

# WOOL DYEING.

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## PREFACE.

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THE wide dissemination of scientific information, and, more important still, scientific method and scientific manner of thought, through the many colleges and schools of technology now in existence, is undoubtedly bearing fruit and exerting a vast influence upon the dyeing trade; and probably at no other period were practical dyers so eager to obtain, or so well prepared to put to practical use, the results of scientific work.

Recent developments in dyeing processes have also mainly been in directions which involve more exact manipulation and the display of greater chemical knowledge; and therefore a scientific as well as a practical knowledge of all works-processes is essential if the maximum efficiency and economy is to be secured. Experience and experiment must work hand in hand in order to produce the best possible results.

In the present volume an attempt has been made to deal with the various subjects treated of, in a scientific manner; the theories which underlie different operations being explained so far as are known. Equal prominence is, however, given to the more practical side, working details of all important processes being attached.

It is hoped that the book will thus prove interesting to all who are either practically engaged in, or in any way connected with, the art of dyeing.

Part II. will be devoted to a description of the mordants and mordant-dyestuffs; and the subject of wool-dyeing will be completed in a third part dealing with non-mordant dyes, dyer's drugs, dyeing machinery, etc., etc.

The publication of the book in parts, and at a low price, will bring it within the reach of all.

WALTER M. GARDNER.

TECHNICAL COLLEGE,  
BRADFORD,  
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## WOOL DYEING.

### I.—THE WOOL FIBRE.

IT is a matter of common experience that the quality of the fibre, as indicated by its lustre, strength, and feel (handle), is frequently much poorer in the finished article than in the original raw material, and it is of the utmost importance to enquire whether this cannot be avoided, at any rate partially, by greater skill and care throughout the different manufacturing processes. But in order to comprehend and thoroughly appreciate the effect which the various stages of manufacture—*e.g.*, scouring, dyeing, finishing, etc., will exert upon the wool fibre, it is essential to have a very clear idea of its physical and chemical composition and properties.

What is wool? From the physical point of view it is a peculiar development of the skin of certain animals, and is closely allied to hair and fur. All these are indeed modifications of the same structure, the differences being entirely of degree, and not of kind. In the original wild condition most animals have in fact two skin coverings, the outer one composed of long, straight, stiff *hairs*, at the roots of which grow much finer, softer, and more numerous fibres, forming the *wool* or *fur*. A little confusion arises from the use of the term fur to signify the long hair attached to the skin used for clothing or decorative purposes, in addition to its employment in the more correct sense indicated above. Sealskin is a true fur, the coarse hairs with which the animal is also covered being removed from the skin during the dressing.

By a long course of cultivation and selective breeding, the outer hairy covering with which sheep were originally provided,

has been in many cases entirely eliminated, and the growth of the wool correspondingly developed; but it is a well-recognised fact that by neglect, and in the absence of suitable selection, the fleece of any wool-bearing animal rapidly degenerates, the coarser fibres again asserting themselves.

Figs. 1 and 2 illustrate this, being respectively diagrams of skin sections of wild and cultivated wool-bearing animals; the absence in Fig. 2 of the long, coarse hairs in Fig. 1, is well shown.



FIG. 1.—SKIN SECTION OF WILD SHEEP.

#### PHYSICAL STRUCTURE.

The mode of growth and structure of a typical wool fibre will be readily understood by reference to Fig. 3, which is a diagrammatic view of a section of the skin of a sheep showing a single fibre *in situ*. (See page 10.)

The root of the fibre is enclosed, as will be seen, in a sort of minute bag or sack, formed by a convolution of the skin. This receptacle is termed the *hair follicle*. In the skin itself there are four distinct layers, the outermost of which is the

*scarfskin* or *cuticle*. It consists of the dried up or dead cells of the underlying *epidermis* or *rete mucosum*, which is the true skin. The third layer from the surface is termed the *papillary layer*, which is filled with a network of minute blood-vessels, glands, and nerves; and underneath this again is the *dermis* or *corium*, in which exist the globular masses which in the illustration have the appearance of bunches of grapes. These are the *adipose cells*, the function of which is to throw off moisture which is carried by the *sudoriparous glands* to the

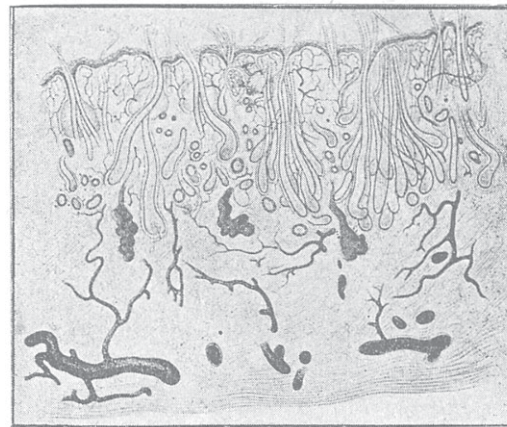


FIG. 2.—SKIN SECTION OF MERINO SHEEP.

surface, and eliminated as perspiration. The pear-shaped receptacles which terminate in the hair-follicle near its orifice, are the *sebaceous glands*, which exude a greasy substance which adheres to the surface of the fibre. This is known as *yolk* or *suint*, and in the natural economy of the animal serves as a lubricant and preservative of the fibre. It is subsequently removed during the wool-scouring process.

Turning our attention now to the fibre itself, the embryo hair in the follicle is produced by the exudation of a plastic lymph which gradually becomes granular or cellular. By the



multiplication of these cells in the globular extremity of the follicle the fibre is gradually pushed up or grows out of the tube of the follicle until it reaches, and eventually projects from, the surface of the skin. There is little doubt that the original cells composing the fibre are all of the same spherical form, the peculiar structural formation of the fibre being largely conditioned by the shape of the follicle. It will readily be seen that if a mass of semi-plastic spheres (representing the cells)

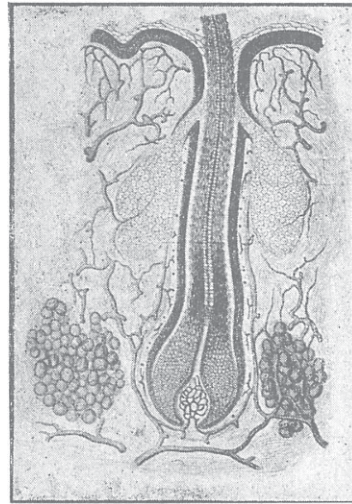


FIG. 3.—DIAGRAM ILLUSTRATING MODE OF GROWTH OF WOOL FIBRE.

are forced up a tube of diminishing diameter (the follicle), by lateral pressure the spheres will be deformed, becoming elongated and of smaller diameter. Friction will cause this effect to be greater at the sides of the tube, the now spindle-shaped cells being there more or less completely flattened into plates. Aggregations of these flattened plate-like cells take the form of lustrous horny scales, which constitute the outer bark or covering of the fibre. These scales, which have been compared to the scales of a fish or to the slates on a housetop,

overlay each other in the manner which the comparison suggests, the free edges protruding more or less from the bulk of the fibre, while the lower or covered edges are embedded and held in the inner layer of spindle-shaped cells. The free edges invariably point away from the root of the fibre just as do the bracts of a fir-cone.

The fact that the surface of the fibre is of a serrated or scaly character is readily proved by gently rubbing a wool or hair fibre between finger and thumb, when it will always travel in the direction of the root of the fibre.

Situated centrally along the axis of the fibre there is usually a well-marked core or pith, termed the *medulla*, or *medullary canal*. This is composed of cells which have retained more or less their spherical character, and to a certain extent their plastic nature.

We are thus able to distinguish three separate formations in the wool fibre: (1) A central cavity filled with comparatively soft and approximately globular cells; (2) surrounding this and forming the larger portion of the fibre, a thick layer of fibrous or spindle-shaped cells; (3) an outer casing of flattened horny scales.

When viewing a transverse section of a wool fibre, there is, of course, no sharp line of division between the three portions indicated above, the change from one to another being quite gradual. The separation into zones, though well marked, is therefore indefinite as regards boundaries.

It will be shown that the physical properties of wool, which render it of such value as a textile fibre, are intimately connected with, in fact are dependent upon, the peculiar structure which has just been described.

#### VARIATIONS IN PHYSICAL STRUCTURE.

When we remember that such apparently dissimilar things as the wool of the sheep, human hair and finger nails, birds' feathers, and horses' hoofs are all of very similar composition, and differ only in structural arrangement, it does not seem im-

probable that wool fibres should vary somewhat in themselves. This, of course, is actually the case; fibres from different breeds of sheep, or even from different parts of the same animal, vary greatly, not only in length, thickness, etc., but also in actual structure. A typical fibre, such as may be obtained from a good Merino or Southdown fleece (See Fig. 4.), will possess the typical structure described above, but frequently the type is departed from to such an extent that the central core of

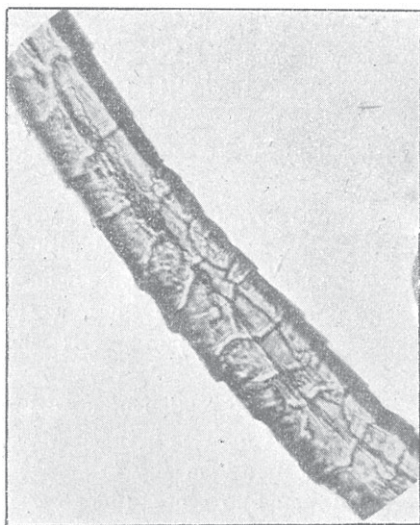


FIG. 4.—TYPICAL WOOL FIBRE.  
(From a Microphotograph.)

globular cells is entirely absent. The serrated character of the outer cells also reaches a much higher state of development in some samples of wool than in others.

#### PHYSICAL PROPERTIES OF WOOL.

It is very interesting, as well as instructive, to examine the various characteristic properties of wool in the light of the peculiar structure of the fibre, which has already been described. The physical properties of wool which render it of

such value as a textile material are: (1) Strength and elasticity; (2) curliness; (3) felting property; (4) lustre; (5) avidity for liquids.

Each of these properties should receive careful attention and thought during all stages of manufacture, so that it may be utilised to the fullest extent in the process which is being carried out, or preserved uninjured either for use in some subsequent process, or to enhance the value of the finished article.

(1) *Elasticity and Strength.*—These are properties which, in common with silk, wool possesses in a greater degree than the vegetable fibres. When submitted to *torsional* strain the wool fibre exhibits a remarkable strength, and when the breaking point is reached the fracture always takes place at the juncture of two rings of the outer scales, the embedded edges of the lower layer being pulled out of their seats. The scales themselves are never broken. By reason of its structure, wool is also able to withstand an enormous *crushing* strain; in fact its resistance is so great that for all practical purposes the effect of any direct pressure may be neglected. The structure is again beautifully adapted to prevent injury by *flexure*. When any elastic body is subjected to a bending strain, compression is produced on the inner surface of the bend and a torsional strain on the outer surface. In the wool fibre, the cells in the central zone being more or less plastic are, no doubt, temporarily deformed, the spindle-shaped or fibrous cells are bent, and the outer scales, being free for a portion of their length, slide over one another to the extent necessitated by the degree of flexure. When the strain is removed the central or medullary cells tend to assume their spherical shape, the fibrous cells to straighten themselves, and the outer scales to resume their original position, thus causing the fibre to straighten. The same remarks adequately explain the behaviour of the fibre, as an elastic body, when stretched. It has been found that the limit of elasticity is from 0.3 to 0.5 per cent of its length, and if stretched beyond this amount the fibre

will not return to its original length, but takes a permanent set; the strength of the fibre is then much impaired, mechanical disintegration having, no doubt, commenced. This is well seen by stretching a white horse-hair, the sudden change from a semi-translucent to an opaque appearance marking the period when the elastic limit is exceeded. The breaking strain of the wool-fibre depends a good deal upon its diameter, which varies considerably, and is usually between 15 and 35 grms. ( $\frac{1}{2}$  oz. to  $1\frac{1}{4}$  oz.). Both the breaking load and the elastic limit are, however, very variable quantities, and are often unequal in different parts of the same fibre. It has also been found that the amount of moisture which the fibre contains at the moment has a certain influence on the strength and elasticity.

