well and dry.

185. Steaming. Steam the dried cotton for one hour at a pressure of 15 lbs., or for two hours without pressure; then wash well.

186. Brightening. Brighten the dyed material in a closed apparatus at a pressure of 5 lbs. Work in a solution of 4.5 gms. of soap in 1 liter of water; leave the cotton in it for ten minutes and wash well.

187. Colors Produced Directly upon the Fiber by Oxidation. Aniline black, the typical member of this group, is produced directly on the cotton fiber by the oxidation of aniline, and the two following methods will suffice to indicate the manner in which dyestuffs of this kind are formed:

(a) *One-bath black.*

The bath should contain

Aniline,	5 per cent
Hydrochloric acid,	12 per cent
Dichromate of soda,	6 per cent
Copper sulphate,	5 per cent.

The cotton should be entered into the bath and worked for one hour; then the bath should be raised to boiling during another hour, and the fiber worked at this temperature for another half hour; wash and dry.

(b) *Oxidation black.*

Aniline hydrochloride,	126 gms.
Water,	300 cc.
Chlorate of potash,	40 gms.
Acetate of alumina, 21.6° Tw.,	150 gms.
Ammonium chloride,	5.7 gms.
Copper sulphate,	3 gms.

and the whole made up to 1 liter.

The fiber is thoroughly impregnated in this solution,

wrung out and dried. It is then worked for half an hour at 60° C. in a bath containing

Dichromate of soda,	2.5 per cent
Aniline hydrochloride,	.5 per cent
Sulphuric acid, .8° Tw.,	.2 per cent

and then washed and dried.

188. Cotton Printing. (a) *General Remarks.* The various methods of printing depend upon the use of a color solution to which a thickening agent has been added. In most cases fixing agents (acids, etc.), or substances with which the dyestuffs can form lakes (tannic acid, chrome compounds) are added, and the material is printed with this mixture. The process is usually carried out on the roller or cylinder machine. After printing, the goods are dried, or they are dried and slightly moistened again. They are then steamed with or without pressure. In the case of many dyestuffs the correct development of shade and proper fixation take place during this process. This is followed by an after treatment with fixing agents, the details of which depend upon the goods in question and the requirements. The goods are then rinsed, etc.

(b) *Thickening Agents.* In order to obtain good results it is necessary to choose a suitable thickening agent. Below are given several that may be considered typical of their group.

Tragacanth Solution. This is used as a thickening for printing basic colors on cotton and silk and is suitable for producing ground colors and very delicate light shades.

Gum tragacanth,	$\frac{1}{2}$ lb. gum
Water,	1 gallon

mix and stir at intervals for some time. The mixture is next boiled for four to six hours, and the water that has boiled off is replaced, so as to bring the mixture to about its original volume.

This is used with an addition of acetic acid for producing light shades with basic colors.

Acetic Acid-Starch-Dextrine Thickening:

Wheat starch,	24 gms.
Dextrine,	20 gms.
Water,	52 gms.
Tragacanth solution, 5 per cent,	40 gms.
Acetic acid, 9° Tw.,	60 gms.
Glycerine,	4 gms.

Boil ten minutes.

Weight of cold thickening, 176 gms.

A soft smooth thickening suitable for printing Methylene Blue on cotton.

Egg Albumen Thickening:

Egg albumen,	40 gms.
Cold water,	38 gms.
Ammonia liquor,	2 gms.

are mixed together and allowed to stand for twelve hours, after which the mixture is sieved or passed through calico. This serves as a thickening for the substantive and pigment colors. As a rule it is used only for clear, light shades.

Casein Thickening:

Powdered casein,	15 gms.
Cold water,	84 gms.
Ammonia liquor,	1 gm.

This thickening is used instead of albumen for cheap articles.

(c) *General Methods of Preparing Basic Colors:*

Color,	10 gms.
Acetic acid, 9° Tw.,	100 gms.
Water,	130 gms.
Thickening,	700 gms.
Tannic acid,	30 gms.
Acetic acid, 9° Tw.,	30 gms.

When preparing the printing pastes, the dyestuff is either dissolved in water and a little acetic acid, and this solution then added to the thickening, which has already been prepared, or the thickening agents are mixed in the color solution and boiled with the necessary amount of water and acetic acid. In both cases, the tannic acid necessary for the formation of the color lake is dissolved in an equal weight of acetic acid and then slowly stirred into the printing paste, but not until this has cooled down.

After printing, the pieces are dried, steamed for one-half to one hour without pressure, and then worked for five to ten minutes in a lukewarm solution containing 100 gms. of tartar emetic to every 40 liters of water, in order to fix the color completely. They are then rinsed and soaped.

(d) *General Method of Preparing the Colors of the Eosine Group:*

Color,	20 gms.
Hot water,	230 gms.
Thickening,	600 gms.
Acetate of alumina, 15° Tw.,	50 gms.
Acetate of magnesia, 15° Tw.,	100 gms.

Print, dry, steam for one-half hour at one-half atmospheric pressure, but do not rinse.

(e) *General Method for Substantive Dyes with Albumen:*

Color,	20 gms.
Hot water,	250 gms.
Glycerine,	30 gms.
Egg albumen 1 : 1,	700 gms.

stir the mixture well.

Print, dry, and steam for one-half hour at 5 lbs. pressure, and rinse.

Those who desire to make a more exhaustive study of this subject should refer to the literature published by the various color companies.

CHAPTER V

PIGMENTS AND LAKES

189. Pigments. Pigments are mineral or organic bodies, usually insoluble in water, oils, or other neutral solvents. They are used to impart color to a base by admixture with its substance. The color of a pigment depends upon the amount and kind of light it reflects. It is necessary that the pigment be opaque in order to give good "covering power," that is, it should entirely conceal the surface to which it is applied.

The chief pigments are given in the following table:

<i>Whites</i>	<i>Blues</i>	<i>Greens</i>
White Lead	Ultramarine	Ultramarine
Lead Sulphate	Prussian Blue	Brunswick Green
Lead Oxychloride	Smalt	Chrome Green
White Zinc	Cobalt Blue	Guignet's Green
Zinc Sulphide	Copper Blue	Copper Greens
Barytes	Indigo	Arsenic Green
Gypsum	Chinese Blue	Violet Lake
Whiting	<i>Violet</i>	
Lithopone	Ultramarine	
<i>Yellows</i>	<i>Reds</i>	<i>Blacks</i>
Chrome Yellow	Red Lead	Lamp Black
Yellow Ocher	Chrome Red	Ivory Black
Cadmium Yellow	Red Ocher	Bone Black

<i>Yellows</i>	<i>Reds</i>	<i>Blacks</i>
Orpiment	Venetian Red	Graphite
Litharge	Vermilion	
Gamboge	Realgar	
Indian Yellow	Antimony Red	
	Carmine	
	<i>Orange</i>	<i>Browns</i>
	Orange Mineral	Umbers
	Chrome Orange	Vandyke Brown
	Antimony Orange	Sepia

Many other pigments, both natural and artificial, are in use, but as they do not have a distinctive color, they are not included in the above classification. For a detailed description of these pigments consult references in the Manual of Industrial Chemistry.

The preparation of some typical pigments will be taken up, illustrating the method of handling artificial colors.

190. Prussian Blue. Dissolve 1000 gms. of sulphate of iron (*ferrous*) in 15 liters of water and add 50 gms. of hydrochloric acid. To this solution add a solution of 1000 gms. of ferrocyanide of potassium and 50 gms. of hydrochloric acid in 30 liters of water. Now slowly add to the mixture 400 gms. of bleaching powder, when the white precipitate will be changed to a blue one. To hasten the oxidation, it is well to boil with open steam for a short time. This pigment being finely divided is slow to settle at first, and should be allowed to stand for several days. When the Prussian Blue has settled somewhat, the top liquid is siphoned off, and the tank again filled

with water. This process of washing is continued until the test of the wash-water shows only slight traces of sulphates. The pigment is next passed into the filter press, washed again, removed, dried, and ground to a fine powder in the ball mill.

191. Red Oxide. Place about 1000 gms. of ferrous sulphate in an iron crucible and heat until nearly all of the fumes of sulphuric anhydride are given off. Then add 100 gms. of slaked lime and heat until uniform color is obtained.

192. Vermilion. Put 300 gms. of mercury and 114 gms. of sulphur into a mortar and triturate until the ingredients are thoroughly incorporated. Dissolve 76 gms. of caustic potash in a 14-inch evaporating dish, using 600 cc. of water, and add the contents of the mortar. Heat to 45° C. and maintain this temperature for several hours, stirring very thoroughly at frequent intervals and keeping the volume of water constant for the first two hours. Regulate the temperature so that it will not exceed 45° C., nor fall below 40° . In the course of two or three hours the mass becomes brown and then gradually turns a bright red. When the desired vermilion color is acquired, usually after six or eight hours, wash by decantation until free from alkali. Filter by means of the press, and dry on glass plates at not over 45° .

193. Antimony Vermilion. One hundred and forty gms. of sodium thiosulphate is dissolved in 250 cc. of water, making a solution of 40° Tw. One hundred cc. of antimony chloride is mixed with 40 cc. of water and added to the thiosulphate placed in a casserole. This is slowly heated, and when the red color is developed, it is filtered.

194. Scheele Green. Dissolve 150 gms. of arsenious oxide in 1000 cc. of water containing 200 gms. of sodium carbonate. Next dissolve 150 gms. of copper sulphate in 2000 cc. of water, and when both solutions are ready, mix them together, allow them to settle, wash, and filter.

195. Chrome Yellow. Dissolve 2000 gms. of lead acetate in 8 liters of water and filter if necessary. Then dissolve 1550 gms. of sodium dichromate in 8 liters of water and mix the two solutions. Wash by decantation, filter, and dry. If all of the color is not precipitated, add a little more lead acetate.

196. Chrome Red. Weigh out 500 gms. of lead acetate and 175 gms. of sodium dichromate; dissolve each separately in 10 liters of water, then mix the two solutions. Allow the precipitate of chrome yellow to settle out, pour off the supernatant liquor, add a solution of 75 gms. of caustic soda in 500 cc. of water, and boil for about one-half hour, or until the yellow has assumed a red color, then filter, wash, and dry.

197. Chrome Orange. Dissolve 500 gms. of lead acetate in 10 liters of water; next weigh out 125 gms. of sodium dichromate and 125 gms. of sulphate of soda and dissolve in 10 liters of water. Mix the two solutions together and allow the precipitate to settle. Pour off the clear liquor, then add a solution of 50 gms. of caustic soda in 1000 cc. of water, boil the mixture for about twenty minutes, filter, wash, and dry.

198. Satin White. Weigh out 800 gms. of lime and, after slaking with water, make into a thin cream with more water. Weigh out 1700 gms. of aluminium sulphate and dissolve in 5 liters of water. Mix the two and heat

to a boil; then allow to settle. Decant the clear liquor, wash, and allow to settle. Filter and dry.

199. Lithopone. Into a large crucible or muffle furnace place 500 gms. of barium sulphate and an equal quantity of finely ground soft coal. Place the crucible in the furnace and heat to dull redness until a test sample shows that the sulphate has been converted to sulphide. Remove the heat and, when the mass has cooled, dissolve in water, filter off the insoluble material, and concentrate the filtrate to a density of about 15° Bé. Now prepare a solution of zinc sulphate of about the same strength and add a sufficient quantity to combine with the barium sulphide present. Filter off the precipitate, which consists of a mixture of barium sulphate and zinc sulphide, wash until free from zinc sulphate, and dry. The dry powder is again placed in a muffle furnace or crucible and heated to dull redness. While still hot, it is plunged into water, and the precipitate is filtered off, dried, and ground.

200. Chrome Green. This is made by heating chromium hydroxide.

201. Brunswick Green. Four shades of Brunswick Green may be made by using the proportions given under the following separate heads. In each case, however, the sulphate of iron and lead acetate are dissolved in separate vessels and added to the suspended barytes, whereas the yellow prussiate of potash and dichromate of soda may be dissolved together and added to the above mixture.

202. Pale Brunswick Green. One thousand gms. of barytes, 130 gms. of acetate of lead, 10 gms. sulphate of iron, 10 gms. potassium ferrocyanide and 40 gms. dichromate of soda.

203. Medium Brunswick Green. One thousand gms. barytes, 135 gms. acetate of lead, 15 gms. of sulphate of iron, 15 gms. potassium ferrocyanide, 45 gms. dichromate of soda.

204. Deep Brunswick Green. One thousand gms. of barytes, 140 gms. acetate of lead, 20 gms. ferrous sulphate, 20 gms. of potassium ferrocyanide, 45 gms. of dichromate of soda.

205. Extra Deep Brunswick Green. One thousand gms. of barytes, 100 gms. acetate of lead, 40 gms. of ferrous sulphate, 40 gms. of potassium ferrocyanide, and 50 gms. of dichromate of soda.

206. Lakes. Color lakes are insoluble compounds formed by precipitating suitable dyestuffs on an insoluble base. This base is similar in action to the fibers in ordinary dyeing. According to its nature, it may assist in the preparation of the dyestuffs, or may act merely as a carrier for the color. The physical condition of this base is also important, as it affects the covering power, cheapness, and character of the pigments produced. The precipitants used are such chemicals as will render the dyestuffs insoluble. Those ordinarily employed are barium chloride, calcium chloride, lead acetate or nitrate, zinc sulphate, tannic acid, etc. The lakes are precipitated according to the particular directions, washed, filtered, and either dried or used in pulp form. These lakes are used for various purposes, and the characteristics must, therefore, be suitably varied. They are usually classified according to their method of practical application. The general classes are as follows: Colors for the manufacture of lithographic inks, paint colors, colors for kalsomine or wall finishes, colors for wall-paper and coated-paper surfaces.

207. Crimson Lake. This is one of the most important lakes; and its preparation takes place in three stages: first, the preparation of the cochineal liquor; second, the preparation of the hydroxide of aluminium; and third, the combination of these two.

Weight out 200 gms. of cochineal, mix with 2000 cc. of water, and boil for five minutes; add 30 gms. of cream of tartar and boil again; then add 4 gms. of alum, boil, strain, and put into the decoction 50 gms. more of cream of tartar. The cochineal liquor is ready. To prepare the alumina, weigh out 420 gms. of alum (or its equivalent of sulphate of aluminium), dissolve in 3000 cc. of hot water, and add this to 140 gms. of ammonium carbonate in 2000 cc. of hot water, stirring well; then wash thoroughly with hot water. This alumina precipitate is next mixed with the cochineal liquor and the whole boiled together. When the crimson lake is formed it is filtered off, washed, and dried.

208. Some Basic Colors Used for Lakes. The use of basic colors in producing lakes depends upon their affinity for acid radicals and is best illustrated by example.

209. Violet Lake. Weigh out 100 gms. of barytes and mix with 500 cc. of boiling water. Add to this mixture 2 gms. of Methyl Violet and stir well. When the dye has dissolved, add 2 gms. of tartar emetic, then a solution of 3 gms. of tannic acid in 100 cc. of hot water. If on settling, the top liquor is highly colored, it is an indication that insufficient tannic acid has been added. If necessary, add more tannic acid, wash, filter, and dry.

The following lakes may be made in a similar manner, using the foregoing method.

210. Blue Lake. One hundred gms. of barytes, 3 gms. tartar emetic, 3 gms. Methylene Blue, 6 gms. tannic acid.

211. Crimson Lake R. One hundred gms. barytes, 3 gms. tartar emetic, 3 gms. Safranine, 6 gms. tannic acid.

212. Brown Lake. One hundred gms. of barytes, 3 gms. tartar emetic, 3 gms. Bismarck Brown, and $4\frac{1}{2}$ gms. of tannic acid.

213. Scarlet Lake. One hundred gms. barytes, 3 gms. of tartar emetic, 1 gm. Safranine, 2 gms. Auramine, and $4\frac{1}{2}$ gms. tannic acid.

214. Some Acid Colors Used for Lakes. The following is a list of acid colors adapted for the manufacture of lakes precipitated with barium chloride. The example given will serve for the entire list.

Mix 15 parts of sulphate of aluminium in 150 parts of water with a solution of 10 parts of Acid Green L in 100 parts of water. Then precipitate with 32 parts of barium chloride in 640 parts of water. On adding $6\frac{1}{2}$ parts of soda ash in 65 parts of water, the alumina is precipitated in the lake, thus producing a complete separation.

215. Reds. Acid Magenta; Azo Cochineal; Azo Crimson S; Azo Eosine; Azo Phloxine 2 G; Bordeaux Extra; Brilliant Crocein 3 B; Fast Red A B T; Ponceau 2 R L, 5 R L.

216. Orange. Orange II, B, G L; Mandarin G.

217. Yellow. Fast Yellow Extra; Fast Light Yellow; Naphthol Yellow S.

218. Greens. Acid Green L; Brilliant Acid Green 6 B; New Acid Green 3 B X, G X.

219. Blues. Alizarine Blue S A P, S H E; Cotton Blue; Fast Blue Greenish; Gallocyanin; Indulin B.

220. Violets. Acid Violet 4 B, Extra, 1 R Ext. 8 B; Alkali Violet; Fast Acid Violet.

221. Brown. Fast Brown.

222. Grays. Acid Black 8 B.

223. The Alizarine Colors are very fast to light and water and are extensively used in paints as well as lithographic inks, etc. An example will serve for the class:

I

Mix 243 cc. of a 10 per cent solution of aluminium sulphate,
125 cc. of a 10 per cent solution of soda ash.

Wash out three times. To this add

30 cc. of a 10 per cent solution of calcium chloride,
70 cc. of a 10 per cent solution of sodium phosphate.

Wash twice and add

3 cc. of a 10 per cent acetic acid solution.

II

Dissolve or stir up

Alizarine red,	30 parts
Turkey-red Oil,	5 parts
Calcium chloride, 10 per cent solution,	5 parts
Water,	2000 parts

Add I to II and allow to remain for some time; afterwards, bring to a boil, boil for several hours, or until the red is completely developed, wash and filter.

224. Colors which are Precipitated with Lead Salts. Use 30 parts of aluminium hydroxide, 20 parts of barytes, $1\frac{1}{2}$ parts of color, 1 part of lead nitrate.

All acid colors given above; also, Uranine, O N; Eosine, all brands; Erythrosine, all brands; Phloxine, all brands; Rose bengal, G B.

225. Crimson Red. Dissolve 200 gms. of logwood extract in 2000 cc. of hot water and add a solution of 150 gms. of sulphate of aluminium in 2000 cc. of water. Mix well and introduce 65 gms. of soda ash dissolved in 1000 cc. of water. Boil for ten or fifteen minutes, wash, and filter.

Ferric chloride gives a bluish black precipitate under the same conditions; copper sulphate, a violet black; antimony chloride, a violet; chromic acid or dichromate, a bronze black precipitate.

By mixing various amounts of fustic or Persian berry, the shade may be modified.

226. Para Red. Dissolve 70 gms. of para-nitraniline in 2000 cc. of water to which has been added 150 gms. of concentrated hydrochloric acid. Heat until all of the para-nitraniline has dissolved, cool below 40° F. with ice, and slowly add a solution of 50 gms. of sodium nitrate dissolved in 200 cc. of water. Now stir in 150 gms. of sodium acetate in 1000 cc. of water, and 1000 gms. of blanc fixe. While the above is standing for one hour, prepare a solution of 70 gms. of β -naphthol in 2000 cc. of water, to which has been added 30 gms. of caustic soda. Cool with ice to below 40° F. and slowly add, with constant stirring, to the foregoing solution. Filter off the red lake produced, wash well, and dry at a low temperature.