These valuable properties of the fibre are, of course, injuriously affected by any process which tends to modify its structural arrangement, or act upon the fibre substance; and it will be seen when the action of reagents upon the fibre is treated of that the various substances used in manufacturing processes behave very differently in this respect.

The strength and elasticity of the fibre are very important factors in the spinning and weaving processes, and it is not too much to say that the milling or felting process could not possibly be conducted in the manner usually employed if the elasticity of the fibre were not capable of taking up the original sudden strain produced by the blow or pressure.

(2) *Curliness*.—The curly, wavy, or twisted character of wool is caused by the unequal contraction of the surface of the fibre, and depends in great measure upon its hygroscopic nature. The curl may temporarily be entirely removed by wetting the fibre in hot water and drying in a stretched condition, or may be artificially induced by unequal drying—a fact which is turned to practical account in the curling of feathers and of hair. The amount of curl in different samples of wool is very variable, being, as a rule, greatest in the finer qualities, and only slightly developed or even entirely absent in coarser varieties. The diameter of the wool fibre varies from  $\frac{1}{2000}$  to

$\frac{1}{500}$  in., and the number of curls from 30 per inch in fine wool, to one or two in the thicker fibres. The property of curliness is most valuable to the spinner, since it enables him to produce a much finer thread than would be possible in its absence; and by reason of the greater development of curl in wool than in cotton it is possible to spin a thread containing fewer fibres with the former than with the latter. The curl has also a considerable, though not a principal, influence in the milling process, as will be explained directly.

(3) *Felting Property*.—The property possessed by wool and similar fibres of felting together to form a commercially valuable fabric, is quite distinctive: silk and the vegetable fibres being incapable of producing such a material. Historically, felting is a much older process than spinning and weaving, a fact of which it is not difficult to give an explanation. Primitive man, clothing himself in skins to which the hair was adhering, would find that in process of time the hair became matted together into a compact mass, especially where it was most subjected to pressure and friction. In an extreme case it might have occurred that the matting together of the hair had gone so far that by cutting it away from the skin a garment of felted hair would be obtained; and it would then be but a small step to produce such a material by purposely applied pressure and friction upon previously shorn hair or wool. In this way the manufacture of felt would naturally long antedate the much more complicated and less obvious process of spinning.

The felting property of wool depends primarily upon the scaly or serrated character of the outer surface of the fibre, but is also influenced, as already mentioned, by its curliness and elasticity. With regard to the action of the latter, in addition to its use in preventing the destruction of the fibre by the sudden strains induced during the process, the elasticity has a direct effect in aiding the felting, because it is evident that if a bundle of elastic threads were alternately stretched and freed, pounded, released, and rolled about, they would



become entangled into a more or less coherent mass. Considering again, for a moment, the action due to the curly nature of the fibre alone, it is equally obvious that a mass of watch hair-springs would become inextricably entangled if treated in a similar manner. In order to understand the action of the fibre-surface it is necessary to remember that the outer scales have protruding free edges which always point away from the root of the fibre. If two fibres, therefore, are placed parallel to each other, root to root and tip to tip, they may even when pressed together be drawn over each other in either direction; but if one of them is turned, so that the fibres lie in opposite directions, and the process is then repeated, the fibres will slide over each other readily if the root end of either is drawn, but will refuse to move if compressed and the tip end of one is pulled. The reason is that in the former case the free edges of the scales of one fibre slide over those of the other, but when drawn in the reverse direction they interlock, causing movement in that direction to be impossible. Fig. 5 will render this clear, but it must, of course, be understood to be merely diagrammatic and enormously exaggerated. (See page 17.)

It is now easy to see that in the early stages of the felting process the curl and the elasticity are the principal causes of the matting together of the fibres, the necessary conditions for the interlocking of the scales, *i.e.*, contiguity and partial parallelism of the fibres in reverse directions, combined with pressure, not obtaining to a great extent. As the process proceeds, however, and the mass of fibres becomes denser, the number of parallel contacts increases, the action of the scales becomes more and more pronounced, and in the later stages is all important.

That the curliness of the fibre is not, as is sometimes thought, the principal cause of felting is evident from the fact that some varieties of wool which are almost without curl will felt quite readily; while, on the other hand, others—of which mohair is an example—possess a well-developed curl, and yet felt with great difficulty. This fact is turned to practical account in the

production of some “curl” and “crepon” cloths, in which wool and mohair are combined together in a woven fabric which is afterwards milled. The wool then felts together and contracts, leaving the mohair in slack threads, which, by reason of the twist in the thread, form little bunches or curls. Alpaca and mohair partake both of the nature of wool and hair. They resemble wool in curliness and fineness of fibre, but are allied to hair in the almost entire absence of free edges to the outer scales; and this is the reason why the mohair does not felt.



FIG. 5.—DIAGRAM TO EXPLAIN FELTING OF WOOL.

Other things being equal, the wool containing the largest number of scales per inch will be the best felting wool; but the most important point is that the scales should have free protruding edges. The reason why “skin” wool felts more easily than “fleece” wool is to be found in the fact that in the former, the lime or other substance used in the process of removing the wool from the skin has caused the scale edges to protrude to an abnormal extent. This indicates that by the action of certain agents the felting property may be increased,

which is, of course, a well-known fact. By adding some acid or alkaline salt to the water used in the milling or felting process, or even by using hot instead of cold water, the scales are caused to stand out more prominently, and the felting action is increased. The greatest possible effect in this direction is produced by milling in a hot acid solution, and to such an extent may the process be then carried that the felt almost acquires the density of wood, and may be sawn and turned in the same manner. Such felt is used for polishing glass and jewellers' work. Felt hats are also made by felting or "planking," as it is termed, in an acid solution.

A microscopical examination of a piece of hard felt shows that the outer scales of the fibre have lost their plate-like form and become partially distended. The fibres also have become conglomerated and welded together in such an intimate and permanent manner that it is difficult to distinguish their individuality. It is evident, then, that when pushed to its extreme limits the felting process brings into play the slightly plastic nature of wool, the fibres being more or less completely cemented together. The ordinary milling process, however, as carried out in cloth manufacture, is of a much less severe character, the integrity of the fibres being maintained, and the flat, horny nature of the outer scales being injured as little as possible; since it is upon the retention of this that the lustre of the finished fabric largely depends.

To sum up in a few words it may be said that the essentials for carrying out the felting process are a wool having a certain elasticity and a large number of well-developed outer scales, which must be submitted to intermittent frictional pressure in the presence of water; the process being accelerated by heat, acids, or alkalies.

(4) *Lustre*.—This quality, in respect of which wool holds a position intermediate between cotton and silk, is due to the reflection of light from the horny outer sheath of the fibre. Of all the physical properties of the fibre it is the most variable in degree; alpaca and mohair surpassing all other varieties

in this respect. The texture of the outer scales in different qualities of wool varies considerably; under the microscope they have sometimes the appearance of polished ivory, others look like porcelain, while some fibres possess scales which may be compared in appearance to clear slightly tinted glass. The effect, however, largely depends upon the method of observation; the same fibre appearing very different when viewed as an opaque and as a transparent object, and under different illuminations, etc.

For the maximum of lustre it is above all things necessary that the outer surface of the individual scales or plates should be polished and glassy. Anything which tends to pit or roughen the scales will, of course, destroy the optical continuity of the reflecting surface, thereby causing dispersion of light with consequent diminished lustre of the fibre; and since all liquids do this in greater or lesser degree the property of lustre is most difficult to maintain throughout manufacturing processes.

It should be generally recognised that acid or alkaline solutions, or even pure water, are capable under certain conditions, of actually *dissolving* the wool substance, and they undoubtedly attack the fibre to some extent during the course of an ordinary scouring or dyeing process.

The lustre of the fibre is not only injured by processes in which solutions are employed, but it is also deteriorated by an excessively great, or too long-continued heat, a fact which will be further touched upon directly.

Of all the valuable attributes of the wool fibre, lustre is therefore the most easily destroyed, and the degree to which it is retained in the finished article depends largely upon the care and skill brought to bear in the different treatments to which the fibre is necessarily subjected during its manufacture into cloth. The moral is obvious.

(5) *Avidity for Moisture*.—Wool is very hygroscopic, readily absorbing moisture from the atmosphere and retaining it with considerable energy. Under average conditions the fibre con-



tains about 14 per cent of its weight of moisture; but the amount is greatly influenced by the hygrometric condition of the atmosphere. In addition to surface condensation the moisture is in all probability located actually within the individual cells, since the membranous cell walls are permeable to water. This is no doubt the reason why it is so difficult to drive off the whole of the water, it being in fact impossible to remove the last portion of the moisture without partial disintegration of the fibre.

By drying wool at 50° C. (120° F.) a loss in weight of from 7 to 10 per cent takes place, and on raising the temperature to 100° C. (212° F.) a further loss of 5 to 8 per cent occurs. After drying at 50° C. the wool will again absorb moisture from the atmosphere up to the original amount, but when dried to 100° C. only part of the loss is regained, showing that some change has taken place apart from the mere loss of moisture. This view is supported by the fact that wool heated for a long time at a temperature considerably below 100° C., gradually acquires a yellow colour, becomes lustreless and brittle, and is much reduced in tenacity and strength.

To account for these effects, Bowman suggests that the water exists in the fibre in two conditions, *i.e.*, as moisture, and as chemically combined water of hydration. But it appears reasonable to reserve a definite opinion on this point until the chemical constitution of the fibre has been determined.

This question of the drying of wool is one of much greater practical importance than is commonly supposed. Wool dried in the open air will always yield an article of higher quality than can be obtained from the same wool when dried by means of artificial heat. A case in point was recently brought under the notice of the writer by a manufacturer of felt hats, who stated that he could always produce a better quality hat in summer than in winter from the same raw material. Enquiry elicited the fact that it was the practice in these works to dry the hat bodies in the summer by placing them in an open shed, whereas in the winter season they were dried, at several

stages of the manufacture, on gratings placed over the steam boilers. This fact sufficiently explained the variable result obtained.

Although it is important that wool should be dried at as low a temperature as possible, it is of much greater importance that the fibre should not be submitted to the action of *dry* heat; that is to say, the drying process should not be continued after the moisture has all been evaporated. In fact, to put it another way, one may say that practically no injury is sustained even with a considerable degree of heat, *so long as moisture is being given off*, but immediately moisture ceases to be evolved, the deterioration above mentioned commences. This naturally suggests a consideration of the methods in vogue for drying

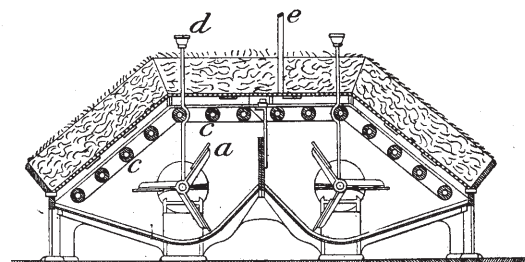


FIG. 6.—WOOL DRYING TABLE, TRANSVERSE SECTION.

wool, and from what has been stated above, the advantages and defects of the different forms of drying apparatus illustrated will be apparent.

Fig. 6 shows in sectional end elevation an ordinary table-drying apparatus. The wool is placed on the top and slanting sides of the machine, which are made of wire grating, hot air being forced through the mass by means of the fans shown in the illustration. It is evident here that some portions of the wool will dry more quickly than others which may not be so favourably situated, and partial over-drying inevitably results. Recognising this, attempts have been made to devise machines in which the wool is sufficiently moved about to ensure equal

drying. In Fig. 7 the wool is carried by the feeding-apron A to the top of the drying chamber, travelling along which it falls on to a lower endless band B, which is moving in the opposite direction. In this manner the wool gradually travels down the machine, presenting a fresh surface to the current of hot air each time it passes from one apron to another, and thereby the drying is considerably equalised. A current of warm air is forced through the apparatus by means of a fan.

Fig. 8 is an illustration of the latest type of wool drying machines. It consists of a framework cylinder of considerable diameter and length, supported on small live rollers, which cause it to rotate on its axis. The bed supporting the cylinder

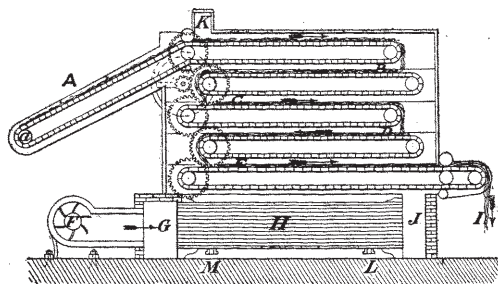


FIG. 7.—WOOL DRYING MACHINE, CONTINUOUS.

is somewhat inclined to the horizontal, the wool being fed into the higher, and delivered from the lower end of the cylinder. The latter is fitted internally with a series of longitudinally placed forks, and at the delivery end is a cowl through which passes warm air. When using the apparatus the wool is fed in at the upper end, and by the rotation of the cylinder is caught by the forks and carried upwards inside the cylinder, hanging from the forks in the best possible position for drying. As the forks approach more and more a vertical position, the wool slips off and falls to the lower portion of the cylinder, to be again caught by the forks and carried upwards. On account, however, of the inclined position of the cylinder, each time th

wool slips from the forks it falls somewhat nearer the delivery end, from which it eventually escapes in a dry condition. In this apparatus, as in those of the type illustrated in Fig. 7, the speed at which the wool travels through the machine, and the heat of the air current, can be so regulated that the fibre is just, and only just dry on reaching the exit.



FIG. 8.—CYLINDER WOOL DRYING MACHINE.

#### CHEMICAL COMPOSITION OF WOOL.

When treating of the structure and mode of growth of the fibre it was pointed out\* that certain glands situated beneath the skin of the animal exude a greasy substance which, coating the fibres to a considerable thickness, serves to protect them from injury. The amount of this encrusting substance is very variable, and on the raw fibre it is always associated with more or less sand, dirt, and other foreign matter; the proportion of pure wool fibre in different samples of raw wool is therefore equally variable, as is shown by the following

\* See page 9 ante.

analyses, which, however, do not, by any means, represent extreme cases:—

ANALYSES OF RAW WOOL AFTER DRYING AT 100° C.			
	No. 1.	No. 2.	No. 3.
Moisture .....	6·26	10·4	8·2
Sand, dirt, etc. ....	11·13	3·1	20·1
Yolk (greasy matter).....	47·30	27·0	40·5
Pure wool .....	35·31	59·5	31·2
	100·00	100·0	100·0

In order to estimate accurately the amount of pure wool in a sample of raw wool, a weighed quantity of the latter should be dried at 100° C, and then successively treated with (1) ether, (2) water, (3) alcohol, and (4) dilute hydrochloric acid; then washed, dried at 100° C for a short time, and weighed. Speaking generally, the finer varieties of wool contain a larger proportion of yolk than the coarser and less valuable sorts.

Confining our attention now to a consideration of the pure fibre, it may first be noted that wool, in common with hair, is chemically the most complex of textile fibres. Cotton, being a carbohydrate, is composed of but three elements, carbon, hydrogen, and oxygen; these elements being present in the proportion represented by the formula,  $C_6H_{10}O_5$ . In the silk substance an additional element, nitrogen, is found; the molecule at the same time being much more complex, as is shown by the formula given for fibroin,  $C_{15}H_{23}N_5O_8$ . Wool contains still another constituent, sulphur; and the simplest formula which will at all conform to the percentage composition contains thirty-nine atoms of carbon.

It has been considered that the wool fibre consists of a definite chemical compound (keratine), to which the formula  $C_{39}H_{65}N_{11}SO_{13}$  has been given; but this view is probably incorrect, the fibre appearing to be composed of a mixture of at least two, and possibly several, closely allied very complex substances. It is possible, and even probable, as was suggested when dealing with the structure of the fibre, that the outer epidermal scales have a different composition to the rest of the fibre; but whether

this is the case or not, we now know with some degree of certainty that wool is not a simple definite chemical compound. This at once accounts for the discrepancy observed in the figures obtained by different chemists on submitting wool to ultimate analysis, the following figures giving an indication of this extent of the variation:—

PERCENTAGE COMPOSITION OF PURE WOOL FIBRE.			
	Mareke and Schültze.	Mulder.	Bowman.
C. ....	49·54 per cent.	50·5 per cent.	50·8 per cent.
H .....	7·29 "	6·8 "	7·2 "
O (by difference).....	24·13 "	20·5 "	21·2 "
N .....	15·60 "	16·8 "	18·5 "
S. ....	3·44 "	5·4 "	2·3 "
	100·00 "	100·0 "	100·0 "

In addition to the above constituents, there is always present in wool fibre a small quantity of mineral matter, amounting frequently to between 1 and 2 per cent; and consisting principally of salts of potassium, calcium, iron, and aluminium, with traces of silica, phosphorus, etc. The character of this mineral matter is, no doubt, largely determined by the nature of the soil upon which the sheep has been pastured.

It will be seen on referring to the analyses of wool which are given above, that sulphur is by far the most variable constituent, sometimes as little as 1·5 per cent, and occasionally as much as 6 per cent, being found. It appears, as was first pointed out by Chevreul, to be present in two different conditions; one portion being in the free state, or only in very feeble combination, while the remainder, which (according to Knecht), amounts to about 30 per cent of the total sulphur, cannot be removed without entire disintegration of the fibre. The sulphur in feeble combination very readily forms sulphides if the wool is brought into contact with metals; and dark-coloured stains are frequently produced in this manner. The formation of black sulphide of lead, when wool is heated with a solution of



plumbite of soda, serves, indeed, as a test to distinguish wool and hair from all other fibres, since they alone contain sulphur.

In order to guard against the formation of these stains it is sometimes necessary to remove the active sulphur from wool which is intended for white or delicately coloured goods, and this may be done by steeping for twenty-four hours in milk of lime, and subsequently washing, then treating with dilute hydrochloric acid to remove the lime, and again washing in water.

That portion of the sulphur which cannot be removed by this treatment is evidently in a different state of combination, since it will not form sulphides, and does not, therefore, produce a black colour with plumbite of soda.

With regard to the chemical *constitution* as apart from the *composition* of the fibre, little is known. When submitted to dry distillation (heated without access of air) it yields first sulphuretted hydrogen and a little carbon disulphide, then ammonium carbonate, and afterwards pyridine, acridine, and a residue or coke of carbon. Schützenberger also obtained nitrogen, carbon dioxide, oxalic and acetic acids, leucine and tyrosine on heating wool with barium hydrate, and these decomposition products tend to show that wool is, or at any rate contains, an albuminoid.

An interesting series of experiments bearing upon the chemistry of wool has been made by Knecht.\* On heating purified wool with sulphuric acid he obtained a light brown solution which readily gives precipitates with solutions of the acid colouring matters. The substance which produces these precipitates or lakes he considers to be identical with the constituent of the fibre which combines with the acid colours in an ordinary dyeing process; the amount of this lake-forming substance being from twenty-five to thirty per cent of the weight of wool.

To obtain the above-mentioned acid solution of wool, 100 parts of the fibre are boiled for about two hours with 200 parts

\* *Jour. Soc. Dyers and Colourists*, August, 1888.

of strong sulphuric acid diluted with 300 parts of water. The wool gradually dissolves, producing a brown solution, which when neutralised by alkali deposits a yellowish white precipitate consisting of the lake-forming substance.

A body called *lanuginic acid* was already known, being produced by a process the reverse of that above described, *i.e.* by dissolving wool in caustic soda and neutralising with acid. Knecht found that an acid solution of lanuginic acid has exactly the same properties as his lake-forming substance, and therefore considered the two to be identical.

Campion, who originally prepared lanuginic acid,\* gave its composition as  $C_{28}H_{60}N_{10}O_{20}$ , but Knecht states positively that it contains sulphur in the inactive condition.

Lanuginic acid not only possesses *basic* properties, as indicated by its forming compounds with acid colouring matters, but also shows well marked *acid* properties, giving precipitates both with metallic hydrates and basic colouring matters. It is thus an *amido acid*, the acid group apparently being stronger than the basic group, since wool dissolves more readily in alkali than in acid.

It is further considered that lanuginic acid is an albuminoid, since it gives the characteristic reactions of that class of bodies.

Knecht does not consider that lanuginic acid exists as such in wool, but that it is a simple decomposition product. This idea is borne out by the fact that wool will not combine with the free sulphonic acid colouring matters, excepting in the presence of free acid; that is to say—since the acid colouring matters are always used in the form of sodium salts—a large excess of acid over and above that required to liberate the free sulphonic acid must be added to the dye-bath. If the wool is *previously* boiled with sulphuric acid it may, however, be dyed direct with free sulphonic acids. Coupled with the above, the fact that wool itself will dye from a colourless solution of

\* *Comptes rendus*, LXXII., 330.

rosaniline base, again shows the superior activity of the acid groups it contains.

Richard\* thinks that the behaviour of wool towards colouring matters and mordants may be explained—as is generally admitted—by the supposition that it contains an amido acid. He has attempted to prove the existence of an amido group by treating wool with nitrous acid and then with an alkaline solution of some phenol (phenol, resorcinol, naphthol, etc.). He thus obtained various brown and red colours upon the wool, which are probably azo colours, the wool substance itself entering into the composition of the colour. However, as Richard himself points out, if the substance obtained on treatment with nitrous acid is a diazo compound of lanuginic acid (or other constituent of wool), it possesses properties differing from those usually associated with diazo compounds, since by treatment with boiling hydrochloric acid it is not decomposed, but is still capable of giving colours with the phenols.

Brein† has made some very interesting experiments which, though not bearing directly upon the constitution of the fibre, may conveniently be noted here, since, taken in connection with the above remarks, they have an important practical significance. He found that if woollen cloth is splashed with alkali or acid and then heated (dried or steamed), those portions which are thus treated will dye up a much darker shade than the rest of the fabric; and he points out that this is probably a frequent cause of irregular dyeing. In all probability the action is a two-fold one, partly chemical and partly physical. It is evident from what has been already stated that in those places splashed with acid or alkali there will be a liberation of active lanuginic acid; and the re-agents will also tend physically to open or raise up the outer scales of the fibre, thus giving the dye solutions freer access to the inner and more easily dyed portion.

\* *Proc. Soc. de Mulhouse*, January, 1888.

† *Zeit. für ang. Chem.*, December, 1888.

*Colouring Matter of Wool.*—The natural colour of wool varies from white to dark brown or almost black; and with regard to the colouring substance little is known. Iron or manganese is always present in the ash of dark wool, and magnesia is frequently found in light-coloured samples; but in all cases an oily organic matter of a similar colour to that of the wool may also be extracted, and it is uncertain to what the colour of the wool is actually due.

#### CHEMICAL PROPERTIES OF WOOL.

The animal fibres—silk, wool, fur, and hair—are chemically of a much more active nature than cotton and other fibrous vegetable products; that is to say, speaking generally (for there are important exceptions), they more readily combine with, or are affected by, chemical reagents.

#### ACTION OF WATER.

The action of moisture on the fibre has already been incidentally alluded to\*; and although pure water at a temperature of about 400° F. is capable of actually dissolving wool, the only noticeable effect of *boiling* the fibre in water for a reasonable length of time is a diminution in its lustre.

Much more destructive is the action of steaming. Scheurer† states that “by steaming thoroughly cleansed woollen cloth at a temperature of 210° F., the fibre is weakened to the extent of 18 per cent in three hours. The destructive action then becomes less, and equals 12 per cent for each successive interval of twelve hours, the total weakening effect amounting in sixty hours to 75 per cent of the original breaking strain—a destructive effect equal to that produced on cotton only after 420 hours’ steaming.”

#### ACTION OF ACIDS ON WOOL.

Wool absorbs acid from solution with much avidity, and retains it with considerable energy. It is not yet established whether the whole of the acid withdrawn from solution exists

\* See p. 19, *ante*. † *Bull. Soc. Ind., Mulhouse*, 1893, p. 89.

in chemical combination with the fibre or some constituent of the fibre; but that a portion at any rate does so is evident from the fact that on long-continued washing, part of the acid is removed as an ammonium salt, the ammonia being eliminated from the fibre substance itself. No doubt, however, the cellular structure of wool, which determines its very absorbent nature, has something to do with the difficulty found in removing the acid by washing.