α -naphthylamine may be used in place of para-nitraniline, and in this case a crimson lake is obtained. Different shades may also be secured by using other reducing agents in place of β -naphthol.

CHAPTER VI

DRIERS, VARNISHES, PAINTS AND STAINS

227. Driers. When some substances such as acetate of lead, acetate of manganese, red lead, manganese borate, etc., are added to paints and varnishes, the latter become dry or hard much quicker than otherwise. Linseed oil, mixed or heated with such bodies, dries much quicker. From this fact has arisen the practice in the manufacture of paints and varnishes of adding so-called "driers" for the purpose of facilitating the drying or hardening.

A very small quantity of drier will cause the drying of a large quantity of oil; from 0.5 to 1 per cent of the weight of the oil is usually sufficient. Manganese compounds are more energetic drying agents than lead compounds. Driers that dissolve easily in the oil, such as linoleates and resinates, are more active than those which, like red lead or manganese, do not dissolve in the oil.

When raw linseed oil is mixed with lead oxide or manganese borate, very little is taken up, and the rapidity of drying is not much increased; but, if the oil be heated with them, they are more readily dissolved, and the treated oil dries much more rapidly.

Some of the substances used for driers are acetate of lead, red lead, orange lead, white lead, chrome yellow, litharge, manganese dioxide, manganese sulphate, manganese acetate, ferrous sulphate, and ferric oxide. Of

late years, however, the linoleates, resinates, and borates of lead and manganese have come into common use, on account of the fact that they dissolve completely in oil, so that no "foots" are produced. Also, since they are soluble, no long heating of the oil is needed and paler oils can be produced. Some of these driers are prepared in the following manner:

228. Lead Borate. Dissolve 10 lbs. of acetate of lead in 10 gals. of water and add to it a solution of $6\frac{1}{2}$ lbs. of borax in 10 gals of water; wash, filter, and dry.

229. Manganese Borate. Dissolve 10 lbs. of manganese sulphate in 10 gals. of water and add to it a solution of 8 lbs. of borax in 10 gals. of water; wash, filter, and dry.

230. Lead Linoleate. This is made by preparing a neutral soda soap from linseed oil and caustic soda and pouring this into a solution of lead acetate.

231. Manganese Linoleate. Made in a manner similar to the lead compound.

232. Lead Resinate. This substance is prepared by making a rosin soap and adding it to a solution of lead acetate.

233. Manganese Resinate. Made in a similar manner, using manganese sulphate.

234. Linseed Oil Drier. Linseed oil that has been strongly boiled with a large proportion of drier and then mixed with turpentine, forms a liquid drier which mixes freely with paint, and hence is very serviceable for the use of the painter.

235. Heavy Drier No. 1. Put 10 gals. of raw linseed oil into a copper kettle and heat under the stack, slowly adding 1 lb. of borate of lead. Boil until clear, and,

when cooled sufficiently, slowly add 10 gals. of turpentine and stir well.

236. Drier No. 2. Dissolve as much lead resinate in turpentine as possible and use only in the cold. Of this drier, 5 per cent of the amount of oil contained in the paint is used.

237. Japan Drier.

Raw linseed oil,	2½ gals.
Flake litharge,	7½ lbs.
Kauri gum,	3½ lbs.
Granulated manganese,	2 lbs.
Turpentine,	10 gals.

Cook the oil, litharge, gum, and manganese together until a drop when cold is brittle. Then, after cooling the mass to between 250° and 300° F., add the turpentine slowly.

238. Fine Light Coach Oil. Place in the varnish kettle

Raw linseed oil,	6½ gals.
Borate of manganese,	2 oz.
Litharge,	6 oz.

Heat to 375°-450° F. for several hours, or until a sample placed on glass becomes solid within a few minutes.

239. Medium Coach Oil. Following the above directions, use

Raw linseed oil,	6½ gals.
Borate of manganese,	2 oz.
Red lead,	4 oz.
Litharge,	6 oz.

240. Strong Coach Oil.

Raw linseed oil,	6½ gal.
Borate of manganese,	2 oz.
Red lead,	8 oz.
Litharge	8 oz.
Black oxide of manganese,	8 oz.

241. Paint and Varnish Remover. In order to remove paint and varnish from wood or other material, it is necessary to treat it in such a manner as to soften the coating. When the coating or film has been softened, it becomes an easy matter to remove it by means of a flat putty knife. There are many materials that will have the desired effect, but some of them possess marked disadvantages. Caustic soda solution, or a solution of washing soda, will readily soften the film, but as they have a harmful action on the wood, their use is not to be recommended. A most effective varnish remover may be made by mixing together

Benzene, 5 parts
Acetone, 5 parts
Alcohol, 5 parts

Then melt 1 part of paraffine wax and, while still liquid, pour into the mixture. This preparation is applied with a brush, allowed to stand for ten minutes, and then scraped off with a knife.

The use of wax in varnish remover is covered by a basic patent. This has resulted in much litigation on account of the fact that it is impossible to make a satisfactory remover without the admixture of some medium

capable of holding the solvent in place until it has done its work.

242. Rust Remover. Powdered emery and paraffine oil.

243. Protective Varnish for Iron. Dissolve 1 lb. of paraffine wax in 1 gal. of benzine.

244. French Polish. This polish is prepared by dissolving

Gum copal,	4 oz.
Gum arabic,	3 oz.
Gum shellac,	1 lb. in
Alcohol,	1 gal.

245. Spirit Varnishes. These varnishes are made in the cold and are useful when a quick-drying varnish is desired.

246. Shellac Varnish. Dissolve 4 lbs. of shellac in 1 gal. of alcohol.

247. French Varnish. Dissolve 5 lbs. of white shellac in 1 gal. of alcohol, filter, and add 5 oz. Venice turpentine.

248. Brown Hard Spirit Varnish. Dissolve 1 lb. of sandarac, 2 lbs. of orange shellac, 4 oz. gum elemi, and 8 oz. of Venice turpentine in 1 gal. of alcohol.

249. Mastic Varnish. Dissolve $4\frac{1}{2}$ lbs. of gum mastic in 1 gal. of turpentine.

250. Dammar Varnish. Dissolve 4 lbs. gum dammar in 1 gal. of warm turpentine.

251. Collodion Varnish. Dissolve 4 oz. of gun-cotton in a mixture of $2\frac{1}{2}$ qts. of amyl alcohol and $1\frac{1}{2}$ qts. of amyl acetate.

252. Oil Varnishes. Oil varnishes are divided commercially into a number of groups, although there is very little difference between them, either in the method

of making or in the materials used. The general process in outline consists of the following stages: (1) Melting the resin or gum (or "running," as it is known in the trade); (2) boiling the oil; (3) mixing the melted gum and boiled oil; (4) boiling the varnish; (5) thinning the boiled product; (6) clearing.

(1) *Running*. The running of the gum is carried out in copper kettles heated by direct flame. This part of the process must have the utmost care, as overheating will give a poor grade of varnish, and underheating will not give a uniform product. To make a good varnish, it requires experience and a knowledge of just when to stop heating.

(2) *Oil Boiling*. While the gum is being "run," the oil to be used is boiled. This is done in the boiling pot. The oil is heated to about 500° F. for one to two hours, when it is ready to mix with the gum.

(3) *Mixing*. When the gum has been properly melted, the necessary quantity of boiled oil is poured into the melted gum with constant and vigorous stirring.

(4) *Varnish Boiling*. When the gum and oil are first mixed, a rather cloudy mass is produced, and, in order to make this transparent and to make the varnish string, it is necessary to boil again. The point at which to stop boiling depends upon the varnish; but as a rule, if a drop taken between the thumb and finger forms strings when the fingers are separated, the end point is indicated.

(5) *Thinning*. After the boiling operation is complete, the pot and its contents are removed from the fire and placed at some distance from any flame. Here it is mixed with turpentine to a working consistency. Although it is necessary to mix the turpentine with the varnish at as

high a temperature as possible, nevertheless, it is advisable to allow the hot mass to cool down somewhat before adding the turps (350° F.). The turpentine should be introduced in small quantities at a time, in order that the mixing may be properly done without too great a loss of volatile solvent.

(6) *Clearing and Aging.* Freshly made varnish is most unsatisfactory for use, and all the best qualities are subjected to an aging and clearing process before they are sent out. Thus, on standing, the more insoluble particles separate, and the top layer becomes clear.

The following list will serve to demonstrate the manufacture of oil varnishes in general. The preparation of materials and compounding of varnishes therefrom cannot be set forth actually so as to produce commercial products at all times, owing to the variation of the raw materials. With a little experience, however, the few formulas given below will be found to give satisfactory results.

253. Finishing Body Varnish for Coaches. Melt or run 8 lbs. of best African animi, pour in 160 lbs. of boiled linseed oil, and boil slowly for four to five hours, or until it strings well. Allow to cool (350° F.) and add 28 lbs. of turpentine.

254. Elastic Carriage Varnish. Run 8 lbs. of gum copal, mix with 20 lbs. of boiled linseed oil, add $2\frac{1}{2}$ oz. of dried copperas and $2\frac{1}{2}$ oz. of litharge, boil until it strings, cool, and thin with 44 lbs. of turpentine.

255. Extra Fine Copal Varnish.

	African copal,	15 lbs.
	Fine light coach oil (hot),	30 lbs.
thin with	Turpentine,	48 lbs.

256. Elastic Varnish.

	African copal,	5 lbs.
	Animi gum,	5 lbs.
	Medium coach oil (hot),	30 lbs.
thin with	Turpentine,	48 lbs.

257. Tool Varnish.

	Gum kauri,	15 lbs.
	Rosin W G,	15 lbs.
	Strong coach oil (hot),	12 lbs.
thin with	Turpentine,	29 lbs.
	Benzine,	24 lbs.

258. Waterproof Varnish. Within the past few years Chinese Wood Oil has come into extensive use in the preparation of varnishes for the reason that it can be used in conjunction with ordinary rosin. The oil is heated to about 400° F. in the presence of rosin and then thinned with turpentine. A great difficulty is encountered in making this varnish on account of the tendency of the oil to polymerize or become solid. When this happens, the batch is worthless.

259. Lithographic Varnish. In making this varnish a great deal depends upon the time of treatment and the temperature attained. The longer the treatment and the higher the temperature, the thicker will be the resulting product.

In making a standard varnish, place the linseed oil in a copper kettle and slowly raise the temperature to 550° F., holding it at this point for about one hour. Now lower the heat and allow the varnish gradually to cool. The result should be a fairly heavy-bodied product.

260. Ready-mixed Paint. There is hardly an industry which has made more rapid progress in the past fifteen years than the manufacture of "ready to use" paints. Nearly every user of paint goes through the stage of desiring to become his own paint manufacturer, and there

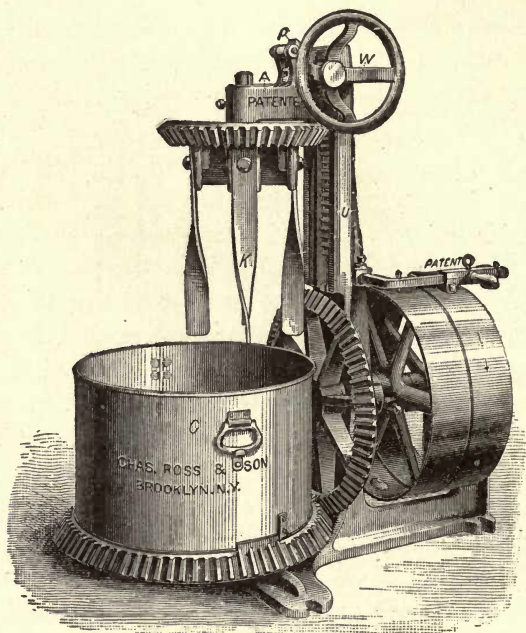


FIG. 29.

are, even at present, many who fondly imagine that they can prepare, by means of a wooden paddle and a wash-tub, mixed paints capable of ranking as protective and decorative agents with that material compounded by the aid of powerful and special machinery.

There has been much dissatisfaction in regard to

ready-mixed paints, and justly, as numerous brands have been placed upon the market that are not much better than chalk and water. Conscientious manufacturers, however, have done much to bring these frauds to light; and we may look to a time in the not far distant future when all brands of paint will be properly labeled and the public educated to the requirements of a ready-to-use paint. In fact, many states already have pure paint laws.

261. In the manufacture of ready-mixed paint, the necessary amount of raw linseed oil is placed in the mixing machine, Fig. 29. The body containing the coloring matter is introduced in small portions at a time, new lots being added only as the paste becomes limp. The mixture is then run through the mill, Fig. 30, until perfectly smooth when rubbed out on glass. It is then thinned as desired.

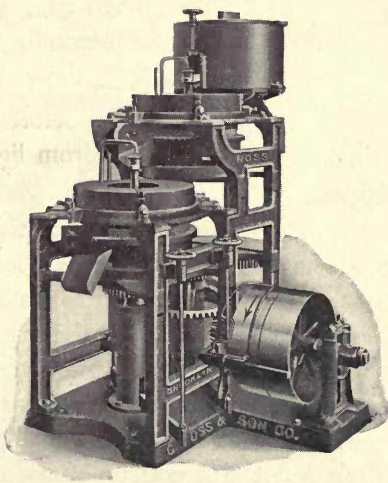


FIG. 30.

The method of procedure varies to quite an extent in different plants, although all are based on the same general principle.

From the following formulas the general method may be noted, and various shades produced.

262. White. Pour into the can of the mixer 11.6 lbs. of raw linseed oil and set the machine in motion. Now add slowly a mixture of

Dry white lead,	25 lbs.
Zinc oxide,	25 lbs.

Mix thoroughly, or until a smooth paste is obtained which will run easily from the can. This paste is passed through the mill, the stone being so adjusted as to secure a product free from lumps when rubbed out on glass. After passing through the mill, it is brought into the mixer again and thinned by adding

Raw linseed oil,	15 lbs.
Boiled linseed oil,	10 lbs.
Japan drier,	3.5 lbs.
Turpentine,	1.5 lbs.

When thoroughly mixed it is run into cans and is ready for use.

A quantity of the above thinning mixture should be made up, kept in stock, and labeled *Standard Oil*, to be subsequently referred to as such. The following formulas should be worked out in the same manner as just given for white, taking the precaution to have the mill thoroughly cleaned from one color before introducing another. A good plan also is to run from light to darker shades, thus obviating the necessity of so much cleaning of the mill and mixer. Where facilities will permit, it is advisable to use one mixer and mill for each color manufactured.

263. Outside White.

Linseed oil,	11½ lbs.
White lead,	21 lbs.
Zinc oxide,	18 lbs.
Whiting,	4½ lbs.
Barytes,	3 lbs.

thin with	Linseed oil, (raw)	15 lbs.
	Turpentine,	1 $\frac{3}{4}$ lbs.
	Japan drier,	$\frac{3}{4}$ lb.

264. Light Colonial.

	Dry white lead,	25 lbs.
	Zinc oxide,	25 lbs.
	Lemon chrome yellow,	1 lb.
	Raw linseed oil,	12 lbs.
thin with	Standard oil	27 lbs.

265. Cream.

	White lead,	25 lbs.
	Zinc oxide,	25 lbs.
	Lemon chrome yellow,	.6 lb.
	Medium chrome yellow,	.3 lb.
	Raw linseed oil,	11.5 lbs.
thin with	Standard oil,	26.5 lbs.

266. Yellow.

	White lead,	20 lbs.
	Zinc oxide,	20 lbs.
	Chrome yellow,	1 lb.
	Raw linseed oil,	11.5 lbs.
thin with	Standard oil,	26.0 lbs.

267. Straw.

	White lead,	20 lbs.
	Zinc oxide,	20 lbs.
	Golden ocher,	18 lbs.
	Orange yellow,	1 lb.
	Raw linseed oil,	14 lbs.
thin with	Standard oil,	28.5 lbs.

268. Flesh.

	White lead,	20	lbs.
	Zinc oxide,	20	lbs.
	Golden ocher,	5	lbs.
	French ocher,	2.9	lbs.
	Princess metallic,	.3	lb.
	Lamp-black,	.1	lb.
	Raw linseed oil,	11.5	lbs.
thin with	Standard oil	26	lbs.

269. Light Blue.

	White lead,	22.5	lbs.
	Zinc oxide,	22.5	lbs.
	Chinese blue,	5.0	lbs.
	Raw linseed oil,	11.5	lbs.
thin with	Standard oil,	30.0	lbs.

270. Dark Blue.

	White lead,	20	lbs.
	Zinc oxide,	20	lbs.
	Prussian blue,	10	lbs.
	Raw linseed oil,	11.5	lbs.
thin with	Standard oil,	30.0	lbs.

271. Azure Blue.

	White lead,	25	lbs.
	Zinc oxide,	25	lbs.
	Chinese blue,	.1	lb.
	Lemon chrome yellow,	.3	lb.
thin with	Standard oil,	26.0	lbs.

272. Drab.

	White lead,	24.5 lbs.
	Zin oxide,	24.5 lbs.
	Lamp-black,	.2 lb.
	Raw linseed oil,	11.5 lbs.
thin with	Standard oil,	30.0 lbs.

273. Slate.

	White lead,	22.5 lbs.
	Zinc oxide,	22.5 lbs.
	Lamp-black,	2.0 lbs.
	Raw linseed oil,	11.5 lbs.
thin with	Standard oil,	30.0 lbs.

274. Cement Paint (Drab).

	Linseed oil,	30 lbs.
	White lead,	25 lbs.
	Sub. white lead,	25 lbs.
	Zinc oxide,	35 lbs.
	Barytes,	15 lbs.
	Drop black	2.5 lbs.
	Chrome yellow,	1.2 lbs.
thin with	Linseed oil,	35.0 lbs.
	Turpentine,	8.0 lbs.
	Japan drier,	4.0 lbs.

275. Machine Gray.

	Linseed oil,	11 lbs.
	White lead,	20 lbs.
	Zinc oxide,	20 lbs.
	Graphite,	1 lb.
	Lamp-black,	.5 lb.
	Chinese blue,	.2 lb.

thin with	Linseed oil,	15 lbs.
	Japan drier,	2 lbs.
	Turpentine,	1 lb.

276. Dark Lead.

	White lead,	21.2 lbs.
	Zinc oxide,	21.2 lbs.
	Golden ocher,	6.5 lbs.
	Chinese blue,	.1 lb.
	Graphite,	1.2 lb.
	Raw linseed oil,	11 lbs.
thin with	Standard oil,	24 lbs.

277. Apple Green.

	White lead,	22.5 lbs.
	Zinc oxide,	22.5 lbs.
	Lemon chrome yellow,	5.5 lbs.
	Chrome green,	24.0 lbs.
	Raw linseed oil,	13.5 lbs.
thin with	Standard oil,	22.5 lbs.

278. Sage Green.

	White lead,	20 lbs.
	Zinc oxide,	20 lbs.
	French ocher,	13 lbs.
	Chrome green,	5.8 lbs.
	Lamp-black,	1.8 lbs.
	Venetian red,	.3 lb.
	Raw linseed oil,	12 lbs.
thin with	Standard oil,	28 lbs.