*Acetic acid* and organic acids generally have very little chemical action on wool under any conditions of practical work; but defects may possibly arise if their solutions are allowed to concentrate upon the fibre. Acetic, oxalic, and tartaric acids are all frequently used in dyeing operations.

*Sulphuric acid*, if sufficiently concentrated, will, as has been already mentioned, dissolve wool. This effect requires acid of 25 to 30 per cent strength—say 45° Tw. Much more dilute acid than this will, however, exert an injurious action, the safe limit being about a 6 per cent solution of acid (10° Tw.). Very prolonged boiling with even a 3 per cent solution of sulphuric acid will, however, weaken the fibre. The most easy and definite method of determining whether the fibre has sustained serious injury is afforded by a microscopical examination which will show in this event that the outer scales have been raised up and protrude abnormally from the body of the fibre, or even in extreme cases that they have been entirely removed.

Dilute sulphuric acid has, however, a much greater action on cotton and vegetable fibres generally than on wool. If heated with acid of a strength of 2° to 4° Tw., the cotton fibre is destroyed, not by a process of decomposition, but rather by one of disintegration, the individual cells being apparently separated from each other. This, of course, entirely destroys the fibrous character of the cotton, and reduces it to the form of a fine powder, and on this action the common method of separating wool from vegetable fibres is based. This is termed the

CARBONISING PROCESS,

and has for its object the removal of any vegetable matter with which the wool is contaminated. Raw wool always contains cellulose particles, termed burrs, which become mechanically attached to the fleece during the life of the sheep. Specially troublesome are the small bean-like seeds armed with minute prickles which are frequently found in certain classes of foreign wool, notably Buenos Ayres. These burrs must be removed previous to the scribbling process, else they cause great waste by breaking the fibre. The majority of them can be mechanically removed by the so-called "burring machine," which consists of a series of drums or rollers fitted with teeth which beat out the burrs as the wool travels through. Frequently, however, even in the case of fleece wool, a chemical treatment is necessary; and invariably so in the removal of cotton from shoddy or rags.

The process consists in steeping the material in sulphuric acid of the strength indicated above until saturated, then squeezing and drying. During the drying the vegetable matter is disintegrated, and is removed in the form of dust by shaking or beating. This process is known as *liquid* or *wet carbonising*.

*Dry* or *gas carbonising* consists in treating the fibre with hydrochloric acid gas in a heated chamber, its action being the same as that of sulphuric acid.

Still another method of conducting the carbonising process has been proposed, and may conveniently be mentioned here. In this, certain metallic salts, such as magnesium or aluminium-chlorides, are used instead of acid. This process, though satisfactory on an experimental scale, is not in practical use.

Returning now to the action of various acids upon wool, *hydrochloric acid* has a similar action to sulphuric acid, *i.e.*, its effect if dilute is negligible, but when concentrated it entirely dissolves the fibre. *Nitric acid* behaves differently to sulphuric or hydrochloric acid. When heated with dilute nitric acid, wool, like all skin products, becomes permanently coloured yellow.



This action is not thoroughly understood, but the common statement that the yellow colour is due to a production of picric acid is certainly incorrect.

Colouring matters almost without exception are destroyed by a treatment with boiling dilute nitric acid, and since the wool fibre is little affected thereby, save for the production of the above-mentioned yellow colour, this process is frequently resorted to for "stripping" off the colour of dyed material previous to re-dyeing.

*Sulphurous acid* when cold and dilute has no appreciable action upon the fibre, but tends to remove the pale yellow colour which so-called white wool always naturally possesses. It is, therefore, largely employed as a bleaching agent for wool (see page 64.) Hypochlorous acid, which in the form of bleaching powder is the bleaching agent *par excellence* for cotton and vegetable fibres generally, cannot be employed for bleaching wool, because it tends to develop, rather than to remove, the yellow colour. It is found, however, that wool which has been treated with a solution of bleaching powder or hypochlorous acid possesses a greatly increased affinity for most colouring matters; and this fact, which was first noticed by Mercer, is turned to practical account in the printing of mixed wool and cotton goods, the dyeing of skin mats, etc., etc., the effect apparently being due to an oxidation of the fibre. Chlorine gas in aqueous solution acts in a similar manner to hypochlorous acid, but more energetically. The very considerable action of extremely dilute solutions of chlorine is probably due to the power which wool possesses in a marked degree of *concentrating or condensing upon itself* any re-agent with a solution of which it is brought into contact. There is no doubt that this action comes into play largely in mordanting and dyeing processes.

#### ACTION OF ALKALIES.

It has been mentioned that the action of acids upon cotton is, on the whole, much more destructive than is the case with wool. With regard to alkalies and alkaline salts the position

of the two fibres is reversed; for while cotton may be boiled with a solution of carbonate or caustic soda with impunity, such a treatment would be absolutely destructive to wool.

*Caustic alkalies* (caustic soda or potash) should never under any circumstances be used in conjunction with the wool fibre. Even when cold and dilute they exert a powerful detrimental effect. A solution containing 2 per cent of caustic soda will dissolve wool completely by boiling for a short time, cotton being unaffected thereby; and this affords a simple means of estimating the respective amount of the fibres in a mixed fabric, the process being as follows:—

#### ESTIMATION OF WOOL IN A MIXED FABRIC.

About 5 grms. of the fabric is cut off and accurately weighed; it is then dried at a temperature of 100°—110° C., until no further loss of weight ensues, and again weighed. The loss shows the amount of moisture in the fabric. The dried fabric is then boiled for five minutes in a solution of 0.5 grms. caustic soda in 200 cc. water, which will entirely dissolve the wool. The cotton is removed, washed well with water, then dried and weighed. This gives the amount of cotton present, which, added to the amount of moisture and deducted from the original weight, gives the amount of wool.

*Ammonia* has little action on wool, though in common with all alkaline solutions it has a great tendency to reduce the lustre of the fibre.

*Alkaline carbonates*, such as the carbonates of soda, potash, or ammonia, have very much less injurious action than the caustic alkalies. When used in dilute solution and at a low temperature they have indeed little effect, and are the chief agents used in removing the yolk or suint from the fibre in the scouring process. Soaps, when pure, act in a similar manner to alkaline carbonates; but care must always be taken that the scouring agents employed are free from caustic alkali, soda ash and soft soap being especially liable to contain this impurity.

## ACTION OF METALLIC SALTS.

When wool is boiled with a solution of certain metallic salts, a dissociation of the latter takes place, the metal, probably as a basic salt, combining with or being deposited upon the fibre in an insoluble condition. A portion of the acid is also taken up by the wool, whilst another portion is left in solution, partly in the form of an ammonium salt; the liberation of ammonia from the wool being probably an important factor in determining the fixation of the metal. Upon these facts the mordanting of wool depends, the fibre being boiled in a solution containing the sulphate, chloride, etc., of aluminium, tin, chromium, iron, etc., as the case may be.

## ACTION OF COLOURING MATTERS.

Unlike cotton, wool has a considerable affinity for colouring matters, readily withdrawing them from solution. It would lead us too far at this stage to speak in detail of the attraction of wool for dyes. The action appears to be in some cases a chemical, and in others a physical, effect; but this will be more conveniently discussed in treating of the various theories which have been put forward to account for the phenomena of dyeing.

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## II.—WOOL SCOURING.

The process of wool scouring, in, of course, a crude and primitive form, is of very great antiquity, since in the earliest days of the use of wool or animal skins as a covering for the human body, the desirability of removing the naturally contained grease would at once become apparent. Originally a simple washing in water would no doubt be resorted to, but this process, as will be shown directly, would remove only a portion of the greasy matter. Later, some sand, clay, or ashes was used during the washing, which, by mechanically rubbing off and absorbing the grease and dirt, would improve the effect.

The efficacy of wood ashes in removing the grease and cleansing the wool, would then, doubtless, be soon noticed by our observant progenitors, and it is a moot point whether this or stale urine was the first form of alkali to be employed for the purpose. At a very early period the common soap-wort (*saponaria officinalis*) and the Egyptian soap-wort were also employed; these two plants being referred to by Dioscorides and Pliny as being used for cleansing wool in their time. At the present day the soap-nut (*sapindus mukorossi*) and soap-bark (*quillaya saponaria*) are used to some extent, the former principally in India, and the latter in South America. The employment of soap and soda-ash, as scouring agents, is also of considerable antiquity.

## OBJECT OF THE WOOL-SCOURING PROCESS.

In treating of the structure of the wool fibre, and again when discussing its chemical composition, reference has been made to the greasy matter secreted by the sebaceous glands and exuded upon the fibre during its growth. This is termed the *yolk* or *suint*, and is of great service during the growth of the fleece in preventing the mechanical injury to the fibres which would arise if they became matted together. The suppleness and general suitability of wool for textile purposes are probably largely due to the protective action of the yolk. The presence of the greasy matter upon the shorn fleece also serves as a complete protection against the depredations of moths; the fact that moths will not attack unwashed wool, and that any material may indeed be protected from moths by contact with greasy wool, being first published by Reaumer in 1738. There is a certain small proportion of oily matter in wool fibre, amounting perhaps to about 1 per cent of its weight, which appears to have a different function to that of the yolk, with some constituent of which it may or may not be identical in composition. This may be termed the *lubricant* of the fibre, since if it is removed the fibre becomes harsh to the feel, brittle, and less tenacious. The greasy matter constituting the

yolk may be termed the *preservative* agent, and only after its removal does the fibre show its valuable properties to the fullest extent.

In addition to the yolk, raw wool always contains dirt and earthy matter, sometimes to the extent of 20 to 25 per cent of its weight.

Scoured wool must always be oiled before scribbling or combing, various vegetable or sometimes mineral oils being used for the purpose. This, along with any dirt which the fibre may have acquired in the various stages of manufacture, must, of course, be removed from the yarn or cloth.

The object of the scouring process may therefore be defined as that of completely removing from raw wool the yolk or preservative greasy matter, along with all dirt, etc.; or from yarn or cloth all oily matter and dirt which may be present. In no case, however, should the small amount of lubricating oily matter be extracted.

It is, of course, of very great importance that while care is taken not to injure the fibre, the scouring process should be thorough, since any grease left in the wool is likely to cause irregularity of shade in dyeing and other defects in the finished material.

#### COMPOSITION OF WOOL YOLK.

Vauquelin, and also Chevreul, who first investigated the character of the impurities upon raw wool, confined the term *yolk* to that portion which is soluble in water; but it is now commonly used to designate the whole of the encrusting mass of grease, adhering earthy matter, etc. Accepting this definition, the yolk is a somewhat complex mixture of substances, but for consideration in relation to the scouring process they may be classified into (a) that portion which is soluble in water, which has been termed *wool perspiration*; (b) the *wool fat* or portion insoluble in water; (c) earthy matter and other dirt. A short consideration of these substances will explain the theory of the scouring process.

(a) *Wool Perspiration*.—This consists essentially of potash salts or soaps of oleic, stearic, hyenic, and other fatty acids, principally the two former. It also contains small amounts of potassium acetate, valeriate, and salts of other lower members of the fatty acid series; small quantities of potassium chloride and sulphate, as well as about 0·5 per cent of ammonia, and traces of various metals are also present. The composition of the inorganic constituents of yolk is shown in the following analysis of yolk ash by Maumené and Rogelet:—

Analysis of Yolk Ash.	Per cent.
Potassium carbonate .....	86·78
„ sulphate .....	6·18
„ chloride .....	2·83
Silica, Phosphorus, Lime, Iron, Alumina, &c.....	4·21
	100·00

Dried yolk contains about 60 per cent of organic and 40 per cent mineral matters, the latter consisting principally—in fact, almost entirely—of potash salts, as shown above. Yolk, therefore, is a very valuable source of potash salts, which are of considerable commercial value.

In England it is not usual to distinguish between the wool-fat and wool-perspiration during the scouring, the whole being removed at one operation and the potash salts being entirely lost; in Belgium and France, however, the wool is submitted to a steeping process previous to the scouring process. This is termed “wool-steeping,” and will be described later. That portion of the yolk which is soluble in water is insoluble in benzene and other volatile solvents.