279. Dark Olive Green.

	Golden ocher,	35 lbs.
	French ocher,	6 lbs.
	Drop black,	9.8 lbs.
	Chrome green,	1.2 lbs.
	Lemon chrome yellow,	.2 lb.
	Raw linseed oil,	13.5 lbs.
thin with	Standard oil,	34.5 lbs.

280. Brunswick Green.

	Extra deep Brunswick green,	40 lbs.
	Lemon chrome yellow,	1.6 lbs.
	Linseed oil,	11.5 lbs.
thin with	Standard oil	28.5 lbs.

281. Outside Green.

	Linseed oil,	25 lbs.
	Yellow ocher,	38 lbs.
	Lamp-black,	2 lbs.
	Chrome green,	8.2 lbs.
thin with	Standard oil,	56 lbs.
	Kauri varnish,	8 lbs.
	Japan drier,	8 lbs.

282. Indian Red.

	Venetian red,	66.2 lbs.
	Princess metallic,	12.3 lbs.
	Raw linseed oil,	24 lbs.
thin with	Standard oil,	54.3 lbs.

By changing the proportion of pigment, any range of shade and tint may be obtained. The above are merely given, therefore, to illustrate the method of procedure.

283. Putty. This substance is very important, for setting glass, filling nail holes, etc. It is made on a commercial scale by mixing the constituents together under a heavy stone roller running in a steel foundation. The process of manufacture may, however, be demonstrated in the laboratory by means of a kneading machine and is carried out as follows:

Three lbs. of raw linseed oil is placed in the machine. A mixture of 11 lbs. of whiting and 5 lbs. of lead carbonate is slowly added to the oil, the arms being kept in constant motion. Care must be taken to have the oil and mixture smooth before introducing new portions. The finished product should not stick to the hands, but, if such is the case, a small amount of the mixture in excess of the above may be added. The putty is then allowed to stand several days to sweat, when it is again kneaded with the addition, if necessary, of a small amount of whiting.

284. Oil Stains and Wood Fillers. The following list of formulas will serve to illustrate the large class of substances put on the market as wood fillers and oil stains. They consist of a base to which various pigments may be added according to the effect desired. The mixing and grinding are conducted as given under ready-mixed paints.

285. Stain and Filler Base. Twelve lbs. of starch is placed in the mixer and worked into a thin paste with 3 lbs. of water. When thoroughly triturated, 27 lbs. boiled linseed oil, 13 lbs. turpentine, and 38 lbs. of japan are added, the whole being run until perfectly smooth. This should be made in large amounts, ready for use, and labeled "Stain Base." It is then used as a body for the various stains.

286. Light Oak or Ash Stain. Place in the mixing machine 7 lbs. of boiled oil and $3\frac{1}{2}$ lbs. turpentine. To this slowly add a mixture of $2\frac{1}{2}$ lbs. raw sienna, 12 oz. burnt umber, and 4 lbs. silica. When thoroughly incorporated, transfer to mill, grinding until perfectly smooth when rubbed out on glass. Return to mixer and thin with 72 lbs. of stain base. Work well and can while still in suspension.

Prepare the following oil stains according to the general directions given for light oak or ash:

287. Dark Oak Stain.

Silica	3.2 lbs.
Burnt Italian sienna,	3.2 lbs.
Burnt umber,	1.6 lbs.
Boiled linseed oil,	7.2 lbs.
Turpentine,	3.6 lbs.
Stain base,	72 lbs.

288. Chestnut Stain.

Silica,	6.4 lbs.
Burnt Italian sienna,	1.6 lbs.
Boiled Turkey umber,	.8 lb.
Boiled linseed oil,	7.2 lbs.
Turpentine,	3.6 lbs.
Stain base,	72 lbs.

289. Yellow Pine or Maple Stain.

Raw Italian sienna,	1.6 lbs.
Chrome yellow,	1.2 lbs.
Silica,	5.8 lbs.

Boiled linseed oil,	7.2 lbs.
Turpentine,	3.6 lbs.
Stain base,	72 lbs.

290. Walnut Stain.

Burnt Turkey umber,	9.2 lbs.
Burnt Italian sienna,	1.2 lbs.
Boiled linseed oil,	7.2 lbs.
Turpentine,	3.6 lbs.
Stain base,	90 lbs.

291. Mahogany Stain.

Burnt Italina sienna,	10 lbs.
Burnt Turkey umber,	1.6 lbs.
Boiled linseed oil,	7.2 lbs.
Stain base,	90 lbs.

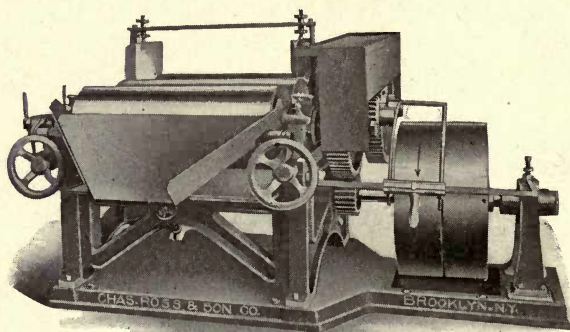


FIG. 31.

Oil stains are often made from coal-tar colors soluble in oil.

Water stains are prepared by dissolving various coal-tar colors in water.

Spirit-soluble stains are made by dissolving the color base in alcohol or a good spirit varnish.

Oil varnish stains may be made by substituting a good grade of oil varnish for the linseed oil in the above formula; or they may be made by dissolving oil-soluble, coal-tar colors in varnish.

292. Enamels. These protective coatings are made by grinding the pigment in varnish. The process, however, cannot be carried out in a buhr stone mill, as is the case with paint; it must be done in a roller mill. A mill of this type is shown in Fig. 31.

CHAPTER VII

SOAP AND ALLIED PRODUCTS

293. Soap. In the manufacture of soap there are three general processes, namely, cold-made soaps, half-boiled soaps, and boiled soaps. The three classes will be taken up in order.

The installation of a miniature soap works requires provision for a lye tank, kettle, crutcher, frames, slabber, cutting machine, chipper, mill, plodder, press, and dies, all of which may be secured from manufacturers of soap machinery. Some of the following formulas can also be worked out on a small scale by using a good-sized iron kettle, a small garden rake with a wooden box to serve as a frame, and a piece of piano wire or thin knife to cut the cake.

The proportions given hereafter are for a crutcher having a capacity of 100 lbs., but they may be varied to suit the requirements.

294. Cold-made Soap. The term "cold-made" soap is applied to those soaps in which the fat and oil are heated only sufficiently to melt them (not over 120° F.) and the cold lye then introduced. This process gives a hard soap, but is quite apt to contain either unsaponified fats or free alkali.

In making soap by the cold process, care must be taken to have all the materials perfectly fresh and clean. The

lyes should never be used the same day they are made. Use the lye cold.

Process. Weigh out 34 lbs. of tallow and place in the crutcher, Fig. 32, turn on the dry steam, and heat until melted. When all of the tallow is in a liquid state, introduce 17 lbs. of cocoanut oil, 17 lbs. of cotton-seed oil, and agitate. Secure a temperature of between 115° - 120° F., and then add 35 lbs. of 36° B \acute{e} . caustic soda lye. The mass is then crutched (stirred) until the soap begins to set, indicated by the formation of flakes

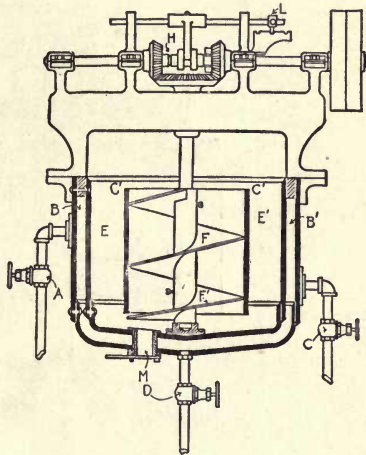


FIG. 32.

when a sample is taken upon the paddle. It is next run into a frame, Fig. 33, and allowed to stand for twenty-four hours to complete saponification and harden. The sides and ends of the frame are then removed, and the cakes passed through the slabber and cutting machine, Figs. 34 and 35. The bars of soap thus formed are placed on racks in the drying room and allowed to stand for several days until thoroughly dry. The

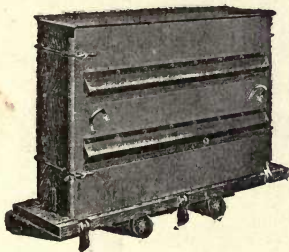


FIG. 33.

bars are now run through the chipper, Fig. 36, where they are cut into flakes about one-sixteenth of an inch thick.

These chips are spread on wire racks and returned to the drying room. When the chips are brittle and snap,

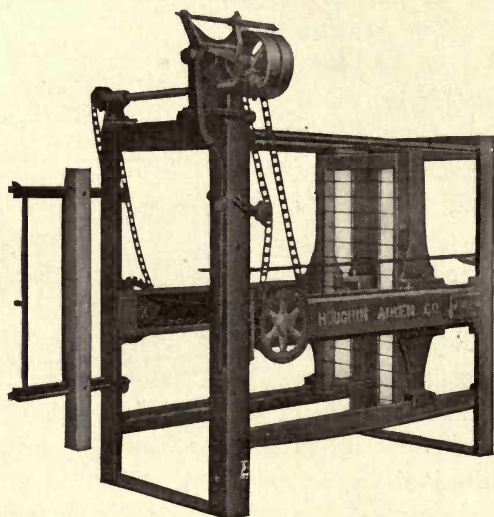


FIG. 34.

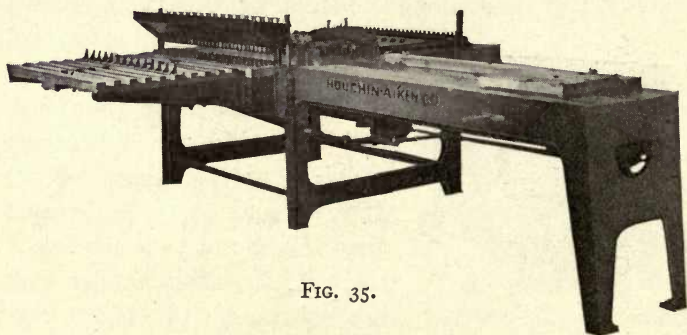


FIG. 35.

they are ready for the milling process, conducted as follows:

Milling. The chips are run through the granite roller mill, Fig. 37, and collected in a box sufficiently large to

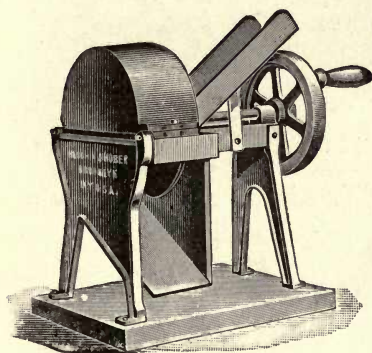


FIG. 36.

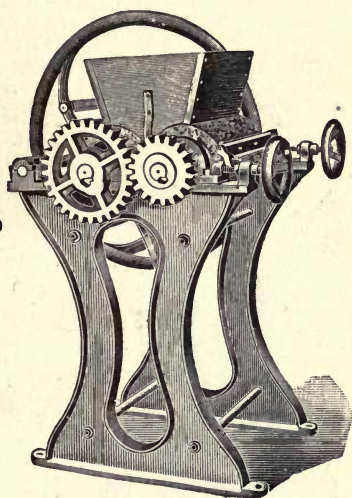


FIG. 37.

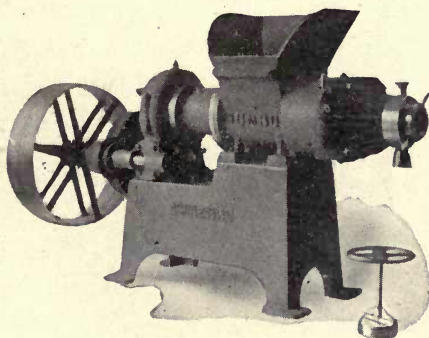


FIG. 38.

hold the entire batch. Eight ozs. of a good soap perfume (Oil of Rose Geranium) is then sprinkled over the

chips, which are shaken up uniformly, to distribute the oil, and then run again through the mill, the granite rollers being a little closer this time, and finally set tight. Repeat the milling several times, or until a uniform ribbon of about the thickness of paper comes from the knife.

Plodding. From the mill the soap is passed to the plodder, Fig. 38, a machine provided with a screw compressor.

The nose of the plodder is slightly warmed and the milled soap introduced. As soon as the soap begins to discharge, the forming plate is adjusted. Keep the hopper of the plodder filled, as this will, in a measure, keep out air bubbles and prevent the soap from being streaked.

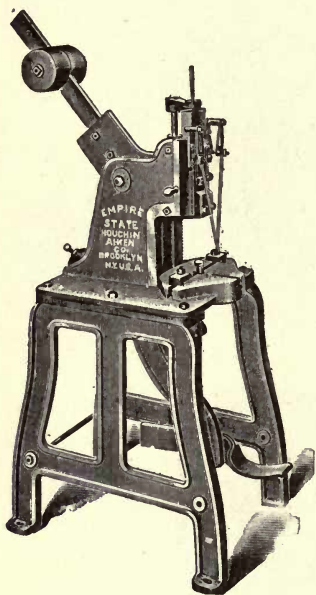


FIG. 39.

The first 15 or 20 feet should be returned to the mill, as the bars have not received the full pressure. As soon as the soap runs smooth and free from streaks, it may be cut into any convenient length and pressed.

Pressing. The dies being adjusted and the press, Fig. 39, well oiled, the faces are then moistened with a strong salt solution and the cake placed in position. Next, a heavy blow is dealt with the foot, followed by a second short blow as the original stroke rebounds. After the first 10 or 12 cakes have been pressed, it will not be necessary to moisten the die so frequently.

295. Green Castile Soap.

Tallow,	25 lbs.
Cocoanut oil,	12 lbs.
Olive oil foots (green),	25 lbs.
Cotton-seed oil,	9 lbs.
Soda lye at 35° Bé.,	36 lbs.

Warm the fats and oils to 110° F., crutch in the lye, and, when the soap has formed, run into frames, allow to harden, and then follow the general directions.

296. Laundry Soap.

Tallow,	30 lbs.
Cocoanut oil,	30 lbs.
Caustic soda lye, 36° Bé.,	30 lbs.
Sodium carbonate solution, 35° Bé.,	8 lbs.
Oil of mirbane,	8 ozs.

This soap is cut into cakes and dried, but is not milled as in the case of toilet soaps.

297. Half-boiled Soaps. By this process very fine soap can be made, almost equal to the best boiled soap. But with them, the same as with cold-process, great care must be taken to have perfectly pure stock.

298. Palm Oil Soap. Place in the crutcher

Palm oil,	18 lbs.
Cocoanut oil,	36 lbs.
Tallow,	9 lbs.

and heat to a temperature of 150° F.

Add 36 lbs. of 35° Bé. soda lye and crutch slowly for about three minutes; now cover up to keep warm, and allow to stand for 1 $\frac{3}{4}$ hours. At the end of this time the

charge will have warmed up to 170° – 180° F. The crutcher is then started very slowly. At first, the soap will be thick and hard to crutch, but will soon thin down and become hotter. If, after crutching for ten minutes, the soap for some reason does not thin down and get hot, turn on a little steam, being careful not to raise the temperature over 180° F. After the soap has been well crutched and is free from lumps, it should be tested by rubbing some in the palm of the hand; if saponification is complete, it will form flakes, whereas if insufficient alkali is present, it will remain like a fat. Also test for strength on the tip of the tongue. The soap should taste a little sharp; if too mild, add a small amount of lye until the latter can just barely be tasted. If, on the other hand, it at first burns the tongue, add a little more cocoanut oil.

After the addition of alkali or fat, crutch well and, when it begins to thicken (shown by the soap dropping free from the paddle in flakes), run into the frame immediately and allow to cool.

For laundry soap, the perfume is crutched in just before framing, whereas, for toilet soap, it is added only at the time of milling. Eight ozs. of citronella oil or other essential oil will perfume 100 lbs. of soap.

299. *Green Castile Soap.*

Tallow,	28 lbs.
Olive oil foots (green),	28 lbs.
Cocoanut oil,	14 lbs.

Mix the foregoing and warm to 150° F., then add a mixture of

Caustic soda lye,	33° Bé.,	36 lbs.
Caustic potash lye,	35° Bé.,	4 lbs.

300. Tar Soap.

Cocanut oil,	25 lbs.
Olive oil foots,	25 lbs.
Caustic soda-lye at 35° Bé.,	30 lbs.
Pine tar,	5 lbs.
Glycerine,	5 lbs.

Warm the oils, tar, and glycerine together. When at 150° F., crutch in the lye and continue crutching until all are well combined; cover up and allow to stand one and one-half to two hours. Then follow general directions.

301. Laundry Soap

Tallow,	31 lbs.
Cocanut oil,	6 lbs.
Rosin W G,	19 lbs.
Caustic soda lye at 33° Bé.,	32 lbs.

Melt the tallow and cocanut oil, then introduce 19 lbs. of rosin; when melted well together, allow to cool down to 130° F., then add the lye, letting the soap stand for one and one-half to two hours. When the soap is finished, and just before framing, add 8 lbs. of sodium carbonate solution, 35° Bé., and 8 ozs. of citronella. Crutch well and frame. When the soap has set sufficiently long to harden properly, it is cut into cakes and, as soon as dry, is ready for use.

302. Dog Soap

Tallow,	50 lbs.
Cocanut oil,	5 lbs.
Caustic soda lye at 36° Bé.,	27½ lbs.
Strong tobacco solution,	5 lbs.
Powdered sulphur,	5 lbs.
Carbolic acid,	5 ozs.

The tobacco solution is made by boiling tobacco stems in water. Mix the sulphur and the tobacco solution together. When the soap is well formed, crutch in the warm sulphur and tobacco; then add the carbolic acid and frame.

303. Boiled Soaps. Owing to the recovery of glycerine, this process is employed very largely; and in fact the bulk of soap is manufactured in this manner. To illustrate the process, we will take, as an example, the manufacture of palm oil soap.

304. Palm Oil Soap.

Tallow, 100 lbs.
Rosin, 100 lbs.
Palm oil, 10 lbs.

Put the tallow into the kettle (Fig. 40) and begin boiling with open steam, adding caustic soda lye at 10° Bé. (about 90 lbs.); continue boiling until the alkali strength is all absorbed. Now, boiling constantly

add stronger lye (about 100 lbs.), at 15° Bé., until the strength is all absorbed. Continue the boiling, but add stronger lye at about 20° Bé. until the soap is strong and ready to grain. Boil well, adding enough dry salt to cause the lye to separate and run free from the soap. Close off the steam and allow the lye to settle.

Rosining. Run off the spent lye and add about 160

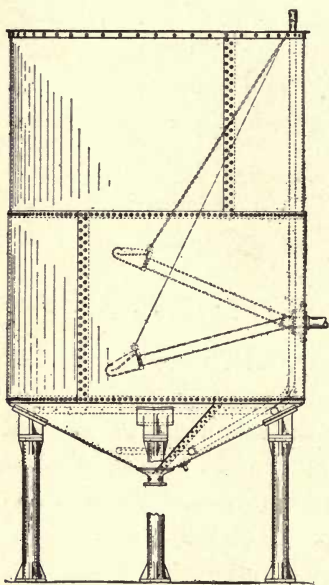


FIG. 40.

lbs. of lye at 15° Bé., boiling well with the open steam until the soap becomes firm and of good grain. While the soap is boiling spread the broken rosin over the surface, and, as the alkali strength is absorbed, add lye at 15° Bé. When nearly all of the rosin has been put in, add salt to keep the soap open, and float the rosin on salt strength instead of the alkali strength. After all of the rosin has been introduced, boil well with closed steam for three or four hours until the curd is round and dry; allow the soap to settle overnight.