(b) *Wool Fat*.—This, the portion of the yolk which is insoluble in water, may be separated into two parts by a treatment with boiling alcohol. The portion which dissolves in this re-agent consists of a neutral, unsaponifiable fat-like body, *cholesterol*, along with *ischolesterol*, and their ethers—these existing both in the free state and in combination with formic and acetic acids. The larger portion of the wool-fat is insoluble in alcohol, and this part is composed of cholesterol and ischo-



lesterol, combined with the higher fatty acids. Both portions are soluble in ether, benzene, carbon bisulphide, etc., and form an emulsion with alkaline detergents.

*Cholesterol* has the composition  $C_{26}H_{44}O$ , and partakes both of the nature of an alcohol and a fat. It is not a glyceride, but in its combinations with the fatty acids appears rather to form compounds analogous to the glycerides. Olive oil, for instance, is a combination of oleic acid with *glycerol*, while the neutral insoluble fatty or wax-like portion of the wool fat may be an oleic acid compound of *cholesterol*.

Cholesterol exists in both vegetable and animal products. It is, for instance, found in an alcoholic extract of beetroot juice which has gelatinised, and also exists in human skin, hair, feathers of birds, etc. It is a principal constituent of gall stones, which were until recently its principal source. It has long been used medicinally, the "oesypus" mentioned by the Greek physician, Dioscorides, who lived 100 A.D., being essentially cholesterol. It was at this time prepared by extracting newly-shorn wool with hot water, treating the liquor with soap-wort to dissolve out all soluble matters and remove dirt, then adding sea water and collecting the fat which rises to the surface. It was afterwards purified by macerating with hot water, kneading with the fingers, etc. "Oesypus" was employed as a healing salve, and remained in use until the end of the seventeenth century, when it was dropped from the pharmacopœia along with many other antique remedies, such as burnt swallows, frog's lungs, etc.\*

Cholesterol is, however, now prepared on a large scale, and finds an extensive and rapidly increasing use for medicinal purposes, for which it will probably entirely supersede vaseline, paraffin, and lard. Its special value lies in the ease with which it is absorbed by the skin, any drugs with which it is associated being thus rendered much more active, whereas their action is considerably retarded if vaseline, paraffin, or lard is the medium used for their application.

\* Langbeck. *Jour. Soc. Chem. Ind.*, April, 1890.

To prepare the cholesterol the wool-yolk is heated with an alcoholic solution of caustic potash, and the clear solution partially evaporated. Water and ether are then added, and after agitating and allowing to settle, two layers are formed, the upper ethereal layer containing the cholesterol. To purify it, it is recrystallised from a mixture of ether and alcohol.

When pure, cholesterol crystallises in white monoclinic tablets. It is insoluble in water, but soluble in alcohol, ether, and volatile solvents generally, or in fatty acids. Its specific gravity is 1.067, and melting point =  $145^{\circ}C$ . It possesses the peculiar property of absorbing or mixing with about 100 per cent of its weight of water to form a solid emulsion, and it is this mixture which is now so well known under the name of "Lanolin," and which forms the basis of "Lanolin Salve," "Lanolin Pomade," etc. The so-called "Lanolin Soap" is an ordinary soap, with an admixture of lanolin, the latter being of itself incapable of saponifying. "Cholesterinol" and "Lanesine" are similar products.

Cholesterol, in an impure condition, is used in America for softening leather, and as a lubricant for horses' hoofs, etc., being specially suitable for these purposes, on account of the avidity with which it is absorbed by animal tissues. Chiefly on this account the value of the grease recovered from wool scouring operations has rapidly risen in that country.

(c) *Earthy Matter and other Dirt*.—The amount and character of this portion of the yolk is, of course, very variable, depending upon the nature of the pasturage, etc. Sometimes it constitutes as little as three or four per cent, but frequently reaches as much as 30 to 40 per cent of the total weight of yolk. It adheres mechanically to the fibre by virtue of the sticky nature of the greasy matter, and is liberated and got rid of by the removal of this during the scouring process. Its nature is therefore of no importance as regards our present purpose.

## THEORY OF WOOL-SCOURING.

From what has been stated above with regard to the character of the substances which have to be removed, the theory of the two distinct methods of wool scouring will readily be understood. To summarise briefly, the following substances may be present:—(1) Wool perspiration consisting of the potash salts of fatty acids. Soluble in water; insoluble in volatile solvents. (2) Wool fat (cholesterol, etc.). Insoluble in water, but soluble in volatile solvents, and forming an emulsion in alkaline solution. (3) Vegetable oil added during the scribbling, combing, spinning, etc. Readily emulsified by alkali, or dissolved by volatile solvents. (4) Mineral oil used in spinning, etc. Soluble in volatile solvents, *but emulsified only, and then with difficulty, in the presence of some saponifiable oil.*

There are two entirely distinct processes which may be adopted for wool scouring, viz.:—The emulsion process and the solution process.

The former is the one commonly employed at the present time, and depends for its efficacy upon the properties possessed by certain bodies, especially alkalis, of so acting upon oils and fatty matters that they are mechanically divided into very minute particles, which do not again easily coalesce. In this condition the oil forms a milky liquid, and is readily removed from the wool. In the emulsion process a solution of carbonate of soda or other alkali is, therefore, employed, the wool perspiration being thereby dissolved and the wool fat and added oil emulsified.

The solution process, perhaps better known as "scouring by means of volatile liquids," is theoretically much simpler and more economical than the ordinary process, and will probably come into general use in the future. At present, however, the mechanical difficulties of applying the large volumes of volatile and usually explosive liquids necessary, have not been adequately surmounted.

In the solution process the wool fat, free oil, etc., is dissolved by a treatment with benzene, light petroleum oil, or other suitable solvent, the potash salts being subsequently removed by hot water. The volatile solvent is then recovered by distillation and used over again.

If this process could be conducted in an absolutely theoretical manner, its cost would probably be *nil*, the only expense being labour and a little steam, which would be more than counterbalanced by the value of the residuals obtained. The residue from the redistillation of the solvent is the proper raw material for the manufacture of lanolin, and the valuable potash salts could be readily obtained from the aqueous solution of the wool-perspiration.

## MANUFACTURE OF POTASH SALTS, ETC., FROM YOLK.

In discussing the composition of the substances which it is desired to remove from the fibre during the scouring process, it was mentioned that the soluble portion of the yolk of wool (wool perspiration) consists almost entirely of the potash salts of certain fatty acids. It may, therefore, well serve as a raw material for the manufacture of prussiate of potash or potassium carbonate; and, in fact, the production of these salts in this manner is a valuable industry in France and Belgium. To this end the wool is treated with hot water previous to the scouring proper, the process, which is termed "wool steeping," being conducted in the following manner: A series of large wooden or iron tanks are filled with raw wool, and hot water is then admitted to the first, where it is allowed to remain for some time. The liquor is then run by gravity or pumped into the second tank where it dissolves more yolk; then into the third tank, and so on until a saturated solution of yolk is obtained. Meanwhile clean hot water is run into the first tank, this second liquor passing along in the same way; the process of filling tank No. 1 with clean water being repeated until the whole of the yolk is extracted. The clean wool is then removed and the tank, refilled with raw wool, becomes

the last of the series; each tank of the battery thus becoming in turn the first and last of the series. By working in this systematic manner the resulting yolk solution obtained is always quite saturated, and the cost of the subsequent evaporation reduced to a minimum.

Fig. 9 illustrates one form of wool-steeping apparatus, the construction and mode of working of which will be readily understood. After filling the tanks with raw wool hot water is supplied from the horizontal pipe. The tanks are emptied

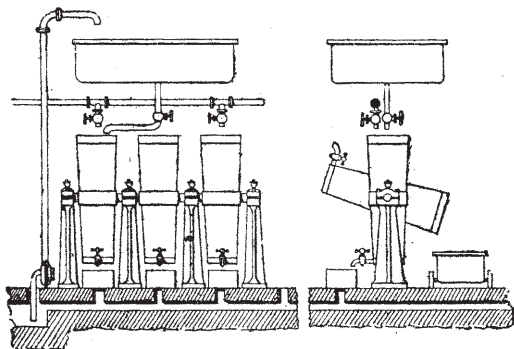


FIG. 9.—APPARATUS FOR WOOL STEEPING.

by means of the cock at the bottom into the channel, from whence the liquor is pumped up into the upper reservoir and delivered by gravity into the next tank of the series. The method of removing the wool is also indicated.

The subsequent treatment of the yolk solution thus obtained varies according to the use for which it is intended. If neutralised with phosphoric acid and evaporated to dryness, a mass of impure potassium phosphate mixed with fatty acids, dirt, etc., is obtained, which forms a valuable manure. The dried residue is also a natural raw material for the manufacture of yellow prussiate of potash, for since it contains potassium, carbon, and nitrogen, it simply requires to be fused with iron filings, the melt extracted with water, and the solution crystallised; while if pure potash salt is desired the

dried residue is calcined in a retort, the crude carbonate of potash then removed by lixiviation of the ash, and recrystallised. By careful crystallisation almost pure  $K_2CO_3$  is obtained, the small amounts of the more soluble chloride and sulphate being left in solution.

During the calcination of the dry yolk a large quantity of gas is evolved. This has considerable illuminating power, and may be used for lighting the works wherein the process is conducted, but more frequently it is burnt and the heat utilised for evaporating the yolk solution.

It is stated that 1,000lbs. of raw wool yield from 70lbs. to 90lbs. of  $K_2CO_3$  and 5lbs. or 6lbs. of  $KCl$  and  $K_2SO_4$ , the value of the potash salts thus obtained probably exceeding £100,000 annually in France alone.

In England this preliminary steeping process is usually omitted, the whole of the yolk being removed during the scouring process itself. It is, however, customary to wash the fleece on the back of the sheep in running water just previous to shearing, and this will have the effect of removing at any rate a portion of the wool perspiration. By this process the valuable potash salts are of course entirely lost.

However, whether the wool is submitted to a preliminary steeping or not, a subsequent scouring is necessary, and having explained the theory of the two distinct methods of scouring in vogue, the reagents used and the processes themselves will now be described.

#### SCOURING AGENTS.

Referring first to those used in the ordinary or *emulsion* process, the following are the alkalies chiefly employed:—Carbonate of soda, soap, ammonia, ammonium carbonate. Sodium silicate and potassium carbonate are also used to a small extent.

*Carbonate of soda* is sold in several different forms. *Soda ash* is an impure carbonate containing 70 to 95 per cent  $Na_2CO_3$ . Amongst the impurities in this substance caustic soda is of frequent occurrence, and the injurious effect upon wool of even



very small amounts of caustic alkali has already been insisted upon. It is therefore very necessary to have a simple means of detecting the presence of caustic soda in carbonate, and the following test does not require much manipulative skill or complicated apparatus for its application.

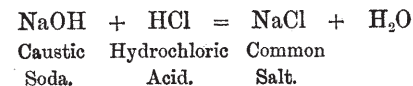
*Test for caustic soda in soda ash.*—Dissolve 5—10grs. (say  $\frac{1}{4}$ oz.) in pure water, and add to the solution an excess of a strong solution of barium chloride. This will precipitate all the carbonate present as barium carbonate, and this being removed by filtration the clear liquid will contain only caustic soda, if present in the original substance, along with sodium chloride, and the excess of barium chloride; and the two latter being neutral salts, caustic alkali is rendered evident by adding some suitable indicator, *e.g.* phenol phthalein, to the solution.

The test, put as concisely as possible, consists in dissolving the soda ash in water, adding excess of barium chloride, filtering, and adding phenol phthalein; when a pink colour is immediately developed if caustic soda is present, but the solution remains quite colourless in its absence.