Strengthening. Run away the spent lye for glycerine recovery. Begin boiling with the open steam, running in about 80 lbs. of lye at 10° Bé.; boil well until the soap becomes thin. Then, while boiling, add lye at 20° Bé. until the soap opens and is sharp with alkali. Continue the boiling for about five hours with the closed steam only, after which the soap should have a hard, strong grain and be rough on the surface. Shut off the steam and allow to settle overnight.

305. Finishing. Run off and save the lye that has settled out, as it contains alkali strength which can be used again. Turn on the open steam and add the 10 lbs. of palm oil, running in a little water to thin the soap. If the soap is weak, add lye at 5° Bé. When the soap is finished, it should be smooth and shining, should boil with a rolling motion; and a sample taken on the trowel should slide off in flakes.

306. Framing. Allow the soap to settle a few days until it has cooled to about 145° F. Run into the crutcher enough for one frame and add 6 to 8 per cent of soda ash solution at 36° Bé. Crutch well for two minutes, add the perfume, and crutch until smooth. Dump into

the frame and stand several days to harden. On setting, it is slabbed and cut, but not milled as in the case of toilet soap.

307. Rosin Soap. Inasmuch as plain tallow soap does not lather, it is always necessary to add something to make it do so. In laundry soap, rosin is used. For a cheaper and harder product, silicate of soda is sometimes employed. The following formula will serve to illustrate a soap of this character:

Tallow,	150 lbs.
Rosin,	75 lbs.
Sodium silicate,	15 lbs.

The method of manufacture is the same in this case as for the previous example. After finishing, however, the silicate of soda is added as follows:

Put the silicate of soda into a small kettle and add water, boiling until it registers 5° Bé. (hot); then add soda ash until it registers 7° Bé., then salt until it makes 8° Bé., while boiling hot. Now run about 70 lbs. of the soap into the crutcher and add 30 lbs. of the hot silicate of soda solution. Crutch well, add the perfume, and continue crutching until the soap starts to form. Dump into the frame and treat as directed above.

308. Soft Soap. "Transparent" soft soap is in good demand under the name of "glycerine soft soap" "crown soap," etc. Its chief constituent is linseed oil, and the lye most suitable is that from caustic potash. The lye is prepared several days before using in order that it may become clear. A very good soap can be made by melting 4 lbs. of rosin in a crutcher or iron kettle and

adding 21 lbs. of linseed oil and 21 lbs. of cotton-seed oil. The temperature is raised to 160° F., and a mixture of 42 lbs. of caustic potash lye, 22° Bé., and 12 lbs. of caustic soda lye, 25° Bé., introduced. The mass is then crutched for five minutes, allowed to stand for two hours, and crutched again until the soap is smooth. It is finally run into the container, and, when cool, is ready for use.

309. Liquid Soap. Heat 40 lbs. of cocoanut oil to 220° F. Introduce 50 lbs. of 22° Bé. caustic soda solution, crutch well for five minutes, or until smooth, and add a mixture of 5 lbs. of alcohol and 5 lbs. of glycerine. Crutch again for five minutes and let stand two hours. Again crutch until smooth. This soap, when cool, will set to a stiff paste. It is used as a base for making liquid soaps, by dissolving 6 ozs. in 1 qt. of boiling water and adding, when cool, 2 ozs. of alcohol.

310. Sand Soap Paste. Prepare a base soap as under *Liquid Soap*, but without the addition of alcohol and glycerine. After standing for two hours, it is mixed with 50 lbs. of sea-sand, crutched well, and run into frames.

311. Soap Powder. There are many kinds of soap powders on the market. They are all, however, prepared from soap and soda ash; sometimes borax is also used. The following will serve as an illustration:

Eight lbs. of tallow and 8 lbs. of cocoanut oil are placed in the crutcher and heated to 150°-160° F.; 8 lbs. of caustic lye, 33° Bé., is now crutched in and the soap allowed to stand for two hours. At the end of this time, crutch until thin and add hot water until quite thin. Crutch in $\frac{1}{2}$ pound of sulphate of soda, dissolved in hot

water, and then add 25 lbs. of soda ash, keeping the soap thin by the addition of hot water. When thoroughly mixed, run into frames and let stand overnight; then cut into bars and open-pile to dry. When thoroughly dry chip and grind; it is then ready for use.

CHAPTER VIII

LEATHER MANUFACTURE

THE object of tanning is to render the skins of animals imputrescible and pliable. As only a small amount of leather is required with the hair on, it is necessary, before entering into the actual tanning process, to remove this hair by preliminary treatments carried out to best advantage in accordance with the outline given below:

312. Soaking. Skins or hides come to the tanner in a flint dry salted, green-salted or pickled condition, and contain, besides the salt, a large amount of blood and dirt. The first step, therefore, is to place them in a pit containing water, where they are allowed to remain until soft. They are then rinsed off in fresh water to remove as much of the dirt and blood as possible. When washing is complete the skins are placed over a round, inclined table called a "beam," where the excess of flesh, earlaps, tail, etc. are trimmed off by means of a sharp knife called a "flesher." Machines are also made for this purpose.

Skins or hides which come to the tanner in the dry-salted or dry (flint) condition require a longer treatment than green-salted stock. They are usually soaked for about two days, then run in a dry mill for an hour or so, and returned to the soak. The stock should be in a soft condition before proceeding to subsequent operations.

Sometimes, in order to hasten the process, a small amount of caustic soda or formic acid is added to the soak water, usually about 0.1 per cent of the weight of the water.

313. Depilating. The next step in the process consists in removing the hair and epidermis and is usually accomplished by means of milk of lime, although other substances are sometimes employed.

(a) *Milk of Lime Process.* Sufficient milk of lime is prepared to cover the skins. To this, sodium sulphide, 0.5 per cent the weight of the lime, has been added. The skins, are introduced into the mixture and turned twice each day in such a manner as to bring the ones on top into the bottom of the pit. This treatment requires from four to eight days, depending on the nature of the skins, the proper time being indicated by the ease with which the hair and epidermis come off when rubbed over with the finger.

The chemical action taking place in this treatment is, in part, the combination of the calcium hydroxide with the fat surrounding the hair follicle and lying between the fibers, thus forming a lime soap, which loosens the hair and epidermis, swelling up and separating the fiber bundles. The action is more largely due, however, to the solvent nature of the lime and enzymes present, which tend to hydrolyze and dissolve the albuminous matter. The hair itself is only very slightly altered. Old limes will unhair much more readily than new ones, which is often advantageous except that, for sole-leathers and others requiring firmness, new limes are preferred.

When properly limed, the skins are removed from the pit and allowed to drain. They are again placed over

the beam, where their plump condition facilitates the removal of the remaining flesh with the fleshing knife, and also the scraping off of the loosened hair with a blunt knife. In the case of hides (heavy leather) much of the lime is also worked out by this means. After unhairing and fleshing, the skins or hides are placed in a pin-mill where they are washed with running water for half an hour, or until the fine hair has been removed. The stock is then in proper condition for subsequent treatment.

(b) *Sulphide Process for Depilating.* The well-soaked and washed stock is put into a paddle containing a 4° Tw. solution of sodium sulphide at 70° F. It is run for about two hours. The wheel is then stopped and, after that, turned for five minutes each hour. The stock is left in the liquor overnight and turned from time to time during the morning. At about noon the plug is drawn and the spent liquor let out. The paddle is filled with water and run for half an hour. One per cent of sodium bicarbonate is now added and run for one hour. Fresh water is turned on and the stock washed for three hours. By this time it should be partly free from sulphide and in a fallen condition. It is then removed from the paddle and may be bated and pickled as desired.

(c) *Depilating with Sodium Sulphide and Calcium Chloride.* Into the paddle put a weighed amount of sodium sulphide, sufficient to make a 20° Bk. solution. To this solution add one-fourth as much calcium chloride as sodium sulphide. Raise the temperature of the solution to about 80° F., add the stock, and run at intervals during the day. The skins are left in the

paddle overnight, and, at the end of twenty-four hours, washed for half an hour, neutralized with 2 per cent of sodium bisulphite, and washed again for about an hour. They are then ready for the bate. The advantage of this process is that the stock resembles limed material and is very free from false grain. Also, the measurement is better than that of straight sulphide stock.

(d) *Painted Sheepskin Pelts. Soaking.* The skins, if not in a perfectly soft condition, are placed in water, until in the necessary flaccid state. They are then horsed up to drain.

Painting. Dissolve 5 lbs. of sodium sulphide in as little hot water as possible, and add to this solution 2 lbs. of lump lime. As the limes lakes, the mass will thicken to the consistency of a heavy cream. If not quite thick enough, a small amount of lime may be added. Hydrated lime may also be used with the sodium sulphide, and enough added to produce a creamy consistency.

Treatment. The drained skin is placed flesh side up on the floor or table, where a liberal coating of the paste is applied. It is then placed on the floor, and a second skin painted in the same manner, is placed flesh to flesh on the previous one. The next skin is placed wool to wool, and so on until the stock is piled. The following day the skins are taken up in order. The white wool is pulled off by hand and placed in one container, and the colored wool in another. During the pulling of the wool, it is often customary to sort four different grades. The stock of wool thus obtained is washed first in water containing 5 per cent of sodium bicarbonate or sodium bisulphite, then in running water, and is whizzed, and dried.

The unhaired pelts are placed in a paddle with 5 per cent of lime and run for two or three days. They are then bated, pickled, and tanned as desired.

314. Puering or Bating. This process frees the skin from lime, produces a soft, flaccid, open condition, and smooths down the grain. There are numerous methods in vogue, most of which depend upon fermentation for their beneficial action. Some of the substances in use are hen, pigeon, and dog manure, sour milk in the form of dermiformer, fermenting bran, puerine, oropon, and many others. Many non-fermenting bates are also on the market, the most common of which is lactic acid.

The fermenting process has the advantage over the non-fermenting of making the skins more open, thus permitting a more uniform combination of the hide substance with the tanning material.

(a) *Oakes Bate.* An excellent process and one which may be carried out with perfect safety is covered with letters patent by Francis J. Oakes, of New York City. It is as follows: Into a paddle-box put enough water to work the skins easily. Add 5 per cent of syrup glucose and $\frac{1}{2}$ per cent of flowers of sulphur, computed on the weight of the skins. Raise the temperature of the bath to 105° F., add 1 lb. of yeast for each 1000 lbs. of skin (or, in small lots, 1 yeast cake for 6 small skins), and let stand twenty-four hours. The bath by this time has started active fermentation, and the skins are introduced, together with one-half the original amount of glucose and sulphur. The skins are run for about five minutes, then allowed to stand for one hour. Next they are run again for five minutes and the treatment continued until a test with phenolphthalein on a piece of the skin,

cut from the heaviest part, shows that all of the lime has been neutralized. At this point, the fibers are found to be well open, and the skin soft and pliable, with a smooth and silky grain. The time required for this operation should be from six to eight hours in the case of heavy skins, with a correspondingly shorter period for lighter ones.

(b) *Dr. Rohm's Bate "Oropon C" for Regular Packs. Treatment of the Skins before the Bate.* The same as usual. It is advisable to wash them thoroughly before the bate, in order to free them as much as possible from lime. In the case of sulphide, neutralize as usual.

Bate:

Quantity of water, same as usual.

Temperature, as usual.

Time of bating, two to four hours for calf skins;
four to six hours for hides.

Quantity of oropon, always figured on the weight of the wet skins and according to their nature, thus:

8-10 oz. per 100 lbs. for calf and split grains;
10-12 oz. per 100 lbs. for unsplit hides (used for furniture, etc.).

The liquor can be used over again, and in this case add, for the second and following packs, about one-half the quantity, or even less of the oropon used for making fresh liquor. It is advisable to warm up the old liquor overnight the first two days in order to accelerate fermentation. After the liquor has been used for two days, it will be noticed that the fermentation grows stronger.

To keep it in control, run off every day about $\frac{1}{2}$ to 1 foot of the liquor (more in summer than in winter). Before running off, stir the liquor well to get rid of as much dirt as possible. As a rule, make the bate up fresh once every two weeks, but, in doing so, leave about one-fourth of the old liquor in the paddle.

Bating Process. Always heat the water first; then put the oropon in undissolved. After stirring thoroughly, put in the skins. The further treatment is the same as usual. Watch the process closely in order to see how the skins fall, and do not take them out until they are as low as desired. In case they fall too quickly, reduce the quantity of oropon proportionately the next time. If they do not fall enough, add to the bate 0.1 to 0.2 per cent of oropon (dissolved in a bucket of water and added while the skins are paddled); at the same time raise the temperature of the liquor, in case it has cooled down too quickly. If, after about one hour, the skins are still too high, repeat this operation until a satisfactory result is obtained. The next time a correspondingly higher concentration is used. If overnight bating is preferred, use about 20 per cent less oropon than indicated above, and a temperature of from 85°-90° F. before skins are put in.

Wash the skins thoroughly after the bate, and proceed according to paragraph 315.

(c) *Dr. Rohm's Bate "Oropon A.B." for Limed Goat Skins.* Take 30 gals. of water for each 100 lbs. of skins; for regular packs take the usual quantity of water.

Treatment of the Skins before the Bate. The skins are washed in running water as usual.

Bating, First Liquor. The skins are first washed in

an old, used bating liquor. If no old oropon liquor is available, substitute a light solution of 4-6 oz. oropon for each 100 lbs. of skins, or better, wash the skins in the used dog-manure liquor.

Temperature. For soft skins 90°-95° F. (before the skins are in). For hard skins, 95°-105° F. (before the skins are in).

Second Liquor. In this liquor the skins are bated overnight (put in about 4 o'clock, paddled ten to fifteen minutes, and left until the next morning). In the morning they are paddled again for fifteen minutes and if low enough, taken out. In preparing the bating liquor, always heat the water first, then put in the oropon without previously dissolving it, and, finally, put in the skins.

Quantity of Oropon:

For South Americans, Brazils, etc., 8-10 oz. per 100 lbs.

For Russians, 8-10 oz. per 100 lbs.

For Chinese, 12-16 oz. per 100 lbs.

For Mochas and Patnas, 14-16 oz. per 100 lbs.

For Northwesterns, 16-18 oz. per 100 lbs.

Temperature. 95°-105° F. (before skins are in), according to the nature of the skins.

In case the skins are not reduced enough, add more oropon, starting with about 20 per cent of the original quantity. If, after about an hour, the skins are still too high, repeat the operation until the skins are as low as desired. The next time, of course, correspondingly more oropon is used. In bating over-

night, the fermentation will raise the skins to the surface of the water, which action is desirable. It is possible that the first two or three packs will not come up, as the fermentation is too weak at the start. It will strengthen gradually, however, and the fourth and following packs will rise overnight.

(d) *Martin Dennis' Puerine.* Take a clean 50-gal. barrel, remove the head, put in 25 gals. of water and heat to 130° F., then, while stirring vigorously, put in 100 lbs. *puerine*. Cover barrel with an old burlap bag to assist in retaining the heat. Twenty-four hours later, add 10 gals. of water at 140° F., stirring vigorously. Twenty-four hours later, add another 10 gals. of water at 140°, stirring vigorously. After twenty-four hours more, the puerine is ready for use. The barrel will now be full, and each gallon taken therefrom will contain 2 lbs. of puerine.

To Use the Prepared Puerine. Always stir thoroughly before taking any from the barrel.

First Pack. Have desired quantity of water in the paddle, heat to 95°, put in stock and then add, for every 1000 lbs. of stock, 12 lbs. puerine (6 gals. of the prepared puerine).

Second Pack. Use 8 lbs. puerine (4 gals. of the prepared) for every 1000 lbs. of stock.

Third Pack. Use 6 lbs. puerine (3 gals. of the prepared) for every 1000 lbs. of stock.

Fourth and All Following Packs. Use 4 lbs. puerine (2 gals. of the prepared) for every 1000 lbs. of stock.

Once a week allow the puer paddle to settle about four hours, then draw off from the top about one-fifth of the bating liquor. This is easily done by having a plug on the side of the pit at the proper distance from the top.

Every three months clean the pit out thoroughly by drawing another plug from the bottom.

It is impossible to explain in writing how long the stock should be bated. The stock should be left in the bate, until, in the judgment of the operator, it is "low" enough.

The puerine bating liquor improves with age, and it must not be expected that the first few packs will be as well bated as the packs that follow. In this respect it is the same as a manure bate. When the bating liquor assumes a bluish, slate color, it is in prime condition. If, at any time, this puerine bating liquor suddenly becomes yellow, it is an indication that too little puerine is being used.

(e) *Ammonium Chloride Bate.* Into a paddle place ammonium chloride, 3 per cent on the weight of the stock, and heat to 90° F. Enter the pack and run until delimed. Toward the end of the treatment neutralize the alkaline condition with a small amount of hydrochloric acid. Before entering the second packs and following add, 1½ per cent of ammonium chloride, then proceed as above. The bath is a continuous one.

315. Pickling. On removing the skins from the bate it is customary to pickle them if they are to be tanned by the one-bath chrome process. For this purpose a stock solution is prepared by dissolving 40 lbs. of salt in 30 gals. of water, adding 5 lbs. of sulphuric acid and making the whole up to 40 gals. with water. Twelve gals. of this solution is used for each 100 lbs. of stock treated; and the skins are run in the drum until struck through.

316. Two-bath Chrome on Pickled Sheepskins. (a) *Glaze Finish. Sodium Dichromate Bath.* The bated or

pickled stock is weighed and placed in the mill, the door closed, and the following solution, estimated on the weight of the stock, prepared:

Sodium dichromate, 6 per cent
Salt, 4 per cent
Hydrochloric acid, 3 per cent
Water, 5 times the weight of the stock.

The mill being set in motion, the above solution is introduced through the trunnion, and the stock turned for $1\frac{1}{2}$ hours, or until struck through. The skins are removed from the drum and horsed up overnight to allow for draining and fixation of the chrome.

Sodium Thiosulphate Bath. The following morning the skins are removed from the horse one at a time, dipped in a 10 per cent solution of sodium bisulphite, and thrown into the drum. The following solution,

Sodium thiosulphate (hypo), 12 per cent
Salt, 3 per cent
Hydrochloric acid, 6 per cent
Water, 5 times the weight of the stock,

is added through the trunnion while the mill is in motion and run for $1\frac{1}{2}$ hours, or until a robin's-egg blue color results. The skins are again horsed up overnight.

Neutralization. The skins, after standing overnight, are returned to the mill and run for one-half hour with

Sodium bicarbonate, $\frac{1}{2}$ per cent
Water, 5 times the weight of the stock.

The plugs are now drawn and the stock washed for one-half hour with running water. The skins are then set out and shaved.

Coloring. The wet, shaved stock is placed in the drum with sufficient water at 125° F. to float it. One per cent of hematine crystals, dissolved in water at 125° F. and made alkaline with ammonia, is introduced through the trunnion while the drum is in motion and run for fifteen minutes. One and one-half per cent of Chrome Leather Black C is dissolved in water at 140° F. and added to the hematine solution. The stock is now run for fifteen minutes, then one-half hour longer in a fat liquor consisting of

Victoria Fat Liquor BXX, 1 per cent
Cod oil, $\frac{1}{2}$ per cent
Water, 2 times the weight of the stock.

The stock is now washed for five minutes in warm water, removed, set out, lightly oiled with 1 to 3 glycerine and hung up to dry.

Degreasing. Should the raw stock feel excessively greasy, it is best to dip it at this stage in naphtha for fifteen minutes and then hang up to dry again.

Finishing. The dried skins are placed in damp sawdust overnight, or until in a proper sammied condition. They are then staked and tacked. On stripping they are trimmed, given a coat of glaze finish, and glazed on the jack. They are now lightly staked and ironed. A second coat of finish is applied and, when dry, they are glazed again.

(b) *Mat Finish.*

Tanning. The bated or pickled stock, having been

weighed, is placed in the drum and the following solution prepared:

Sodium dichromate,	6 per cent
Sulphuric acid,	2 per cent
Salt,	4 per cent

with the necessary amount of water to give proper working conditions. The skins being on the shelf, the solution is placed in the bottom of the drum, the door closed, and the power turned on. The turning is continued for about $1\frac{1}{2}$ hours, or until the stock has become struck through. It is then horsed up overnight and allowed to drain. The next morning it is returned to the drum and the following solution added:

Sodium bisulphite, 6 per cent,

dissolved in sufficient water to float the stock. It is run in this reducing solution for about one hour or until a robin's-egg blue color results. The stock is again horsed or piled for twenty-four hours, to allow hydration to take place. To neutralize the excess of acid run the stock with $\frac{1}{2}$ per cent of sodium bicarbonate, and finally wash well with running water.

Coloring. The washed stock is set out, shaved, and weighed. One per cent of hematine powder is dissolved in water at 130° F. and enough ammonia added to give an alkaline reaction. The stock is placed in the drum and run in the above solution for fifteen minutes. Through the trunnion is added a solution containing

Brilliant Chrome Leather Black C, 2 per cent

at a temperature of 140° F. and run for fifteen minutes longer. One per cent of Victoria Fat Liquor B X X at 140°

F. is now added, without removing the color, and run for half an hour longer. The stock is finally removed from the drum, set, lightly oiled, and hung up to dry.

Finishing. The dried skins are placed in damp sawdust overnight and, when in the proper sammied condition, are staked and tacked. On stripping they are trimmed and given a light staking and a coat of mat finish. The finish having dried, the skins are given a light coat of finishing oil and then ironed.

317. One-bath Chrome. (a) *Box Calf.* *Stock:* Green-salted, 5-7 lb. calf skins.

Soaking. The stock is shaken free from salt and, after trimming, is placed in fresh water for twenty-four hours.

Milling. The excess of salt and dirt is removed by milling for fifteen minutes with running water.

Fleshing. The excess of flesh is removed on the fleshing machine.

Depilating. Five per cent of hydrated lime on the weight of the fleshed stock is placed in the paddle with the necessary amount of water and 1 per cent of sodium sulphide. The temperature is raised to 70° F., and the stock entered and run at intervals during the day. The following morning a new portion of lime and sulphide is introduced, the same amount being used as on the first day. After two days' treatment, the bath is again strengthened in the same manner, the temperature being maintained at 70° F. At the end of four days, and without further additions, the stock is in proper condition for unhairing.

Unhairing. On removal from the depilating solution, the skins are thrown into water at 85° F. for one-half hour, unhaird over the beam, and washed.

Bating. A bath of oropon is prepared, consisting of 8 oz. of Oropon C for each 100 lbs. of stock treated. The temperature at the start is 100° F. The stock is turned from time to time and allowed to remain in the paddle overnight. On the following morning the skins should be in a soft and open condition and free from lime.

Pickling. A stock pickle is prepared by dissolving 40 lbs. of salt in 30 gals. of water, adding 5 lbs. of sulphuric acid, and making the whole up to 40 gals. with water. Twelve gals. of this solution is taken for each 100 lbs. of stock treated, the latter being run in the drum until struck through.

Tanning. The drained and pickled stock is placed in the drum with 8 gals. water and 6 lbs. of salt for each 100 lbs. treated, and run for fifteen minutes. Six lbs. of "Tanolin" for each 100 lbs. of stock is dissolved in boiling water, using 1 gal. of water for 3 lbs. of tanolin. When the solution is cool one-third is added to the contents of the drum and the stock run for one-half hour; then another one-third, and another one-half hour run; finally, the last one-third is introduced, and the drum run for one hour. Eight oz. of bicarbonate of soda is dissolved in a little water, added to the drum, and run for two hours; then 6 oz. per 100 lbs. more bicarbonate of soda in solution is added and the stock run until tanned. The test for complete tannage is determined by boiling a sample of the heavy portion, and, if tanned, it will not curl. When tanned, the stock is horsed up for forty-eight hours, allowing the chromium hydroxide to set.

Neutralizing. After draining for forty-eight hours, the skins are thrown into the drum and a solution of

borax added,—1 lb. of borax in 10 gals. of water for each 100 lbs. The stock is milled for one-half hour and washed in running water for one-half hour. It is then removed from the drum, set out, and shaved.

Coloring. The shaved stock, after being weighed, is returned to the drum and sufficient water at 125° F. added to float the skins. One lb. of hematine is dissolved in 10 gals. of water at 125° F. per 100 lbs. of stock, and, after being made alkaline with ammonia, is added through the trunnion while the drum is in motion, and run for fifteen minutes. One and one-half lbs. of Chrome Leather Black C is dissolved in 10 gals. of water at 140° F. per 100 lbs. of stock, added to the contents of the drum, and run for fifteen minutes longer.

Fat Liquoring. Two and one-half lbs. of Victoria Fat Liquor BXX, dissolved in 10 gals. of water per 100 lbs. of stock, are added to the contents of the drum without removal of the color solution. The stock is then milled for one-half hour. On removal from the drum, the stock is set out, oiled off lightly with Setine No. 2, and hung up to dry.

Staking. The dry stock is placed in damp sawdust overnight, when it is brought to the proper sammied condition. The skins are then staked and tacked out. When dry, they are stripped from the boards, trimmed, and restaked.

Finishing. To the staked leather a coat of glaze finish is applied and, when dry, the stock is glazed. A coat of finishing oil No. 6 is next applied, and the stock ironed. A second coat of glaze finish is then given and, when thoroughly dry, the stock is again glazed. After the second glazing, the skins are boarded in the manner usual for where box grain.

One-bath Chrome. (b) *Mat Calf. Stock:* Green-salted calf skins, 5-7 lbs.

Soaking. The stock is shaken free from salt and, after trimming, is placed in fresh water for twenty-four hours.

Milling. The excess of salt and dirt is removed by washing in running water for fifteen minutes.

Fleshing. The excess of flesh is removed on the fleshing machine.

Depilating. Five per cent of hydrated lime on the weight of the fleshed stock is placed in the paddle with water at a temperature of 70° F., and the stock entered. The paddle is turned from time to time, and the following morning another portion of 5 per cent of hydrated lime is put in. A fresh portion of lime is added on each of the next two days, the temperature being maintained at about 70° F. At the end of five days, the skins are drawn and should be in proper condition for unhairing.

Unhairing. On removal from the depilating solution, the stock is thrown into water at 85° F. for one-half hour and unhairied over the beam.

Bating. A bath of Oropon C is prepared, consisting of 8 oz. of oropon for each 100 lbs. of stock treated. The temperature is 100° F. at the start. The stock is turned from time to time and in the paddle overnight. On the following morning the skins should be soft, open, and free from lime.

Pickling. The washed skins are put in the drum; and in a barrel is placed $\frac{1}{2}$ gal. of water for each pound of stock. To this water is added 12 per cent of salt and $1\frac{1}{2}$ per cent of sulphuric acid. The solution thus prepared is added through the trunnion while the drum is in motion and run for $1\frac{1}{2}$ hours. The stock is then allowed to remain

in the drum overnight and run for another half hour the following morning. It is next taken from the mill, horsed up, and allowed to drain.

Tanning. The drained, pickled stock is placed in the drum with 8 gals. of water and 6 per cent of salt and run for fifteen minutes. Six per cent of tanolin is dissolved in boiling water, using 1 gal. of water for 3 lbs. of tanolin. Of the cooled solution one-third is added to the contents of the drum, and the stock run for one-half hour; another one-third is entered and run for one-half hour; finally, the last one-third is introduced and the drum run for one hour. Eight oz. of sodium bicarbonate, dissolved in a little water, is added to the contents of the drum and run for two hours, then 6 oz. more of sodium bicarbonate for each 100 lbs. of stock, and run until tanned.

Neutralizing. After the skins have been drained on the horse for forty-eight hours, they are thrown into the drum with a solution of 1 per cent of borax milled for one-half hour, washed for one-half hour, set out, and shaved.

Coloring. The shaved stock, after being weighed, is returned to the mill with sufficient water at 125° F. to float the stock. One per cent of hematine, dissolved in water at 125° F., made alkaline with ammonia, is added through the trunnion while the drum is in motion and run for fifteen minutes. One and one-half per cent of Chrome Leather Black C is dissolved in water at 145° F., added to the contents of the drum, and run for fifteen minutes longer.

Fat Liquoring. Four per cent of Victoria Fat Liquor B X X is dissolved in water at 140° F., put into the drum,

without removing the color, and run for one-half hour. On removal from the drum the stock is set out, oiled off with Setine No. 2, and hung up to dry.

Staking. The dry stock is placed in damp sawdust over night to sammie. It is then staked and tacked out. When dry, the skins are stripped, trimmed, and restaked.

Finishing. The staked leather is given a coat of mat finish and allowed to dry. A coat of finishing oil No. 6 is then applied and the stock ironed. If bad on the grain, a light snuffing will improve the appearance of the leather.

318. Bright Calf and Side Leather. *Depilating.* The soaked and fleshed stock may be very readily depilated by running in the paddle, or, if desired, in a still-pit with 5 per cent of hydrated lime and 1 per cent of sodium sulphide, estimated on the wet, fleshed weight.

By calling the pits *A*, *B*, and *C*, and numbering the pack, the method of procedure may be more readily understood. The temperature should be maintained at about 70° F., and the process is as follows:

- 1st day, Pack 1 Entered in pit *A* containing 5 per cent lime and 1 per cent sodium sulphide.
- 2d day, Pack 1 Transferred to pit *B* containing 5 per cent lime and 1 per cent sodium sulphide.
- 2 Entered in pit *A*, once used.
- 3d day Pack 1 Transferred to *C* containing 5 per cent lime and 1 per cent sulphide.
- 2 Transferred to *B*, solution once used.
- 3 Entered in pit *A*, solution twice used.

4th day, Pack 1 Removed.

3 Drawn from *A*, new liquor consisting of 5 per cent lime and 1 per cent sodium sulphide made up for that pit.

2 Transferred to *A*.

3 Transferred to *C*, once used.

4 Entered in pit *B*, twice used.

5th day, Pack 2 Removed.

4 Drawn from *B* and new liquor prepared as above.

3 Transferred to *B*.

4 Transferred to *A*, once used.

5 Entered in *C*, twice used.

5th day, Pack 3 Removed.

5 Drawn from *C*, and new liquor prepared.

4 Transferred to *C*.

5 Transferred to *B*, once used.

6 Transferred to *A*, twice used.

When once started and carried out, the above-mentioned movement entails only very little handling of the stock. This method gives a good grain and full feel.

Unhairing. On removal from the depilating solution, the stock should be thrown into water at about 85° F. for one-half hour and then unhaired on the machine or over the beam.

Bating. The bating may be carried out in the usual way. Care must be taken to see that the stock is not bated too low, otherwise a loose grain may result.

Pickling. A stock pickle should be prepared as follows:

Salt,	40 lbs.
Sulphuric acid,	5 lbs.
Water sufficient to make	40 gals.

Place the stock in the drum and add 12 gals. of pickle for each 100 lbs. of stock. Run for about $1\frac{1}{2}$ hours or until struck through.

Tanning. The drained, pickled stock may now be tanned by any method desired.

Coloring. The shaved stock is weighed and placed in the drum with sufficient water at 125° F. to float it. One lb. of logwood extract for each 100 lbs. of shaved stock is dissolved in 10 gals. of water at 125° F. and made alkaline with ammonia. The logwood solution is then poured through the trunnion and the stock run for fifteen minutes. Chrome Leather Black C., $1\frac{1}{2}$ lbs. for each 100 lbs. of stock, is dissolved in 10 gals. of water at 140° F., added through the trunnion, and run for fifteen minutes longer.

Fat Liquoring. A fat liquor is prepared by dissolving for each 100 lbs. of stock

Victoria Fat Liquor B X X,	$1\frac{1}{2}$ lbs.
Water at 140° F.,	10 gals.

To this is added

Cod oil,	1 lb.
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The fat liquor emulsion is now introduced through the trunnion without removing the color solution and run for one-half hour. The stock may now be removed without washing, or, if desired, a short washing in warm water may be given.

To get a blue black, some tanners prefer to shave out of color. If this is done, only one-half of the color

required for shaved stock would be necessary on the wet tanned weight. Nigrosine is then mixed with the fat liquor to cover up the unevenness produced by shaving.

Finishing. The colored stock, on removal from the mill, is put out and hung up to dry. It is sometimes oiled off with mineral oil. After being is sammied in damp sawdust, it is staked, tacked, stripped, restaked, and given a coat of glaze finish. When dry, it is jacked, given a second coat of finish, and jacked again. The stock may also be smooth-plated if desired.

319. Glazed Goat Skins. *Soaking.* The skins are placed in water at 60° F. and allowed to soak for about three days, or until perfectly soft. They are then washed in the mill for fifteen minutes with running water, and dry milled for five minutes.

Depilating. The washed stock is treated with a mixture of 10 per cent of lime slaked in the presence of 1 per cent of arsenic sulphide. The treatment is carried out in a series of pits and usually takes from twelve to fifteen days, the lime being strengthened as required. The skins are then removed and unhaired over the beam or on the machine, and subsequently fleshed.

Bating. The fleshed stock is weighed, placed in the paddle with $\frac{1}{2}$ per cent of Oropon AA at a temperature of 100° F., run for the remainder of the day, and left in the paddle overnight. The next morning the stock is run for an hour longer, or until perfectly soft and open.

Pickling. If desired, the bated stock is placed in the mill and pickled for $1\frac{1}{2}$ hours with 12 per cent of salt and $1\frac{1}{2}$ per cent of sulphuric acid, using a solution containing 1 lb. of salt to 1 gal. of water.

Tanning. The drained, bated, or pickled skins are placed in the mill and treated with a solution made up as follows, running for $1\frac{1}{2}$ hours, or until struck through:

Sodium dichromate,	6 per cent
Sulphuric acid,	2 per cent
Salt,	4 per cent
Water,	3 times the weight of stock

On removal from the drum, the skins are horsed up overnight and allowed to drain. They are then dipped one at a time in a 10 per cent solution of sodium bisulphite and thrown into the drum. A solution of

Sodium bisulphite,	6 per cent
Water,	3 times the weight of stock

is prepared and added through the trunnion while the mill is in motion. The milling is continued for $1\frac{1}{2}$ hours, or until the stock assumes a robin's-egg blue color throughout. The skins are then removed from the drum and horsed for several days. They are then pressed or set out and shaved.

Coloring. The shaved and weighed skins are placed in the drum with sufficient water to float them, 1 per cent of borax added, and run for one-half hour. They are then washed for one-half hour in running water. One per cent of hematine is dissolved in water at 125° F., made alkaline with ammonia, enough water being used to thoroughly float the stock. The skins are run in the solution for one-half hour; $1\frac{1}{2}$ per cent of Brill. Chrome Leather Black dissolved in 20 gals. of water at 140° F. is now introduced and run for fifteen minutes longer.

Fat Liquoring. The stock having been colored, the

following liquor is prepared and added through the trunion without removing the color:

Chip soap, $1\frac{1}{2}$ per cent
 Neatsfoot Oil, 20° C. T., 2 per cent
 Water, 2 times the weight of stock

and run for one-half hour. The skins are washed for ten minutes in warm water, set out, oiled off with 1 to 3 glycerine solution, and hung up to dry.

Finishing. The dry skins are allowed to remain in the crust for several days and are then sammied in damp sawdust until soft. They are next staked, given a coat of glaze finish, and staked again. They are then glazed, perched, given a second coat of glaze, ironed, and glazed again.

320. Combination Pig-skin Tannage. *Soaking.* The green-salted stock is soaked in water for twenty-four hours and then washed in the mill for fifteen minutes with running water.

Depilating. A 4° Tw. sodium sulphide liquor is prepared at a temperature of 80° F. The stock is entered and run through the day, being allowed to remain in the paddle overnight. The following morning the stock is run for one hour, and, if the skins are in the proper condition, the excess of sulphide is removed. The paddle is filled with hot water, the skins run for one-half hour, and the water drawn off. The paddle is again filled with water, 2 per cent of sodium bicarbonate introduced, and the stock run for one-half hour. A full head of water is now turned on and the stock washed for three hours.

Bating. The bate used for this stock should be Oropon A B and should consist of $\frac{1}{2}$ per cent on the weight of

the stock at 90° F. The stock, being entered in the afternoon, is allowed to remain overnight. The following morning it is removed and is ready for pickling.

Pickle. The pickle to be used should be made up with 8 lbs. of salt to 1 lb. of sulphuric acid, using 12 per cent of salt on the weight of the stock at a concentration of 1 lb. of salt to 1 gal. of water. The skins are run in this pickle for 1½ hours, horsed up to drain, and sammied.

Degreasing. The sammied stock is now degreased with Vacuum Oil Degreasing Compound and allowed to drain.

Tanning. The weighed, pickled, and degreased stock is placed in the mill and a solution of

Sodium dichromate,	6 per cent
Salt,	4 per cent
Sulphuric acid,	2 per cent

with the necessary amount of water added through the trunnion while the drum is in motion. The skins are run in this solution for 1½ hours and then horsed up overnight to drain. The following morning the skins are returned to the drum and a solution of

Sodium bisulphite,	6 per cent
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in the necessary quantity of water added and run for 1½ hours. The stock is again horsed up overnight, sammied, and shaved.

Neutralizing. The shaved skins are weighed and placed in the drum, a solution of ½ per cent of sodium bicarbonate added, run for one-half hour, washed for one-half hour, and allowed to drain.

Retanning. A solution of 20 per cent quebracho extract, standing at 20° Bk., is prepared, added to the stock while the drum is in motion, and run for one hour. The excess of liquor being drawn off, the stock is washed for fifteen minutes, a solution of $\frac{1}{4}$ per cent of tartar emetic at a temperature of 125° F. added, and run for one-half hour.

Coloring. The stock is now in the proper condition and may be colored with any combination of basic colors desired.

Fat Liquoring. The colored and washed stock is best fat liquored by using 3 per cent anhydrous Turkey-red Oil at a temperature of 140° F.

Finishing. The same as given under *Calf*.