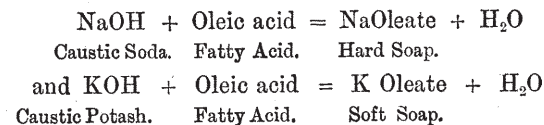
There are now, however, several forms of carbonate of soda in the market, which while guaranteed to be absolutely free from caustic alkali, are only slightly more costly than the much more impure soda ash. Such are the so-called "pure alkali," which is a practically pure and anhydrous carbonate, containing 98 to 99 per cent  $\text{Na}_2\text{CO}_3$ , "crystal carbonate," which is a beautiful preparation of the composition  $\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O}$ , and "sesqui-carbonate," which is represented by the formula  $\text{Na}_2\text{CO}_3\cdot\text{NaHCO}_3$ . Any of these products can be highly recommended. The ordinary preparation, known as "soda crystals," or "washing soda," has the composition  $\text{Na}_2\text{CO}_3\cdot 10\text{H}_2\text{O}$ , and thus contains no less than 63 per cent of its weight of water.

*Soaps*, both hard and soft, are used as scouring agents. The composition of soap has already been incidentally alluded to, but may be again explained in a few words. Any combina-

tion of an acid with a base is termed a salt; thus hydrochloric acid united with sodium produces common salt, which may be prepared according to the following equation:—



Soaps are salts containing *fatty acids* (oleic, stearic, etc.); "hard" soaps resulting when *sodium* is the base, while *potassium* gives rise to "soft" soaps, thus:—



Any soap, when unskilfully made, is liable to contain caustic alkali, but potash soaps are, on account of the method adopted in their manufacture, particularly liable to this defect. It is therefore a great mistake to suppose that soft soap is a less severe scouring agent than hard soap, the exact opposite being usually the case.

The analysis of soaps will be dealt with later, but a very simple test sufficing to show whether uncombined alkali is present may be here introduced: Pour on to the soap a small quantity of an alcoholic solution of phenol phthalein, when the production of a red colour indicates that free alkali exists.

Fatty acids are capable of combining not only with sodium and potassium, but with other metallic bases, such as calcium, magnesium, iron, etc., and the compounds thus formed being insoluble in water and of a sticky nature, are very difficult to remove from wool to which they have once become attached. On this account it is very undesirable that soap should be used as a scouring agent when the water is at all hard, *i.e.*, contains much lime or magnesium, since if these are present a decomposition of the soap ensues with production of the above mentioned insoluble soaps.

*Ammonia* is used as a mild scouring agent. The ammonia liquor of commerce of 0.88 sp. gr. contains about 33 per cent of ammonia, the remainder is water.

*Ammonium carbonate* is not usually employed as such, but in the form of stale urine or "lant," of which it constitutes the active agent. The use of this substance appears to be gradually dying out.

#### THE PROCESS.

For fine quality wools a well made soap, quite free from caustic alkali, with or without the addition of ammonia, may be considered the most suitable scouring agent. When a somewhat more severe treatment is desirable, soap and carbonate of soda, or even the latter alone may be employed; but when dealing with yarn or cloth to which oil has been added to facilitate spinning, the character of the oil used must be taken into account in selecting the scouring agent.

A fundamental rule in scouring should always be to use as little alkali and conduct the process at as low a temperature as is consistent with a thorough cleansing of the wool, since under the most favourable conditions the lustre of the wool is apt to suffer considerable diminution. On the other hand, it is very essential to remove from the wool not only all dirt, but any greasy or oily matter which might otherwise cause trouble in the dyeing process. It is, however, unfortunately the case at present in many woollen mills that the scouring process receives very little supervision, with the result that it is conducted in a very unsystematic and irregular manner, and with lamentable results.

*Loose wool scouring.*—In this country the preliminary steeping process described in the last portion of this article is usually omitted, and therefore the whole of the natural grease and adhering dirt have to be removed during the scouring process proper. A certain advantage may decidedly be claimed for this method of conducting the process in that the potash salts contained in the yolk assist in the emulsification of the neutra

fats, and therefore a smaller amount of added alkali is needed. This, however, is more than counterbalanced, apart from the loss of the valuable potash salts, by the fact that the scouring baths quickly become too dirty for use.

It has been previously mentioned that the finer qualities of wool usually contain the largest amount of yolk. It is, therefore, a mistake to use the most severe scouring agent for the dirtiest wool, since the better qualities of wool are the most easily injured. The choice of alkali should be influenced by the character of the fibre, and not its condition as regards yolk and dirt.

For the best qualities of wool a good neutral soap with the addition of a little ammonia is usually preferred. The soap, however, must be absolutely free from caustic alkali, and the ammonia should not contain aniline or pyridine; these bases, which may be the cause of the development of grey stains, being frequently found in ammonia distilled direct from gas liquors. The amount of soap employed should be from 3lbs. to 5lbs. per 100 gallons of water, but part of this may be substituted by ammonia, or the latter used in addition. The temperature of the solution should not exceed 40° to 50° C. (= 100° to 120° F.); this remark applying in the case of all scouring solutions of whatever nature.

For coarser qualities of wool one of the many forms of carbonate of soda may well be employed; this being, indeed, the most usual scouring agent for loose wool. The strength of solution should be from 1° to 2° Tw.—say 10 to 20lbs.  $\text{Na}_2\text{CO}_3$  per 100 gallons of water. Frequently a mixture of soap and sodium carbonate is used.

With regard to the manipulation of the process for emulsifying the fatty matters, the essential conditions are that a certain degree of agitation either of the material or the alkaline solution should take place. This may be brought about either by moving the material in the liquor, or by keeping the wool in a fixed position and circulating the liquor, with frequent squeezings or pressings of the liquor-saturated fibre.

In the older, and still usual form of machine, the first-named conditions obtain ; and Fig: 10 is an illustration of MacNaught's rake machine, which may serve as a type of this class.

The machine consists of a long trough, in which the scouring liquor is placed. At one end is an endless band or feeding apron, and at the other a pair of squeezing rollers. The trough is fitted internally with a series of transversely situated forks which are supported upon a framework capable, by means of suitable mechanism, of being moved horizontally forward, raised clear of the trough, moved backward, and again dropped into the trough. The same framework supports a perforated plate which serves to immerse the wool, and a sort of fork-scoop to transfer the wool from the trough to the squeezing rollers.

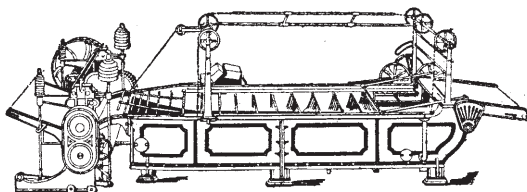


FIG. 10.—WOOL SCOURING MACHINE, WITH RAKES.

The greasy wool is spread more or less evenly upon the travelling feeding apron and is immersed in the liquor by the action of the perforated plate. The fibre is then caused to slowly traverse the trough by the intermittent action of the forks, and, arriving at the delivery end, passes between the squeezing rollers and is thrown off by the flyer.

A single passage through such a machine is usually not sufficient to thoroughly cleanse the wool, since after being in use for a short time the liquor becomes very dirty. It is a frequent practice, therefore, to have two or three machines through each of which the wool passes. The second trough is filled with clean scouring liquor, and the third with water. When the liquor in the first trough becomes saturated with yolk or too dirty for use, it is run out and the trough filled with

that from machine No. 2 ; the latter being supplied with clean liquor. The troughs are fitted with perforated false bottoms, underneath which most of the dirt collects.

All machines for the treatment of loose wool, either in scouring or dyeing, which depend upon movement of the material and not upon the circulation of the liquor, have the inherent defect that the fibre may readily be felted or matted together if the slightest want of care or skill occurs during the process. The liability of this troublesome and annoying action taking place is much reduced in those machines in which the wool remains stationary and the liquor circulates. This ideal condition has not been found practicable in scouring, although it is attained in dyeing processes ; but certain of the newer forms

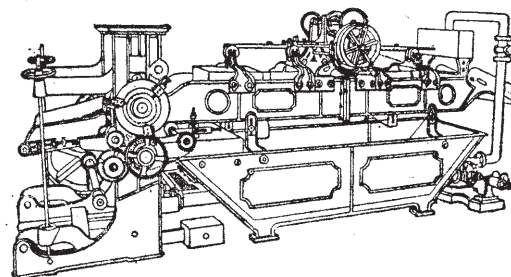


FIG. 11.—WOOL SCOURING MACHINE, CONTINUOUS CURRENT.

of scouring machine go so far in this direction that there is no movement of the wool relatively to the liquor, since forks and other appliances are dispensed with, and the circulation of the liquor itself carries the fibre through the machine.

In the apparatus of this type, illustrated by Figs. 11 and 12, the upper (smaller) trough contains the scouring liquor, the lower cistern simply acting as a storage tank. The greasy wool is fed into the machine by means of the endless band, and is immediately permeated by the liquor, which falls in a shower from the small tank seen in the upper right hand corner of the illustration. It then passes along the scouring trough, being squeezed during its passage by the four oscillating



perforated plungers shown clearly in Fig. 12. On reaching the delivery end of the machine the wool receives a final squeeze between rollers, and the liquor falls into the lower cistern to be again transferred by the pump to the upper, or scouring trough, by way of the small showering or distributing tank. It is claimed that machines of this type, of which there are many modifications, cause less felting of the wool, and are more efficient than the older class of machine.

The use of hard waters for scouring purposes is always very detrimental, but this is specially the case when soap is employed, either alone or with an alkali. The injurious effect arises from the formation of sticky and insoluble lime and magnesium soaps, which adhere to the fibre, and give rise

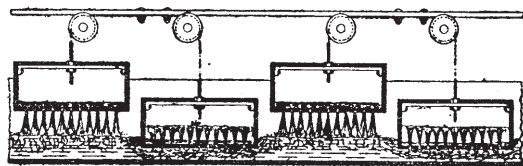


FIG. 12.—DETAILS OF MACHINE IN FIG. 11.

subsequently to defects; either causing dirty spots to appear upon the finished goods if white, or serving to attract or repel the colour during the dyeing process, thus producing unevenly coloured goods. This is probably one principal reason why old scouring baths act more efficiently than freshly prepared solutions, as the lime, etc., in the water will have been precipitated when first used. Another cause for the better action of old baths is probably to be found in the fact that being in a state of slight fermentation the liquor more readily emulsifies the fatty matters.

#### RECOVERY OF GREASE FROM SCOURING BATHS.

Whatever scouring agent is used, the dirty liquors from the process contain much fatty matter, and the amount is largely increased if soap has been employed. These waste liquors cause great pollution of the water if run into a river or stream,

since the organic matter decomposes and putrefies, giving rise to very objectionable effluvia. Moreover, the value of the residual products will well repay for their extraction, and, therefore, for these combined reasons, it is highly desirable to treat all scouring liquors before running to waste.

One process in operation in some Continental works is to precipitate the fatty matter by milk of lime. The lime soap obtained, which contains 70 per cent of its weight of fatty acids, is dried; and after mixing with five times its weight of coal, is used for the manufacture of illuminating gas. It is stated that it yields five or six times the illuminating power of an equal weight of coal, and, used in this manner, yields gas of about one-third the value of the soap employed.

*The "Magma" Process.*—In England the usual process for the recovery of the soap consists in separating the fatty matters by means of acid. The waste liquors are run into a stone tank, and sufficient sulphuric acid added to throw all the grease to the surface. After standing a sufficient length of time for complete separation, the magma of fatty matter is removed from the surface and drained in filter bags. It may then be directly transformed into soap by heating with the requisite amount of caustic soda, but the resulting product is of very inferior quality, and this process cannot be recommended. A better plan is to dispose of the magma to a soap manufacturer, who, by distilling it in a current of steam, obtains the fatty acids in a much purer condition, a residue of heavy lubricating oil remaining in the retort. The purified fatty acids make a good quality of soap.

Even as fuel for use in a boiler fire the magma of fatty matters, after draining by lying in a heap, is valuable enough to pay for the slight labour and cost of its production.

#### SCOURING BY MEANS OF VOLATILE LIQUIDS.

The process of scouring wool by means of volatile liquids, or, as it may be termed, the "solution" process, differs entirely, both in theory and manipulation, from the "emulsion"

process at present in vogue. As already stated, it is theoretically much simpler and more economical than the ordinary process, and will probably come into general use when the mechanical difficulties of dealing with the considerable volume of highly volatile and explosive liquids have been adequately surmounted.

As already stated, the yolk of wool consists, broadly speaking, of *free fats*, insoluble in water but soluble in volatile solvents; *combined fats*, or potash salts of fatty acids, soluble in water but insoluble in volatile solvents; *sand, dirt, &c.* Since the volatile solvents are usually not miscible with water, two operations are essential: a treatment with the former to remove the fats, and a treatment with water to dissolve out the potash salts. The latter process has already been described under the head of "wool-steeping," but instead of taking place previous to the scouring, the steeping process should come last. This is necessary, because otherwise the wool must be dried after steeping, which is apt to permanently discolour it by modifying the fatty matters present.

A considerable number of volatile solvents have been proposed for use in this process, viz., *carbon disulphide, benzene, toluene, petroleum benzene, amyl alcohol, alcohol, and ether.* Of these, however, the first four only can be considered as at all practicable in this country on account of the high price of alcohol and ether.

*Carbon Disulphide*,  $CS_2$ , has been the liquid employed in many of the proposed methods. It is a colourless, transparent liquid, somewhat heavier than water, and is very volatile, emitting vapour at the ordinary temperature and boiling at  $43^\circ C.$  ( $= 110^\circ F.$ ). The vapour is heavy, and therefore is not easily drawn off by a current of air, and when mixed with air is explosive if ignited. The commercial article has a very unpleasant smell, which arises from the presence of certain impurities. Carbon disulphide is a good solvent for fatty matters, and has no action upon wool if applied cold, but if heated tends to discolour the fibre, giving it an objectionable yellow appearance.

*Benzene*,  $C_6H_6$ , is a colourless volatile liquid obtained during the distillation of coal-tar. It boils at  $177^\circ F.$ , the vapour being very inflammable, burning with a bright flame and depositing much soot. It has a peculiar odour, which is not unpleasant.

*Toluene*,  $C_7H_8$ , is very similar in appearance and properties, but has a considerably higher boiling point ( $230^\circ F.$ ). Commercial "benzol," which is a variable mixture of benzene and toluene, has a density of 0.88 to 0.90 compared with water as 1, and will, therefore, float on the surface of water. It is an excellent solvent for fatty matters, and quite without action on wool fibre.

*Petroleum Benzene* or "*Benzine*" is obtained by the distillation of crude petroleum, and usually consists of the fraction which passes over between  $100^\circ$  and  $125^\circ C.$  It forms a colourless volatile liquid which, like coal-tar benzene, will not mix with water. This product is considerably cheaper than coal-tar benzene, but unless very highly purified, has the defect of leaving the wool with a somewhat unpleasant smell.

*Amyl Alcohol*,  $C_5H_{11}OH$ , is a colourless liquid, having a penetrating odour, and is obtained by the fractional distillation of fusel oil. It boils at  $113^\circ C.$  ( $= 236^\circ F.$ ).

*Alcohol*, or ethyl alcohol,  $C_2H_5OH$ , occurs largely in commerce in an impure form, under the name of *methylated spirit*, which consists of about 90 per cent ethyl alcohol and 10 per cent methyl alcohol or wood spirit, with a little shellac. The object of the admixture is to make an undrinkable spirit, since, in this form, it is free of excise duty. Methylated spirit boils at about  $85^\circ C.$  ( $= 185^\circ F.$ ), burns with a non-luminous flame, and when mixed with air its vapour is explosive. Alcohol may be mixed with water in all proportions.

*Ether* has the composition  $(C_2H_5)_2O$ . It is an extremely volatile and inflammable liquid, boiling at  $40^\circ C.$  ( $= 104^\circ F.$ ). Its specific gravity is only 0.74, and it is therefore considerably lighter than water. The solvent power of ether for fats is very great.

*Carbon Tetrachloride*,  $\text{CCl}_4$ , is probably the reagent which will in the future be employed; it is at present too expensive for use. It is a heavy, pleasant smelling liquid, boiling at  $77^\circ$ , and is *uninflammable*.

The whole difficulty of applying this solution process of wool scouring in a practicable manner is due to the fact that the solvents employed are very volatile, and their vapour, when mixed with air, usually explosive in contact with a light. The liquids also are comparatively expensive, and therefore any considerable loss makes the process a costly one. The problem of how to apply the large volume of volatile liquid required with a minimum of loss and with sufficient economy is by no means of easy solution, as the many inventors who have turned their attention to the subject have found. A large number of different forms of apparatus have been proposed, and some have been tried on a large scale; but at the present time the process is nowhere in practical use, and an entirely successful apparatus has yet to be devised.

It is not difficult to point out the essential features which a successful apparatus must possess. In the first place, the process must thoroughly remove the wool-fat, but must be such that the fibre is not in the slightest degree discoloured or injured in any way. Then, in order to deal with large quantities of wool, the process must be continuous or nearly so; and thirdly, the cost of the process must be as low as possible, and the danger of fire and the emission of poisonous vapours reduced to a minimum.

In order to satisfy the third condition, it is obvious that the amount of solvent used should be as small as is consistent with a thorough cleansing of the wool, and this again implies that before redistillation the solvent must be thoroughly saturated with the dissolved wool-fat. It is, moreover, evident that a solvent partially saturated with fat will never remove the last traces of fat from the wool, and, therefore, in the last stages of the process the wool must be treated with clean solvent in order to satisfy the first condition named. We may

thus state that as a working condition it is essential that the wool should have a cumulative action in saturating the solvent, and this is attained by allowing fresh solvent to act upon the nearly clean wool and fresh greasy wool to come into contact with solvent which is almost saturated. Obviously, the simplest manner to accomplish this is to admit clean solvent at one end of the apparatus and greasy wool at the other extremity, and allow both to travel progressively forward at proper speed, in opposite directions. No apparatus will be commercially successful that does not embody this principle in one form or another.

On account of the nature of the solvents employed it is extremely difficult to reduce the percentage of loss of solvent to a workable figure. This is, indeed, the rock on which the barks of most of the recent inventors have foundered. Evaporation takes place wherever the solvent is exposed to the atmosphere, and it has been found necessary in many cases to entirely enclose the apparatus and draw off all vapours by means of a fan, through a condensing arrangement, in order to minimise such loss. Even this, however, is only partially effective, because the wool itself retains a small amount of solvent with considerable tenacity, especially in the case of carbon disulphide.

Of the twenty to thirty arrangements which have been proposed, it will suffice to describe one or two typical forms. The earliest was that devised by E. Deiss, in 1856, and is the one illustrated in Fig. 13. It consists of four main parts (A, B, C, D), suitably connected by pipes. A is a stone tank, in which the solvent—carbon disulphide—is stored, a layer of water covering it and preventing evaporation. The extracting vessel B is fitted top and bottom with perforated plates, between which the wool is placed. D is a still, heated by means of a steam coil, and connected, as is also the extracting vessel B, with a worm-condenser C.

When in use the extracting vessel B is packed with greasy wool, and  $\text{CS}_2$  forced by means of a pump (not shown in the



figure) up the pipe *a* and through the wool, any vapour passing through *c* and being condensed in C. The CS<sub>2</sub> with wool-fat in solution, then flows out of the top of B down the pipe *b*

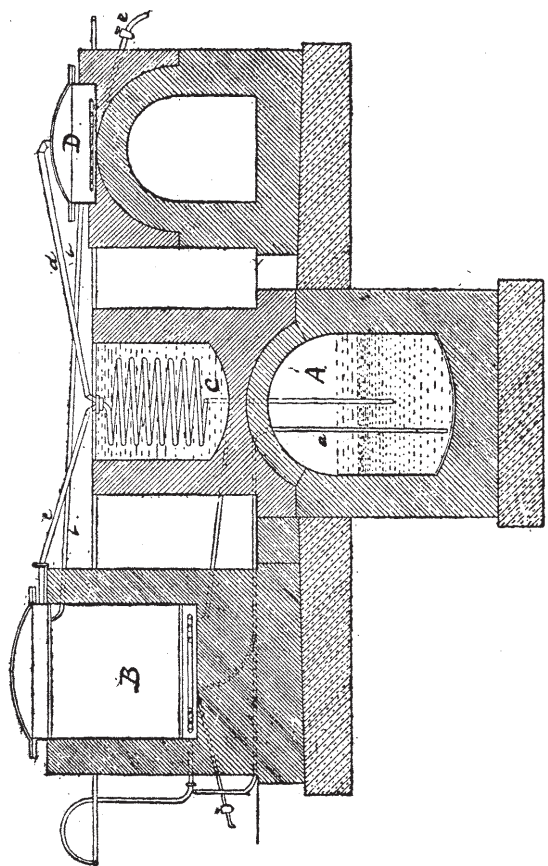


FIG. 13.—APPARATUS FOR SCOURING BY MEANS OF VOLATILE LIQUIDS.

into D, where it is distilled, the volatile solvent passing through *d*, and after being condensed in C is returned to the store tank A to be used again. The melted wool-fat is removed at intervals by the cock *e*. The circulation of the

CS<sub>2</sub> is continued until the wool is entirely freed from wool-fat, at which time the solvent remaining in B is driven off by means of a steam coil, and the wool removed.

Several extracting vessels B may be connected with a common CS<sub>2</sub> tank, still, and condenser; and thus the process made continuous by recharging them in rotation.

One great defect of Deiss' process is that the CS<sub>2</sub>, especially in the later stages of the extraction, is, when redistilled, far from saturated with wool-fat; much more solvent has, therefore, to be employed than is actually necessary, and the cost of redistillation and the inevitable loss by leakage is proportionately increased. Probably, however, this defect might be easily remedied by so arranging the apparatus that the CS<sub>2</sub> passed successively through a battery of extracting vessels, each of which in turn became the last when recharged with greasy wool, and the first when, the wool being freed from wool-fat, it is ready for removal. A cumulative effect might thus be attained.

Another defect arises from the fact that whenever wool is treated with heated CS<sub>2</sub>, as is the case when removing the last portion of the solvent by means of the steam coil, the fibre acquires a permanent yellow colour; but it appears that this defect also could be readily removed by a slight rearrangement of the apparatus.

Deiss' apparatus never attained a practical success for wool scouring, but was largely employed for the extraction of oil from seeds.

Passing over a large number of different forms of apparatus, in which the above-named defects were more or less successfully surmounted, the apparatus of T. J. Mullings (1879) will be shortly described, since this was probably the first arrangement which was adequately tested on a large scale. It differs in form from any which either preceded it or have been proposed since: consisting essentially of a specially arranged centrifugal machine, provided with a movable cover resting in a water seal. Internally it is fitted with removable iron cages, into

which the wool is packed. After filling the machine and fitting on the cover,  $\text{CS}_2$  is admitted to the bottom and flows out from a pipe near the top of the apparatus, being thence conveyed to a distilling vessel. The only point in which it radically differs from Deiss' apparatus is in the method of eliminating the  $\text{CS}_2$  from the wool when the process is complete, which is done by revolving the apparatus in the usual manner and then admitting water to displace the last traces of solvent: this in turn being removed by the centrifugal action.

One of the defects in the apparatus of Deiss, viz., the yellowing of the wool, is therefore absent in the Mullings form of machine, but unless a number of the centrifugal machines—which are costly—were connected up to form a battery through which the  $\text{CS}_2$  can circulate, the want of a cumulative effect still exists.

The two forms of apparatus which have most nearly fulfilled the necessary conditions are those of Singer and Judell (1887) and Burnell (1888), and it is quite possible that one or other of these may form the basis of a practical solution of the problem. Both machines show evidence of much thought and ingenuity, and the way in which the details are worked out is in both cases most praiseworthy. It is impossible to convey an idea of the construction of the apparatus without a long description, but in both cases the plan of cumulative solution is adopted. Messrs. Singer and Judell proposed to use carbon disulphide as solvent, while in the Burnell apparatus petroleum benzene was the solvent employed.\*

#### YARN SCOURING.

During the scouring process the raw wool is deprived of its natural lubricating greasy matter, and in the purified condition is more easily susceptible to mechanical injury if roughly treated. If, for instance, it were attempted to card

\* For further details respecting Singer and Judell's apparatus refer to the *Journal of the Society of Chemical Industry*, January, 1889. Burnell's process is described in the *Journal of the Society of Dyers and Colourists*, January, 1895.

and spin the fibre in this dry condition, not only would the resulting thread lack uniformity and cohesion, but much waste would be produced through breakage of the fibre. In order to facilitate these processes, it is, therefore, necessary to add a certain amount of oil to the wool, and if it is desired to dye it in the form of yarn, this added grease, along with any dirt acquired during the carding and spinning, must be removed by a second scouring process. The yarn-scouring process is, therefore, equally as important to the dyer as the scouring of loose wool.