322. Quebracho-tanned Sheepskins. The pickled skins are weighed and placed in the mill. A solution of 30 per cent of quebracho extract on the weight of the stock is prepared at a concentration of 30° Bk. To this solution is added 10 per cent of salt on the weight of the stock, and the whole added to the skin in the mill. The mill is now set in motion and run for $1\frac{1}{2}$ to two hours, or until skins are tanned. The plugs being drawn and the water turned on, they are washed until perfectly clean. If desired, the washing may take place in a separate mill, and the new lot of skins run in the spent liquor for one-half hour to utilize the unabsorbed tan. The plugs being drawn, a new liquor of 25 per cent of quebracho at 30° Bk. with 10 per cent salt will suffice to complete the tannage. The plugs are then returned, the above liquor added, and the milling continued for $1\frac{1}{2}$ hours. The washed stock is now fat liquored with 1 per cent of anhydrous Turkey-red Oil at a tempera-

ture of 110° F. for one-half hour, washed again, set out, shaved, and hung up to dry. When dry it is degreased, buffed if necessary, and colored, in the following manner:

Preparation of the Stock. A very important factor in the coloring of vegetable-tanned leather is the proper preparatory treatment of the material. The stock is placed in the mill and run in water for some time in order to clear the grain. Two per cent of sumac is now added and milled for about one hour. It is then washed to remove adhering tanning material. Should the leather be very dark in color, it is customary to treat it with dilute sulphuric or formic acid, using about 1 lb. of acid for each 100 lbs. of water, running leather in it for about twenty minutes. The excess of acid is removed by washing in running water.

Very fatty leathers become dirty and specked and make an imperfect product when finished. In order to prevent this defect, they are treated with benzine to remove the fat. For leathers that are not too fatty, a treatment with water containing $\frac{1}{2}$ part of borax or soda per 1000 is sufficient. After milling, the goods are well rinsed and again milled for half an hour in a liquor containing 12 oz. of lactic acid per 20 gals.

Mill Coloring, Basic Colors. The tanned stock is introduced into the mill with 2 per cent of sumac, and sufficient water to float the stock, and is run for one-half hour. One-half per cent of tartar emetic is added and the milling continued for one-half hour longer. The plugs are drawn and the stock thoroughly washed.

The color solution is prepared by dissolving the necessary amount of color in water at 115° F., which has been

made acid with formic acid (4 oz. per 100 lbs. of stock). The plugs being returned to the drum, sufficient water at 115° F. to float the stock is introduced, and the mill put in motion. One-third of the color solution is added through the trunnion and run for three minutes; a second third is added and run for two minutes; and finally, the last portion is introduced. The stock is then run for one-half hour, washed thoroughly, and nourished with 1 to 4 per cent of Turkey-red Oil. On removal from the drum, the stock is set out carefully. Sometimes it is advisable to oil off before setting. This is done with a mixture of neatsfoot oil and glycerine applied before the stock is set out.

Paddle Coloring, Basic Colors. The stock is most conveniently retanned in a drum and the color set by means of tartar emetic or potassium titanium oxalate, as described above. In preparing the color solution in the paddle, the necessary amount of water at 115° F. is introduced and 4 oz. of formic acid for each 100 lbs. of stock added. With the paddle in motion, the wet skins are entered in a spread condition and run for one-half to one hour, or until of the required depth of color is obtained. On removal, they are oiled, if desired, and set out.

Tacking. The skins, which have been well set out, are tacked on the frames, care being taken to get all the spread that is possible. When dry, they are stripped, and may be finished as desired.

Roll Finish. A flaxseed liquor is prepared by boiling 1 lb. of flaxseed in 3 gals. of water and straining off the solid portion. The liquor thus prepared, when cold, is applied by means of a sponge to the grain side of the

stock. Before the stock has become dry it is rolled, brushed, and staked. A coat of leather seasoning is applied, and, after the stock has dried, it is rolled again. This treatment produces a bright finish that usually suffices for this grade of stock.

Glaze Finish. Should a higher glaze be desired, the stock may be jacketed after the application of the leather seasoning.

Ooze Finish. In preparing the stock, great care must be taken. For this variety, either the grain or flesh side may be finished, although the flesh side is preferable. The tanned stock, after drying, is carefully buffed on the flesh side, first using coarse, and then very fine emery on the wheel. The leather may be colored as given above, washed and fat liquored with 1 per cent of anhydrous Turkey-red Oil, carefully set out on the flesh side, and tacked. On removal from the boards, the skins are trimmed, staked lightly, blocked, and dry milled.

323. Dark Tan Chrome. The following formula may be used on calf or side leather. The proportions given are for 100 lbs. shaved weight.

Coloring. The washed stock is run for one-half hour in the mill with

Fustic (powdered)	3 lbs.
Water,	30 gals.

at a temperature of 125° F.

A solution of

Titanium potassium oxalate,	4 oz.
Water,	10 gals.

is prepared at a temperature of 125° F., added through the trunnion to the contents of the mill, and run for fifteen minutes longer. The stock is then washed for five minutes with warm, running water (temperature about 125° F.), and the color solution prepared with

Flavo-phosphine G G Conc.	22 oz.
Vesuvine R,	3 oz.
Methylene blue B,	$\frac{1}{8}$ oz.
Water at 140° F.,	40 gals.

Ten gals. of water at 150° F. is added to the stock, the door is closed, and the mill set in motion. One-third of the color solution as prepared above is strained and added through the trunnion; at the expiration of five minutes, a second portion of the strained color is introduced; and, after three minutes, the final stained portion. The stock now run for one-half hour, the excess color solution removed, the stock washed, and the following fat liquor introduced through the trunnion:

Victoria Fat Liquor B X,	3 lbs.
Ammonia,	6 oz.
Egg yolk,	1 lb.
Water at 100° F.,	20 gals.

running for one-half hour longer.

The stock should be washed with warm water until clear, then set out carefully, and hung up to dry. The dried stock is sammied by dipping in hot water and placing in piles overnight, or until soft. The damp stock is staked and tacked. After drying on the boards the stock is stripped, trimmed, restaked, given a coat

of flaxseed liquor, and, while yet damp, brushed and rolled. A coat of leather season is then applied, and the stock dried, brushed, and rolled.

324. Acid and Direct Colors on Chrome-tanned Leather.

The tanned and shaved stock is placed in the drum with the necessary amount of water to float the skins at a temperature of 140° F., and the mill set in motion. A solution of borax, using 1 per cent on the weight of the stock, is prepared, added through the trunnion, and run for one-half hour. The plugs are now drawn and the stock washed in warm running water for fifteen minutes. The plugs being returned, the stock is floated with water at 125° F., and 3 per cent of developed fustic dissolved in water at the same temperature introduced. After running the stock for one-half hour in the fustic, the excess of liquor is drawn, and $\frac{1}{4}$ per cent of titanium oxalate at 140° F. added to the contents of the drum and run for fifteen minutes. (For light shades tartar emetic may be used in place of the titanium salt.)

The skins being washed from the titanium salt, a dye-bath is prepared, using, for example, the following colors:

Fast Leather Yellow RXX,	$\frac{1}{2}$ per cent
Fast Leather Brown R,	1 per cent
Fast Leather Red B,	$\frac{1}{4}$ per cent

in water which has been softened with 1 per cent of borax at a temperature of 140° F. Sufficient water at 140° F. to float the stock is placed in the drum, the latter set in motion, and the above color solution introduced in three portions. After the stock has run for one-half hour with the color, 3 per cent of formic acid diluted

with water is slowly added and run for fifteen minutes longer. The excess of color is next drawn.

Now prepare a fat liquor by dissolving

Anhydrous Turkey-red Oil, 2 per cent

in water at 140° F. and add this also to the contents of the drum, running for one-half hour. Next, remove the plugs, wash well with warm water, horse up, set out, hang up to dry, sammie by dipping in hot water, mill, stake, and tack. The stock may then be finished as desired.

325. Fast Brown on Chromed-tanned Calf or Side Leather. The well-neutralized stock is run for half an hour with 5 per cent of gambia extract, or 3 per cent mat gambia, at a temperature of 125° F. One-half per cent of titanium potassium oxalate at a temperature of 140° F. is introduced and run for one-half hour longer. The stock is washed for five minutes with warm running water and drained. Sufficient water at 140° F. to float the stock is placed in the drum and 1 per cent of borax added. The drum being set in motion, the following color solution is added in three portions and run for one-half hour:

Water, 3 times the weight of stock	
Borax,	$\frac{1}{2}$ per cent
Fast Leather Brown 2 R,	$\frac{1}{2}$ per cent
Fast Leather Yellow R W powd.,	$1\frac{1}{2}$ per cent
Sodium Dichromate,	$\frac{1}{2}$ per cent

After running in the above color for one-half hour, 3 per cent of formic acid in a small amount of water is introduced through the trunnion and run for one-half

hour. The excess of color solution is now drawn; $2\frac{1}{2}$ per cent of anhydrous Turkey-red Oil dissolved in water at 160° F. is added, and the mill turned for one-half hour. The plugs being again drawn, the stock is washed with running water, set out, dried, and finished in the usual manner.

326. Letters on Colors.

B.....	Blue
R.....	Red
Y or G.....	Yellow
S.....	Acid
D.....	Direct
F.....	Fast to light
FF.....	Extra fast to light
O.....	Concentrated

Primary Colors.

Blue + Yellow + Red = Black.

Secondary Colors.

Blue + Yellow = Green.

Blue + Red = Violet.

Yellow + Red = Orange.

The addition of the third complementary color to the above, when properly balanced, produces gray, or the tone is grayed or softened by a smaller amount. Thus:

Blue + Red + Yellow = Gray.

Red + Green = Gray.

Blue + Orange = Gray.

Yellow + Violet = Gray.

327. Harmony of Colors.

Scarlet with Blue or Green.
 Gold or Yellow with Blue or Violet.
 Violet with Light Green or Yellow.
 Blue with Yellow or Red.
 Carmine with Green or Orange.
 Brown with Blue or Red.
 Wine with Yellow or Green.
 Rose with Light Blue or Yellow.
 Orange with Violet or Blue.
 Blue Gray with Buff or Pink.
 Olive Green with Red or Orange.
 Flesh with Blue or Dark Green.
 Dark Green with Crimson or Orange.
 Light Green with Rose or Violet.
 Light Brown with Blue or Green.

328. Rule for Mixing or Blending.

Rose, Diluted Magenta.
 Brick Red, Brown and Magenta.
 Golden Brown, Yellow and Brown.
 Wine, Purple and Magenta.
 Royal Purple, Magenta and Light Blue.
 French Red, Magenta and Flesh.
 Navy Blue, Purple and Blue.
 Plum, Magenta and Blue.
 Drab, Yellow and Purple.
 Myrtle Green, Blue and Green.
 Landscape Green, Blue and Yellow, or Green and Yellow.

329. Colors of Different Shades.

Red B + Blue R = Violet.
 Red B + Orange = Red S.

Red G + Yellow R = Orange.

Red G + Violet = Red B.

Blue R + Red B = Violet.

Blue R + Green G = Blue G.

Blue G + Violet = Blue R.

Blue G + Yellow B = Green.

Yellow R + Red G = Orange.

Yellow R + Green = Yellow G.

Yellow B + Orange = Yellow R.

Yellow B + Blue G = Green.

330. Alum Tannage for Pickled Sheep. Triturate 5 per cent of flour with a small amount of water. Dissolve 3 per cent of sulphate of aluminium and 2 per cent salt in 20 per cent of water. Slowly add to this $\frac{1}{4}$ per cent of sodium bicarbonate, and then mix in $1\frac{1}{2}$ per cent of egg yolk and 0.1 per cent of olive oil. Add the flour paste and drum for two hours. Pile up in liquor overnight and then dry at a moderate temperature. Stake, buff, drum with 1 per cent egg yolk and 5 per cent French chalk. Set out, tack, and dry. Season with flaxseed and milk, size with egg albumen, and iron, or glaze. Use shellac finish if desired.

331. Buckskin Leather. Triturate 5 per cent of flour with a small amount of water. Dissolve 3 per cent of sulphate of aluminium and 2 per cent of salt in 20 per cent of water. Slowly add to this $\frac{1}{4}$ per cent of sodium bicarbonate, mix in $1\frac{1}{2}$ per cent of egg yolk and 0.1 per cent of olive oil. Now add the flour paste. The skins being placed in the drum, the above mixture is introduced, the stock run for two hours, then left in the liquor overnight. The following morning the mill is run for one-

half hour, the excess of liquor drawn, and a solution of 10 per cent of gambia at 19° Bk. and 10 per cent of salt introduced. The mill is run for 1½ hours. The stock is now washed, fat liquored with 2 per cent of anhydrous Turkey-red Oil, set out, and hung up to dry. When perfectly dry, the stock is staked, buffed, retanned with 5 per cent of gambia, washed, set out, oiled off with glycerine, and tacked. On stripping, the stock is given a light coat of flaxseed liquor, to which has been added half the quantity of milk, rolled while damp, staked, dried, restaked, and blocked.

332. Acid Hemlock Sole Leather. The beamed stock is placed in the rocker with a 5° Bk. hemlock extract to which has been added 0.6 per cent of sulphuric acid. The stock is allowed to remain in this liquor for about seven hours, when it should be sufficiently plump to transfer to the first tail liquor of the yard. The tail liquor should stand at 8° Bk. and then by stages should be raised to about 16° Bk. In the handlers it should go from 18° to 26° Bk. and in the layers the liquor should be at 40° Bk. The stock should remain in the rockers for eight days, in the handlers sixteen days, and in the layers twenty days. On removal from the layers, the hides are run in the mill with 100° Bk. quebracho extract for about two hours and then piled down for two days. The stock is now stripped with 2 per cent of borax, neutralized with ½ per cent of sulphuric acid, washed, run in sumac at 50° Bk., washed, set out, filled with sugar and Epsom Salts, oiled off with cod oil, dried, sammied, rolled, and, when dry, rolled again.

333. Union Sole Leather. *Soaking.* The green-salted hides are shaken free from salt, trimmed, and cut into

sides. The sides are thrown into water, where they are allowed to remain for twenty-four hours. They are then removed from the water, placed in the drum, and turned for one-half hour. If not sufficiently soft, they are again returned to the still-soak for another day.

Liming. In liming this grade of stock, the hides are first treated with a mixture of 10 per cent of hydrated lime and 2 per cent of sodium sulphide, and left in the liquor for one day. On the second day they are transferred to another paddle or pit containing 10 per cent of lime and 1 per cent of sodium sulphide. On the third day the hides are transferred to straight lime liquor containing 10 per cent on the weight of stock. On the fourth and fifth days they are also changed to straight lime. On the sixth day they are thrown into the warm pool for half an hour, after which they are ready for unhairing.

Unhairing. On removal from the warm pool, the sides are placed on a table, spotted for white hair, and then the remainder of the hair removed by the unhairing machine or over the beam.

Fleshing. The unhaired stock is next fleshed on the machine and thrown into a wash-paddle for one-half hour. It is subsequently skudded on the beam, then placed on sticks and put into 0.6 per cent solution of lactic acid overnight.

Rockers. On removal from the acid liquor the sides are placed in the tail rocker liquor, which consists of an 8° Bk. solution of 70 per cent hemlock extract, 20 per cent chestnut extract, and 10 per cent myrabolans, with an acidity of 0.6 per cent. The liquor is changed from day to day until, by pressing forward, it increases in strength to 12° Bk. This should take fourteen days.

First Layer. The stock, as it comes from the head rocker, is placed in the first layer liquor where it is further tanned in a mixture of 50 per cent of hemlock and 50 per cent of chestnut, standing at 20° Bk. The liquors are pressed forward for twenty days, the stock coming out of a 25° Bk. solution.

Second Layer. The second layer is made up of straight chestnut extract at 30° Bk., and the stock remains in it for seventeen days. As the sides are thrown into the pit, each one is sprinkled over with chestnut oak bark chips.

Third Layer. The third layer liquor is made up of a 40° Bk. ordinary quebracho extract, and the stock is piled down in it for thirty days.

Hot Liquor. As the hides are removed from the last layer, they are thrown into a warm 40° Bk. quebracho liquor for about one hour and then, after pressing, are transferred to the mill.

Extracting. After the hides have been placed in the mill, a 100° Bk. ordinary quebracho extract at 130° F. is introduced, using a sufficient quantity to give 2 lbs. of extract to a side. The stock is run in the liquor or extracted for one hour.

Tempering. From the extract mill the stock is thrown into a tempering pit containing 40° Bk. clarified quebracho extract and allowed to remain for four days.

Bleaching. The stock from the tempering pit is placed on sticks and dipped for five minutes in a warm 1 per cent solution of sodium carbonate, followed with a five minutes' dip in warm water, then in a 1½ per cent solution of sulphuric acid, again in a ½ per cent solution of sulphuric acid, and finally in warm water. From the bleach the stock is again pressed and is ready for filling.

Filling. To the stock in the drum is added a strong solution of 2 lbs. of glucose syrup, $1\frac{1}{2}$ lbs. of magnesium sulphate, and 1 lb. of sole leather oil for each side. The hides are then run in this compound for one-half hour.

Drying. Sole leather should be dried out very slowly by placing it in a cold, dark loft. After two days the temperature may be raised if desired.

Sammie. The dried sides, on removal from the loft, are dipped in a warm, dilute solution of glucose and magnesium sulphate and piled down overnight.

Rolling. The wet stock is given a liberal coat of sole leather sponging compound and then rolled while still wet. This should be done in the morning so that the stock may again be rolled in the afternoon when dry.

Brushing. Many tanners do nothing more after rolling, but some prefer to apply a coat of brushing compound, and, when this is dry, pass the stock through the brushing machine.

334. Patent Leather Daub. (For heavy leather, bark or chrome sides.)

For the "sweet-meat" coat (called the "short-daub"), boil 40 lbs. linseed oil to 200° F., add 1 lb. burnt umber, and run temperature up to 600° F. Hold temperature at 600° until "long daub" is obtained, not quite ready to break. Then cool to 525° F. Hold at 525° F. until it breaks short from ladle, then take off from the fire and let stand until it forms a jelly (takes only a few minutes). Now add 70 gals. of 63° naphtha at nearly 400° - 500° F. When naphtha is added, break up all big lumps ("cat-heads") with fork; stir jelly until all lumps are out. Color with 4 lbs. lampblack.

The foregoing is the "slicker coat." For the second coat use either slicker, brush, or sponge; then apply the finish coat of varnish as for Japan kid below. If the first coat of varnish does not look well, apply light coat of varnish on top of the finish.

Dry the daub coat in the sun; the second coat in the steam oven at 150° F. for twelve hours; then in the sun to finish.

335. Long Daub for Japanning Kid. Boil to 550° F. 40 gals. aged linseed oil (Kellogg & Crave aged linseed oil). Hold there until it will pull about 12 ins., then cool down to 130° F. (If oil curdles while boiling, add a little castor oil.) Add 18 gals. amyl acetate and stir until cold. The above is ready for use at any time.

First Coat. Take of the foregoing "long daub" 1 gal. Add 2 gals., 8 or 9 oz., "cotton solution." Add amyl acetate to make the specific gravity equal 24° Bé. at 60° F.

Second Coat. Same as first coat, only reduce specific gravity to 26° Bé.

For coloring use A. A. Blue.

Varnish for the above:

40 gals. aged Linseed Oil. Heat to 300° F.,
10½ lbs. China Blue.

Run heat up to 600° F. Hold temperature until you get a varnish; then let cool down till cold. Add benzine and turpentine (or "Turpsine") to make specific gravity equal 38. Let varnish stand one week before using. Apply with sponge. Dry this varnish coat in oven twelve hours at 150° F.

336. One-bath Chrome Liquor No. 1. Chrome alum 25 parts, sal-soda, 5 parts, or until Congo paper is turned blue. To keep the stock well open, it may be advisable to add about 2 per cent of salt.

337. One-bath Chrome Liquor No. 2. Dissolve

Sodium dichromate,	10 lbs. in
Water,	8 gals. and add
Sulphuric acid	10 lbs.

Now introduce slowly

Syrup glucose,	6 lbs.
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This solution should be made up to 20 gals. with water.

338. One-bath Chrome Liquor No. 3. Dissolve

Sodium dichromate,	10 lbs.
Salt,	10 lbs.
Water,	8 gals.

Add to this solution

Sulphuric acid,	5 lbs.
Sodium bisulphite,	10 lbs., dry

Make up to 20 gals. with water. This liquor contains $\frac{1}{2}$ lb. of dichrome per gallon.

339. One-bath Chrome Liquor No. 4.

Sodium dichromate,	10 lbs.
Water,	10 gals.
Glycerine,	6 lbs.
Sulphuric acid,	10 lbs.

Make up to 20 gals. with water.

340. Mat Calf Finish. Boil for one-half hour

Irish moss,	1 lb.
Potassium ferrocyanide,	3 oz.
Water,	3 gals.

Strain; then add $\frac{1}{2}$ lb. of flour, mixed to a paste, and boil again. To this add 5 oz. of nigrosine and make up to 5 gals. with water.

341. Gun Metal Finish. Boil for one-half hour

Nigrosine,	6 oz.
Flaxseed,	8 oz.
Chip soap,	8 oz.
Water,	4 gals.

When cool, strain and add water to make 8 gals. Give stock first finish of glaze, then iron, then coat of above finish, and hang up to dry. If harder finish is desired, add 4 oz. of casein and 2 oz. of ammonia together with the above.

342. Gun Metal and Glaze Finish. First make stock of nigrosine liquor by dissolving 1 lb. of nigrosine in 1 gal. of boiling water. Now make up a lactic acid wash with 2 qts. of lactic acid to 4 gals. of water. Add to this 1 pt. of nigrosine stock.

For the glaze, to make 4 gals., take

First Finish for Glaze.

Stock nigrosine,	1 qt.
Logwood liquor (2 gms. in 1 qt.)	1 qt.
Water,	10 qts.
Glycerine solution (1 to 4) ^v	1 qt.
Blood (2 gms. dry in 1 qt.)	2 qts.
Alcohol,	1 qt.
	<hr/>
	16 qts.

Top Finish for Gun Metal.

First finish,	2 gals.
Water,	1½ gals.
Milk,	1 qt.
Alcohol,	1 qt.
Glycerine,	4 ozs.
Leather yellow,	¼ oz.

or enough to make a dark green tint.

Top Finish for Glaze.

First finish,	3 gals.
Water,	1 gal.

If stock is oily, add a little lactic acid. If it is desired to keep the stock solutions, add 2 oz. of white arsenic, or 2 oz. of formaldehyde with a little almond oil extract.

Stock nigrosine,	1 qt.
Logwood,	¼ oz. in
Water,	1 qt.
Blood albumen,	1 qt.
Milk,	1 qt.

If high glaze is desired, omit the milk and add 1 more quart of blood. Then add 6 ozs. of glycerine in 1 qt. of water. Now add sufficient water to make 3¼ gals. and then 1 qt. of denatured alcohol. The foregoing formula should be put together in the following order:

First Finish for Gun Metal.

Stock nigrosine,	1 qt.
Logwood solution,	1 qt.
Water,	10 qts.
Glycerine solution,	1 qt.
Milk,	1 qt.
Blood,	1 qt.
Alcohol,	1 qt.
	<hr/>
	16 qts.

343. Mat Finish. Soak overnight

Irish moss,	$\frac{1}{4}$ lb.
Gum tragacanth,	1 oz. in
Water,	$\frac{1}{2}$ pail

Dissolve in the above after straining

Logwood,	6 oz.
Neut. chrome,	1 oz.
Corvaline,	2 oz.
Diamond green,	$\frac{1}{4}$ oz.
Bark extract,	3 oz.

Now boil for about one hour, strain, and use when cool.

344. Russia Soap Finish. The following is for 50 gals. of finish:

White chip soap,	4 $\frac{1}{2}$ lbs.
White glue,	3 lbs.
Egg albumen,	1 $\frac{1}{2}$ lbs.
Birch oil,	2 lbs.
Flaxseed liquor,	2 lbs.

345. Patent Brilliant Finish. For 50 gallons use

Nigrosine,	6 $\frac{1}{4}$ oz.
Corvaline,	1 $\frac{9}{16}$ oz.
Methylene blue,	$\frac{1}{2}$ oz.
Gelatin,	6 $\frac{1}{4}$ oz.
Prepared blood	2 lbs. 5 $\frac{1}{2}$ oz.
Ammonia,	2 oz.

Give one coat and glaze, then second coat and glaze again. Oil off with mineral oil. A good oil for oiling off mat finish is mineral seal oil to which a small amount of olive and birch tar oil has been added.

346. Glaze Finish. Dissolve 4 oz. of hematine crystals in 1 gal. of boiling water, add 1 $\frac{1}{2}$ oz. of fustic extract and 3 oz. of nigrosine. In a separate vessel soak up overnight 1 $\frac{1}{2}$ oz. of blood albumen in 1 qt. of water. Make up a gum tragacanth paste, 60-1000, and add 1 pt. of it to the logwood solution. Make up a flaxseed liquor by boiling 1 lb. of flaxseed in 3 gals. of water, and add $\frac{1}{2}$ pt. to the above. Finally, add the blood albumen solution to the mixture when cold and strain through muslin.

To preserve the above put in $\frac{1}{2}$ oz. of carbolic acid or 1 oz. of birch tar oil. Make up to 3 gals. before straining.

347. Cotton Finish. Mix together to a smooth jelly

Flexible compound,	1 pt.
Soluble cotton,	$\frac{1}{2}$ pt., 16 oz.

Dissolve $\frac{1}{2}$ oz. of Japan black in

Alcohol,	1 pt.
Amyl acetate,	1 qt.
Benzole,	1 pt.

Now mix the alcoholic solution with the cotton jelly. If too thick, add more alcohol. The same may be used without the black, or with other colors.

348. Glazing Finish.

I. Dissolve

Borax,	3 oz.
Hot water,	1 gal.

Bring to a boil and add

Shellac,	1 lb.
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Stir until dissolved and then add

Ammonia,	1 tablespoonful (1 oz.)
Glycerine,	1 tablespoonful (1 oz.)

II. Dissolve

Gelatin,	$\frac{1}{2}$ lb. in
Hot water,	1 gal.

Use No. I alone or mixed with No. II.

349. Finish for Splits. Prepare a 60 per cent cotton solution from 16 oz. soluble cotton by using amyl acetate.

First coat:

Cotton solution,	40 per cent
Castor oil,	60 per cent

Colored with drop black and embossed either here or later, as desired.

Second coat:

Cotton solution,	60 per cent
Castor oil,	40 per cent

Third coat:

Cotton solution, 70 per cent
 Castor oil, 30 per cent

350. Amyl Acetate Finish. Dissolve

Soluble cotton, 16 oz. 2½ lbs.
 Amyl acetate, 2½ gals.

Mix the above with 2½ gals. of alcohol and then add

Castor oil, 1 lb.

Dissolve 1 oz. of spirit nigrosine N in ½ pt. of alcohol and add to above. If a dull finish is desired, dull with lactic acid.

351. Gray Ooze. Run for fifteen minutes with

Ferrous sulphate, 2 per cent on dry weight

Wash for one-half hour and top with

Silver Gray G 2 gms. for 4 skins

Run for fifteen minutes and wash fifteen minutes. Fat liquor with

Chip soap, 1 per cent
 Neatsfoot oil, ½ per cent

352. Brown Ooze. Mordant the stock with sumac or quebracho and color with

Vesuvine R 3 oz. per 100 lbs.
 Flavo-phosphine G G Conc. 1 oz.
 Methylene Blue, ¼ oz.

353. Black Ooze. Place the wet chrome-tanned skins in the drum and add

Logwood paste, 3 per cent
 Iron striker, 1 per cent

Wash thoroughly and fat liquor with a mixture of soap, Neatsfoot oil, and soluble oil, using 3 per cent of the mixture. Dry and buff. The buffed skins are now sam-mied and run in the drum for twenty-five minutes with

Logwood,	$1\frac{1}{2}$ per cent
Fustic,	1 per cent
Copperas,	$1\frac{1}{2}$ per cent

Wash five minutes. For each 100 lbs. add

Diamond Green B powdered, 3 oz.

Run ten minutes and add

Corvaline B T,	1 lb.
Vesuvine V L II	3 oz.

Run thirty minutes, wash, and add 2 per cent acid fat liquor. Run in fat liquor for thirty minutes, hang up to dry, dip in hot water, and run in dry mill.

354. Bronze Calf Finish. Make lactic acid wash with 1 pt. lactic acid and 8 pts. of water. Then dissolve $\frac{1}{4}$ oz. Acid Violet in 8 oz. of denatured alcohol. Dissolve $\frac{1}{4}$ oz. fuchsine crystals in 8 oz. of hot water. Mix the Acid Violet and fuchsine solutions together and add a little to the lactic acid wash. Apply this wash to the skins; then make a solution of $\frac{1}{4}$ oz. of Acid Violet in 8 oz. of alcohol and give one coat. Dry and glaze; then apply a solution of $\frac{1}{4}$ oz. fuchsine and $\frac{1}{8}$ oz. of lactic acid in 8 oz. of hot water. Dry, glaze, and give coat of finishing oil.

CHAPTER IX

WOOD FIBER, PULP, AND PAPER

355. On a laboratory scale, it is impossible to go very extensively into the manufacture of paper without entailing great expense. A few simple tests may be performed, however, which will enable the student more fully to appreciate some of the operations of this very important industry.

Paper consists of cellulose fibers matted or felted together in a sheet. The raw materials employed are wood pulp, cotton or linen rags, esparto, straw, hemp, flax, jute, etc.

356. Wood Pulp. As the bulk of paper on the market is made entirely or in part from wood fibers, this raw material will be the only one considered in this chapter. For a more detailed account of the processes involved the student is referred to the "Manual of Industrial Chemistry" by the same author. Wood pulp is of two kinds, mechanical and chemical.

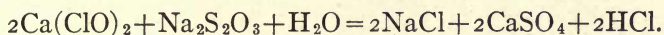
357. Mechanical Wood Pulp. Mechanical pulp may be made in the laboratory by forcing a stick of poplar, hemlock, spruce, pine, or basswood against a revolving emery-wheel, over which a small stream of water plays continuously. The resulting pulp is passed through several screens to remove insufficiently disintegrated particles. The finely divided pulp is then brought into a settling

tank, where it is washed, bleached, filled, and colored as subsequently described under the treatment of chemical pulp.

358. Chemical Pulp. Chemical pulp is prepared by the soda, the sulphite, or the sulphate processes. The soda process is largely used for soft woods. The bark is removed by shaving. The wood is then chipped in a machine, or by hand, cutting across the grain. To separate the knots, the chips are thrown into water, where the heavy parts go to the bottom, leaving the clear wood on top. The digester is nearly filled with chips, which are then covered with a caustic liquor of about 10° Bé. The cover of the digester is safely screwed in place, and the charge heated until a pressure of 90 lbs. is obtained. This pressure is held from eight to ten hours. The effect of this "cooking" is to reduce the wood to a soft mass of grayish-brown color, while the liquor becomes dark brown and somewhat increased in density. Now open the valve of the digester very slowly in order to release the pressure. *Much care must be taken here to avoid any possibility of an explosion.* If the process has been conducted properly, the stock will all crumble to a pulp, which, together with the "black liquor," is transferred to a suitable apparatus and systematically washed, the wash waters being saved until their density falls below 8° Bé.

This liquor may be evaporated to a density of 38° Bé. and then burned. The "black ash" thus obtained may be recausticized with milk of lime, and from 80 to 90 per cent of the original soda recovered. The pulp obtained above is thoroughly washed, then passed through a screen to remove the larger particles.

359. Bleaching. The washed pulp is next placed in a vessel provided with some form of agitator where it is mixed with a weak solution of calcium hypochlorite. Only a clear solution of bleaching powder should be used, so that no dirt will be introduced into the insoluble residue, causing spots in the paper. If the liquid is heated to 90° or 100° F., or a little acid added, the process is hastened. Alum forms aluminium hypochlorites with bleaching powder solutions, which is very effective; a slightly acid alum or "bleaching" alum is commonly employed. From 18 to 25 parts of bleaching powder is used for each 100 parts of pulp. As soon as bleached, the process is stopped, otherwise the fiber is apt to be chlorinated, and color again taken up. The excess of hypochlorite in the pulp is washed out with water, or is destroyed by adding an antichlor, such as sodium thio-sulphate. The action of the antichlor is as follows:



The pulp must be thoroughly washed after bleaching, even when antichlors are used, since injurious substances may be left in the pulp.

Other materials than bleaching powder, such as ozone, hydrogen peroxide, sulphurous acid, liquid chlorine, or sodium peroxide, have been suggested for bleaching, but as yet the hypochlorites are most common.

360. The Paper Making Process. The first operation is "furnishing" or "charging" the stock; the kinds and quantity of material employed depend on the quality of the paper to be produced. In order to give the paper body, weight, and greater smoothness, mineral filler or "loading" material is used. This must be exceedingly

fine, not too high in specific gravity, and insoluble in water. It must be free from dirt, grit, and mica. The loading is done after the fiber has been well beaten with water. The filler is thoroughly mixed with pulp, then the sizing material added, and the whole beaten until a perfect mixture of all the materials is obtained. Some of the fillers adapted to paper making are clay, chalk, and calcium sulphate; while a good size may be prepared from rosin with a concentrated solution of soda ash. The proportion of filler and size varies within quite a wide range; good results, however, may be secured with 5 per cent of filler and 3 per cent of size on the weight of the moist pulp. After the pulp, filler, and size have been well beaten, a solution of aluminium sulphate is added in sufficient quantity to neutralize the soda of the rosin soap. The result of this treatment is to form free rosin and some aluminium hydroxide, which becomes entangled in the openings between the fibers. Paper is usually colored by adding pigments or dyes to the pulp. For white paper, the slight yellow tinge of bleached fiber is neutralized with a trace of blue or pink, ultramarine or coal-tar dyes being used. Some pigments are precipitated in the fiber by adding solutions in the hollander.

The sheet is formed in three different ways, viz., by the hand frame, the cylinder machine, and the Fourdrinier machine.

361. The Hand Frame. For laboratory work the hand frame may be employed. It is simply a rectangular frame covered with wire gauze and having a slight removable ledge around the sides. The frame is submerged in the pulp, mixed to a thin cream with water; when raised, the ledge retains some of the pulp on the gauze, while

the water drains through. At the same time, the frame is slightly shaken from side to side, causing the fibers to "felt" and form a mat of pulp on the gauze. The frame is then inverted over a woolen felt blanket on which the sheet of pulp drops. A number of these pieces of felt, each carrying a sheet of pulp, are piled, one above the other and heavily pressed until the water is expelled. The sheets are then dipped into a fairly strong solution of glue or gelatin and allowed to dry slowly.

The dried sheet is run between a hot roller to produce a final finish. A common photographic burnisher will answer the purpose very well, it simply being necessary to have two polished rolls in place of the usual machine with only one polished roll.

362. Rosin Size. Into an iron kettle place 700 cc. of water and dissolve in this 20 gms. of soda ash. Bring to a boil and slowly add 150 gms. of powdered rosin. A large amount of carbon dioxide is generated, consequently the kettle should be of sufficient capacity to prevent the liquid from going over the side. In order to avoid too great a loss by evaporation, water is added from time to time, the level being regulated by means of a stick with a notch cut at the original height of the liquid. When all of the rosin has dissolved, the mixture is allowed to cool and is practically a 25 per cent solution of rosin soap. A stronger size may be made by using less water.

Casein Size. This size is prepared by dissolving casein in cold soda ash (5 to 1) or borax solution. It is applied in the same manner as that given for rosin size.

363. Colored Paper. Pulp colors may be added to the pulp in the beater in any amount desired to produce the required shade. Very often, however, the color is

produced by direct application of coal-tar dyes, the general method of procedure being as follows:

The pulp and filler are introduced into the pulping machine and well worked. The requisite amount of color previously dissolved in water is now added, worked in thoroughly, and then followed by the rosin size and alum. The well-beaten mass is then formed into sheets and treated as above.

To produce light shades, $\frac{1}{2}$ per cent of color on the weight of the dry pulp is used; for medium shades, 1 per cent; for heavy shades, as much as 3 per cent.

The dyestuffs used for coloring pulp may be any acid color, or direct cotton color. Other colors will require fixing agents.

The following list of colors may be found of service:

Uranine O, soluble	Dianil Red
Eosine, all brands	Dianil Scarlet
Erythrosine, all brands	Dianil Blue
Acid Violet, all brands	Dianil Brown
Naphthol Yellow, all brands	Dianil Black
Orange, all brands	Direct Black

CHAPTER X

USEFUL DATA

364. Thermometer Conversion. In order to convert centigrade (Celsius) into Fahrenheit (above freezing-point), multiply by 9, divide the product by 5, and add 32 to the quotient.

To convert Fahrenheit above freezing-point into centigrade, subtract 32, multiply the remainder by 5, and divide the product by 9.

To convert Réaumur into Fahrenheit, multiply by 9, divide by 4, and add 32 to the quotient.

To convert Fahrenheit into Réaumur, subtract 32, multiply the remainder by 4, and divide the product by 9.

365. Mensuration of Volume. The standard gallon measures 231 cu. ins. and weighs 8.338 lbs.

One cubic foot contains 7.4805 gals. and weighs 998.8 oz. or 62.425 lbs.

To facilitate computation, the weight of a cubic foot of water is usually taken as 1000 oz. or 62.5 lbs.

Water expands from 40° to 202° to the amount of .0467 per cent or .002715 per cent for each degree, giving an increase of 1 cu. ft. in 21.41 cu. ft.

366. To Compute Volume of a Cube. Multiply a side of a cube by itself and that product again by a side.

367. To Compute Volume of a Parallelopipedon. Multiply length by breadth and that product again by depth.

368. To Compute Volume of a Cylinder. Multiply area of base by height.

Example. Diameter of a cylinder is 3 ft. and its height is 7 ft.; what is the volume?

First, to get the area of base, multiply the square of the diameter by .7854, then this product by the height:

$$3 \times 3 \times .7854 = 7.068$$

$$7.068 \times 7 = 49.476 \text{ cubic feet}$$

If it is desired to know the contents in gallons, then

$$49.476 \times 7.4805 = 370.1 \text{ gallons.}$$

369. HYDROMETER SCALE SHOWING RELATIONS OF SPECIFIC GRAVITY, BAUMÉ, AND TWADDELL.

Specific Gravity	Degrees Baumé	Degrees Twaddell	Specific Gravity	Degrees Baumé	Degrees Twaddell	Specific Gravity	Degrees Baumé	Degrees Twaddell
1.000	0	.0	1.180	22	36.0	1.440	44	88.0
1.007	1	1.4	1.190	23	38.0	1.454	45	90.8
1.014	2	2.8	1.199	24	39.0	1.470	46	94.0
1.022	3	4.4	1.210	25	42.0	1.485	47	97.0
1.029	4	5.8	1.221	26	44.2	1.501	48	100.2
1.036	5	7.2	1.231	27	46.2	1.516	49	103.2
1.044	6	8.8	1.242	28	48.4	1.532	50	106.4
1.052	7	10.4	1.252	29	50.4	1.545	51	109.2
1.060	8	12.0	1.261	30	52.2	1.560	52	112.6
1.067	9	13.4	1.275	31	55.0	1.580	53	116.0
1.075	10	15.0	1.286	32	57.2	1.595	54	119.4
1.083	11	16.6	1.298	33	59.6	1.615	55	123.0
1.091	12	18.2	1.309	34	61.8	1.635	56	127.0
1.100	13	20.0	1.321	35	64.2	1.650	57	130.4
1.108	14	21.6	1.334	36	66.8	1.670	58	134.4
1.116	15	23.2	1.346	37	69.2	1.690	59	138.2
1.125	16	25.0	1.359	38	71.8	1.710	60	142.0
1.134	17	26.8	1.372	39	74.4	1.730	61	146.4
1.143	18	28.6	1.384	40	76.8	1.750	62	150.6
1.152	19	30.4	1.398	41	79.6	1.775	63	155.0
1.161	20	32.2	1.412	42	82.4	1.795	64	159.0
1.171	21	34.2	1.426	43	85.2	1.820	65	164.0

370. To Compute Volume of a Cone. Multiply the area of the base by perpendicular height, and take one-third of product.

371. To Compute Volume of a Frustum of a Cone. Add together square of the diameters of greater and lesser sides and product of the two diameters; multiply the sum by .7854 and this product by height; then divide the last product by 3.

Example. What is the volume of frustum of a cone, diameter of greater and lesser ends being 5 and 3 ft., and height 9 feet.

$$\begin{aligned} 5^2 + 3^2 + (5 \times 3) &= 49 \\ 49 \times .7854 &= 38.4846 \\ \frac{38.4846 \times 9}{3} &= 115.4538 \text{ cu. ft.} \end{aligned}$$

372. To Compute Volume of Sphere. Multiply the cube of the diameter by .5236.

Example. What is the volume of a sphere, the diameter being 10 ins.?

$$10^3 = 1000 \text{ and } 1000 \times .5236 = 523.6 \text{ cu. ins.}$$

This rule will apply to the measurement of kettles which, as a rule, are one-half a sphere.

If the kettle is a combination of a sphere and cylinder, the measurement of each may be made and the contents thus determined. In all of the above measurements it is a simple matter to ascertain the number of pounds by multiplying the number of cubic feet by 62.425 and then by the specific gravity of the substance in question.

373. The weight per cubic foot of a few common substances as indicated in the following table, may be found useful:

374. SOLIDS

Substance	Weight per Cu. Ft.	Substance	Weight per Cu. Ft.	Substance	Weight per Cu. Ft.
Charcoal....	27.5	Cannel.....	82.3	Magnesia....	150.
Alum.....	107.	Coke.....	62.5	Magnetic ore.	317.6
Asbestos....	192.	Concrete....	137.5	Marble.....	165.
Asphalt....	140.6	Earth, dry...		Mud.....	101.
Borax.....	107.	soil...	76.0	Quartz.....	166.25
Brick.....	150.	" loose.....	93.7	Red lead....	558.7
Cement,		" moist sand	120.	Ro stone....	123.8
Portland..	81.25	Granite.....	165.	Salt.....	133.
Clay.....	120.6	Gravel.....	137.5	Sand.....	112.
Coal.....		Gypsum.....	135.5	Sulphur....	127.
Anthracite...	84 to 102.5	Lime.....	50.25	Pumice-stone	57.
Caking.....	79.8	Limestone ..	197.25		

375. LIQUIDS AND SEMI-SOLIDS

Substance	Weight in lbs. per Cu. Ft.	Substance	Weight in lbs. per Cu. Ft.	Substance	Weight in lbs. per Cu. Ft.
Acid, acetic....	66.3	Butter.....	58.8	Milk.....	64.5
" Sulphuric....	115.5	Cotton.....	59.3	Oil, linseed..	58.7
" Hydrochloric.	75.	Glycerin 60°.	78.7	Petroleum...	55.
" Nitric.....	70.	Ice.....	57.5	Resin.....	68.
Alcohol 95%...	51.	Lard.....	59.	Starch.....	59.
Ammonia		Leather.....	60.	Sugar.....	100.3
27.9%...	55.6			Tallow.....	58.8

To compute the weight of a body in pounds per cubic foot, divide the specific gravity by 16.

376. To Compute Pressure of a Fluid upon Bottom of its Containing Vessel. Multiply area of base by height of fluid in feet, and product by weight of a cubic foot of fluid.

377. To Compute Pressure of a Fluid upon a Vertical, Inclined, or Any Surface. Multiply area of surface by height of center of gravity of fluid in feet, and product by weight of cubic foot of fluid.

Example. What is pressure upon a sloping side of a pond of fresh water 10 ft. square and 8 ft. in depth?

Center of gravity $8 \div 2 = 4$ feet from surface.

Then $10^2 \times 4 \times 62.5 = 25,000$ lbs.

378. Equivalents of Metric Weights and Measures

1 pound = 453.59 gms.

1 oz. avoirdupois = 28.349 gms.

1 U. S. gallon = 3.785 liters

1 U. S. liquid oz. = 29.574 cc.

Grains per imperial gallon = parts per 100,000.
0.7

Parts per 100,000 $\times 0.7$ = grains per imperial gal.

Grains per U. S. gallon = parts per 100,000.
0.583

Parts per 100,000 $\times .583$ = grains per U. S. gallon.

379. Table of Multiples.

Centimeters $\times 0.3937$ = inches.

Centimeters $\times 0.0328$ = feet.

Centimeters cubic $\times 0.0338$ = apothecaries' fluid ounces.

Diameter of a circle $\times 3.1416$ = circumference.

U. S. gallons $\times 3.785$ = liters.

U. S. Gallons $\times 0.833565$ = imperial gallons.

Gallons, imperial $\times 1.199666$ = U. S. gallons.

U. S. gallons $\times 8.33505$ = pounds of water.

Gallons, imperial $\times 10$ = pounds of water.

Gallons, imperial $\times 4.54102$ = liters.

Grains $\times 0.0648$ = grams.

Inches $\times 0.0254$ = meters.

Inches $\times 25.4$ = millimeters.

Miles $\times 1.609$ = kilometers.

Ounces, troy $\times 1.907$ = ounces avoirdupois.

Ounces avoirdupois $\times 0.9115$ = ounces troy.

Pounds avoirdupois $\times 0.4536$ = kilograms.

Pounds avoirdupois $\times 0.8228572$ = pounds troy.

Pounds troy $\times 0.37286$ = kilograms.

Pounds troy $\times 1.21527$ = pounds avoirdupois.

The square of the circumference of a circle $\times 0.07958$
= area.

380. Board and Timber Measure. In board measure all boards are assumed to be 1 inch in thickness.

381. To Compute the Measure of Surface. When all dimensions are in feet, multiply length by breadth and product will give surface in square feet.

When either of dimension is in inches divide the product by 12.

Example. What is number of square feet in a board 15 ft. long by 16 ins. wide?

$$15 \times 16 = 240, \text{ and } 240 \div 12 = 20 \text{ sq.ft.}$$

382. To Compute Volume of Round Timber. When all dimensions are in feet, add together squares of diameters of greater and lesser sides and products of the two diameters; multiply sum by .7854 and product by one-third of length.

When the length is in feet and diameter in inches, proceed as above, but divide by 144.

If surface or board measure is desired, multiply volume by 12.

383. UNITS FOR COMPUTING SAFE STRAIN THAT MAY BE BORNE BY NEW ROPES, HAWSERS, AND CABLES.

Descrip-	Circumference Ins.	ROPES				HAWSERS		CABLES	
		White		Tarred		White	Tarred	White	Tarred
		3 Strands Lbs.	4 Strands Lbs.	3 Strands Lbs.	4 Strands Lbs.	3 Strands Lbs.	3 Strands Lbs.	3 Strands Lbs.	3 Strands Lbs.
White	2.5-6	1140	1330	—	—	600	—	—	—
"	6-8	1090	1260	—	—	570	—	510	—
"	8-12	1045	880	—	—	550	—	530	—
"	12-18	—	—	—	—	550	—	550	—
"	18-26	—	—	—	—	—	—	560	—
Tarred	2.5-5	—	—	855	1005	—	460	—	—
"	5-8	—	—	825	940	—	480	—	—
"	8-12	—	—	780	820	—	505	—	—
"	12-18	—	—	—	—	—	—	—	505
"	18-26	—	—	—	—	—	—	—	525
Manilla	2.5-6	810	950	—	—	440	—	—	550
"	6-12	760	835	—	—	465	—	510	—
"	12-18	—	—	—	—	—	—	535	—
"	18-26	—	—	—	—	—	—	560	—

Illustration. What weight can be borne with safety by a manilla rope of 3 strands having a circumference of 6 inches?

$$6^2 \times 760 = 27,360 \text{ lbs.}$$

384. WEIGHTS. EVAPORATIVE POWER PER WEIGHT AND BULK OF DIFFERENT FUELS

Fuel, Bituminous	Weight per Cu. Ft. Lbs.	Steam from Water at 212° by 1 lb. Fuel Lbs.	Cu. Ft. in a Ton No.	Fuel, Anthracite	Weight per Cu. Ft. Lbs.	Steam from Water at 212° by 1 lb. Fuel Lbs.	Cu. Ft. in a Ton No.
Cumberland, max.	52.92	10.7	42.3	Peach Mountain..	52.79	10.11	41.6
“ min.	54.29	9.44	41.2	Forest Improve...	53.66	10.06	41.7
Duffryn.....	53.22	10.14	42.09	Beaver Meadow..	56.19	9.88	39.8
Cannel, Wigan...	48.3	7.7	46.37	Lackawanna.....	48.89	9.79	45.8
Blossburgh.....	53.05	9.72	42.2	Lehigh.....	65.32	8.93	40.5
Newcastle.....	50.82	8.76	44	COKE			
Pittsburg.....	46.81	8.2	47.8	Natural Virginia..	46.64	8.47	48.3
Sydney.....	47.44	7.99	47.2	Cumberland.....	31.6	8.99	70.9
Clover Hill, Va...	45.49	7.67	49.2	MISCELLANEOUS			
Cannelton, Ind...	47.65	7.34	47	Charcoal, Oak ...	24.	5.5	104
Scotch.....	51.09	7.08	43.8	Peat.....	30.	5	75
				Pine Wood.....	21.	4.7	106.6

385. FRIGORIFIC MIXTURES.

Mixtures	Parts	Fall of Temperature	Mixture	Parts	Fall of Temperature
Sea salt.....	5	-15° to -25°	Snow.....	1	-40° to -73°
Ammoniumnitrate	5		Calcium chloride..	3	
Snow or powdered ice...	12				
Ammonium chloride.....	5	-5° to -18°	Snow.....	8	-68° to -91°
Snow or powdered ice...	0		Dilute sul- phuric acid	10	

386. INTERNATIONAL ATOMIC WEIGHTS, 1917.

	Sym- bol.	Atomic weight.		Sym- bol.	Atomic weight.
Aluminium.....	Al	27.1	Molybdenum.....	Mo	96.0
Antimony.....	Sb	120.2	Neodymium.....	Nd	144.3
Argon.....	A	39.88	Neon.....	Ne	20.2
Arsenic.....	As	74.96	Nickel.....	Ni	58.68
Barium.....	Ba	137.37	Niton (radium emanation)	Nt	222.4
Bismuth.....	Bi	208.0	Nitrogen.....	N	14.01
Boron.....	B	11.0	Osmium.....	Os	190.9
Bromine.....	Br	79.92	Oxygen.....	O	16.00
Cadmium.....	Cd	112.40	Palladium.....	Pd	106.7
Caesium.....	Cs	132.81	Phosphorus.....	P	31.04
Calcium.....	Ca	40.07	Platinum.....	Pt	195.2
Carbon.....	C	12.005	Potassium.....	K	39.10
Cerium.....	Ce	140.25	Praseodymium.....	Pr	140.9
Chlorine.....	Cl	35.46	Radium.....	Ra	226.0
Chromium.....	Cr	52.0	Rhodium.....	Rh	102.9
Cobalt.....	Co	58.97	Rubidium.....	Rb	85.45
Columbium.....	Cb	93.1	Ruthenium.....	Ru	101.7
Copper.....	Cu	63.57	Samarium.....	Sa	150.4
Dysprosium.....	Dy	162.5	Scandium.....	Sc	44.1
Erbium.....	Er	167.7	Selenium.....	Se	79.2
Europium.....	Eu	152.0	Silicon.....	Si	28.3
Fluorine.....	F	19.0	Silver.....	Ag	107.88
Gadolinium.....	Gd	157.3	Sodium.....	Na	23.00
Gallium.....	Ga	69.9	Strontium.....	Sr	87.63
Germanium.....	Ge	72.5	Sulphur.....	S	32.06
Glucinum.....	Gl	9.1	Tantalum.....	Ta	181.5
Gold.....	Au	197.2	Tellurium.....	Te	127.5
Helium.....	He	4.00	Terbium.....	Tb	159.2
Holmium.....	Ho	163.5	Thallium.....	Tl	204.0
Hydrogen.....	H	1.008	Thorium.....	Th	232.4
Indium.....	In	114.8	Thulium.....	Tm	168.5
Iodine.....	I	126.92	Tin.....	Sn	118.7
Iridium.....	Ir	193.1	Titanium.....	Ti	48.1
Iron.....	Fe	55.84	Tungsten.....	W	184.0
Krypton.....	Kr	82.92	Uranium.....	U	238.2
Lanthanum.....	La	139.0	Vanadium.....	V	51.0
Lead.....	Pb	207.20	Xenon.....	Xe	130.2
Lithium.....	Li	6.94	Ytterbium (Neoytterbium)	Yb	173.5
Lutecium.....	Lu	175.0	Yttrium.....	Yt	88.7
Magnesium.....	Mg	24.32	Zinc.....	Zn	65.37
Manganese.....	Mn	54.93	Zirconium.....	Zr	90.6
Mercury.....	Hg	200.6			

387. Speed of Pulleys.

V and v speed of driving and driven pulleys.

D and d diameter of driving and driven pulleys.

R and r number of revolutions.

$$D = \frac{dr}{R}; d = \frac{DR}{r}; R = \frac{dr}{D}; r = \frac{DR}{d}.$$

In a train of pulleys the final velocity

$$V = VD, D', D'', \text{ etc.} \div d, d', d'', \text{ etc.}$$

388. Unit of Electrical Resistance. The *ohm* is represented by the resistance offered at 0° C. to an unvarying electric current by a column of mercury 14.4521 gms. in mass, of a constant cross-sectional area, and of a length of 106.3 centimeters.

389. Unit of Current. The *ampere* is an unvarying current passing through a solution of silver nitrate in water and deposited at the rate of .001118 gm. per second.

390. Unit of Electro-motive Force. The *volt* is the pressure which, if steadily applied to a conductor having the resistance of one ohm, will produce a current of 1 ampere.

391. Unit of Quantity. The *coulomb* is the quantity of electricity transferred by a current of 1 ampere in 1 second.

392. Unit of Capacity. The *farad* is the capacity of a condenser charged to a potential of 1 volt by 1 coulomb of electricity.

393. Unit of Work. The *joule* is equal to 10^7 units of work in the C. G. S. system and is practically equivalent to the energy expended in one second by an ampere in an ohm.

394. Unit of Power. The *watt*, is equivalent to the work done by 1 joule in one second.

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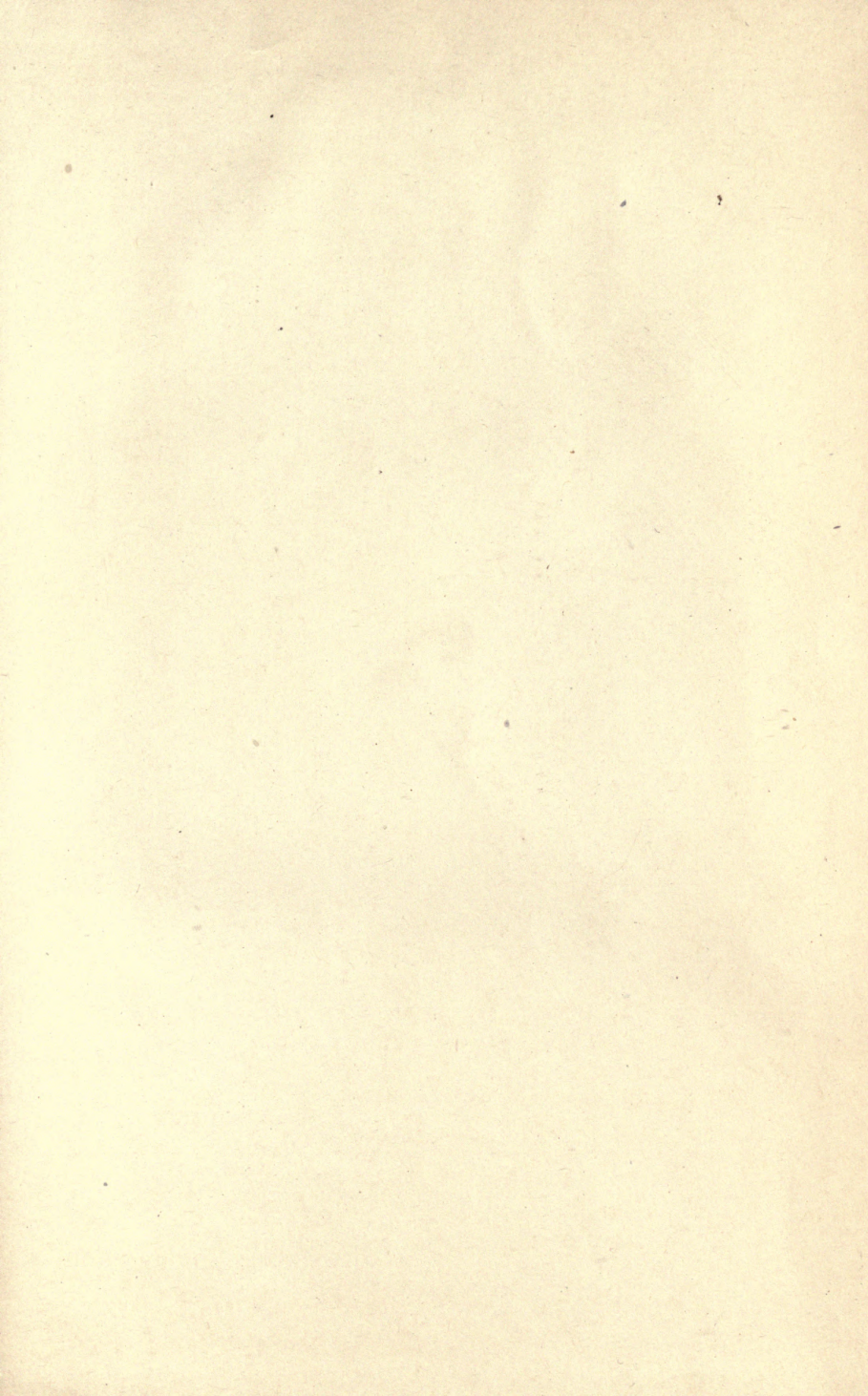
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