A variety of oils are employed for the purpose indicated above, and they behave very differently when treated with alkaline detergents. Some, such as Gallipoli oil, are very easily emulsified, and, therefore, may be removed by a very gentle treatment. Others, such as linseed oil, are readily removed if the scouring process follows the oiling after a very short interval; but on standing they absorb oxygen from the atmosphere, and become changed into an insoluble sticky substance, which is not easily acted upon by the alkali. Again, mineral oil, when used alone, is not capable of forming an emulsion with alkalies, and is, therefore, not removed by ordinary scouring; in presence of some saponifiable oil, it appears, however, to emulsify, and thus the use of a mixture of mineral and vegetable oil in the oiling process, or of soap as a scouring agent, results in the removal of the mineral oil from the wool.

It is, therefore, of great importance that a suitable oil should be selected for oiling the wool, the use of a common cheap oil being a very false economy. A good quality of Gallipoli, olive, or neat's foot oil is perhaps the most highly esteemed for the purpose, but any oil possessing the following characteristics may be considered suitable: It must be readily emulsified by alkali; it must not turn rancid or become oxidised by exposure to air; it should, as far as possible, be devoid of colour and smell, and must, on no account, contain mineral acid: sulphuric acid being occasionally present as an

impurity in some samples of "oleine." It is, of course, obvious that mineral acid will quickly attack the wire teeth of the cards, and thereby necessitate frequent grinding.

As a preliminary to the yarn-scouring process, it is necessary, especially in the case of hard twisted yarns, to remove the tendency of the thread to curl up. This is done by immersing the yarn, in a stretched condition, in a bath of boiling water, and allowing it to cool without relaxing the tension.

To this end a little apparatus, called a "yarn stretching" machine, is made use of. It consists of two series of horizontal bars or pegs, one above another, held apart by two powerful screws, by rotating which the upper series can be raised or lowered. After placing the hanks of yarn upon the pegs it is stretched tightly by raising the upper framework, and then the whole apparatus is immersed in boiling water for a few moments, and, after removal, allowed to lie until cold before removing the hanks.

After this treatment, the *rationale* of which has been explained when speaking of the hygroscopic and elastic nature of the fibre, the yarn remains in a straight condition, and is readily handled in the scouring baths.

There are a number of different methods of scouring wool in the form of yarn, but the most usual plan is the very simple one of hanging the hanks side by side on a series of smooth wooden poles which are placed transversely across an oblong wooden vat. The hanks are then individually and systematically worked about by hand in the liquor until clean, and ultimately squeezed either by hand, or more usually by means of rollers. Another apparatus in which hand labour is partially done away with has a series of rotating reels to replace the poles and a travelling apron to carry the hanks through the squeezing rollers. The only manual labour required here is that of placing the hanks upon the reels, and afterwards removing them from the reels to the travelling apron. A continuous yarn-scouring machine is also employed in which the hanks are deposited by a feeding apron between two broad endless bands, and by

these carried through one or more scouring baths fitted with squeezing rollers.

Any of the alkalis mentioned as scouring agents for loose wool may be employed in yarn scouring, but soap with or without ammonia is most commonly used. If, however, an oil which does not readily emulsify is present, it may be necessary to use some stronger alkali, such as carbonate of soda.

#### CLOTH SCOURING.

The substances present as impurities depend upon the character of the cloth. If it has been dyed in the condition of loose wool, the cloth will contain the oil added before carding, the stiffening material used in sizing the warp, and any dirt contracted during the manufacture. If yarn dyed, it may contain only size: but if manufactured in the grey, both grease, size, and dirt will be present. The method of scouring should, therefore, depend upon the nature of the impurities, but the character of the colouring matter with which the cloth is dyed should be an equally important consideration. Indeed the scouring of wool in the form of cloth is so intimately connected with the milling, crabbing, and other finishing processes, that it is impossible to discuss them separately in an adequate manner.

Grey cloth (that is, cloth spun and woven in the undyed condition) will, as above noted, contain grease and size as principal impurities, and may be scoured either with soap or soda, or a mixture of the two. In the case of certain classes of cloths, which have a tendency to cockle up during the scouring (*e.g.*, certain union goods), it is usual to introduce previously the processes of crabbing and steaming. The first mentioned consists in passing the cloth in the open width, and under more or less tension, through one or two baths of boiling water, and finally through a bath of cold water. This treatment, which is equivalent to yarn stretching, gives the material a permanent set, and removes the tendency to pucker or shrivel up. The steaming process which follows consists



in wrapping the cloth tightly upon a perforated hollow metal cylinder, by means of which steam is forced through the material. After passing steam for a few minutes, the cloth is rewound on to a second cylinder and again steamed in order to equalise the effect: the original outer and inner ends thus being reversed.

Crabbing and steaming exert an oxidising effect which, in the case of certain oils, may render them more or less brown in colour and very difficult to remove. What appear to be mysterious stains or blotches are not unfrequently produced in this manner.

In the case of wool or yarn dyed cloth the scouring process should be so conducted that no injury to the colour results. The process should, therefore, be under the control of the dyer, or, at any rate, the method of dyeing which has been adopted should be taken account of by the finisher, who usually has charge of the process at this stage. On the other hand, since it is necessary that certain kinds of cloth should be heavily milled, it is equally important that the dyer, in selecting his colouring matters, should be aware of, and pay due regard to, the finishing processes to which his colours will be submitted.

In the common form of cloth scouring machine, called the "dolly" scouring or washing machine, the cloth passes through in the condition of a loose strand or chain, because, no attempt being made to keep the pieces in an open width, it becomes irregularly folded or pleated longitudinally. The essential parts of the machine are a pair of weighted squeezing rollers, underneath which is a small trough containing the scouring solution, and an outer and much larger vat which serves as a receptacle for the cloth. The necessary guiding rollers and steam pipes complete the main details. Two, or sometimes three, pieces of cloth are scoured simultaneously side by side in such a machine, each being stitched end to end to form an endless band. The squeezing rollers form the motive power by means of which the cloth is drawn

through the machine, and the lower one, since it runs in the scouring solution, serves to feed the latter on to the material. The cloth may run 20—30 minutes in each of two such machines, and is then washed with clean water in a third.

It is necessary in some cases, and desirable in many, to keep the cloth in the open width during the scouring process. The open width scouring machine is similar to the "dolly," but much broader, and is fitted with straining bars and other devices for the prevention of creases and folds in the cloth.

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### III.—WOOL BLEACHING.

The wool fibre naturally possesses a colour varying from pale yellow to brown, grey, or even black. In the case of "white" wool, the pale yellow colour is intensified by the scouring process, especially if the latter is at all severe. In order, therefore, that goods which are to be finished white or in any pale colours may exhibit their full beauty, it is necessary that this yellow tint should, as far as possible, be removed.

*The "Tinting" Process.*—For some classes of work it is considered sufficient to neutralise the yellow colour by applying to the material a very dilute solution of some blue, purple, or violet colouring matter, which, optically combining with the yellow, changes it to very pale grey. The latter being a neutral tint is much less obvious to the eye, and therefore the wool appears less coloured, or more nearly white than before this treatment.

The colouring matters chiefly employed for this purpose are indigo purple (sulphopurpuric acid), methyl violet, or some suitable acid violet; and the process consists in simply working the wool in a very dilute acid or neutral solution of the dye until the required degree of tinting is attained. It is evident, however, that the "white" obtained by this means will quickly reassume its yellow tint when the fabric is washed.

In order to produce an effect at once more permanent and more nearly approaching a pure white, the natural yellow colour of the wool must not merely be covered by tinting, but actually removed, and to this end a bleaching process is resorted to.

It is not customary to bleach wool which naturally possesses a dark colour, such fibre being used for the production of brown, grey, etc., fabrics, or mixed with white fibre for drab or "natural" coloured goods.

*Bleaching Processes.*—At the present time two entirely distinct methods of bleaching wool are practised, and they differ not only in the agents employed, but are also opposed in theory. In the older, and still most commonly used, process, the colouring matter of the wool is acted upon by certain *reducing* agents, and thereby decolourised. The pigment does not, however, appear to be destroyed, because wool which has been bleached in this manner becomes gradually yellow again, probably by oxidation of the decolourised pigment.

The more modern process is based on the fact that the colouring pigment is destroyed by certain *oxidising* agents, *e.g.*, hydrogen peroxide, and has indeed only been commercially practicable since that substance was placed upon the market at a cheap rate.

In carrying out the older process, which has been in use from time immemorial, sulphur dioxide in the gaseous form or in solution is the reagent almost exclusively employed.

*The Sulphur Dioxide Bleach.*—When sulphur is burnt in air *sulphur dioxide* (sulphurous oxide) gas is produced.

Cold water absorbs about thirty times its volume of this gas, forming *sulphurous acid*, which, like sulphur dioxide gas, readily absorbs oxygen when in contact with easily reducible substances, and is converted into sulphuric acid.

If sulphur dioxide gas is led into a solution of sodium carbonate, sodium hydrogen sulphite (bisulphite) is produced and carbonic acid is eliminated as follows:—



and this body, which is sold as bisulphite of soda, is also employed as a bleaching agent. When bisulphite of soda is treated with a mineral acid, sulphurous acid is liberated.

Wool is usually bleached in the form of yarn or cloth, but is sometimes treated in the form of loose wool, and there are two methods of applying the sulphur dioxide as a bleaching agent, known respectively as the *gas* and *liquid* bleach.

In *gas bleaching*, which is also called *stoving* or *sulphuring*, the  $\text{SO}_2$  is generated by burning the necessary quantity of sulphur, the resulting gas being led directly into the chamber containing the woollen cloth or yarn. The sulphur is ignited by means of a red-hot iron bar or cinder, and after ignition continues to burn without further application of heat. The bleach-house or chamber in which the operation is conducted should consist of a brick room, the ventilating openings, doors, etc., of which are capable of being tightly closed. The roof of the chamber should be so constructed that the moisture condensed thereon cannot drop on to the material, since, being chiefly sulphuric acid of considerable concentration, this condensed liquid is a frequent cause of damage.

In conducting a bleaching process the cloth or yarn is thoroughly wetted out and then placed upon the wooden rods with which the bleaching chamber is fitted. The chamber is then tightly closed, the fumes from the burning sulphur admitted, and the material allowed to remain all night. In the morning the chamber is first thoroughly ventilated and then emptied, the wool being subsequently well washed with water to remove sulphurous and sulphuric acids.

For thin material the rods may advantageously be replaced by rollers placed in series near the top and bottom of the bleaching chamber; and the cloth, travelling slowly, may be bleached by a single passage through the stove, the process thus being continuous.

The process of gas bleaching requires from five to ten per cent of sulphur—calculated on the dry weight of the wool treated—the amount varying according to the thickness, colour, etc., of the material.

In the *liquid bleaching* process a solution of sulphurous acid, or bisulphite of soda, is employed; in the latter case sulphuric acid being added in sufficient quantity to liberate the sulphurous acid. The material is simply steeped or worked in this solution for several hours.

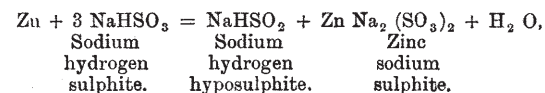
Whether "gas bleaching" or "liquid bleaching" is employed, the subsequent washing should be very thorough, since both the sulphurous acid used and the sulphuric acid formed during the operation are retained very tenaciously by the wool. The presence of any trace of sulphurous acid is especially objectionable when the bleached yarn is to be woven along with dyed threads, since many colours are considerably modified, and others entirely destroyed, by treatment with this re-agent. Sulphurous acid is, moreover, much more difficult to remove than sulphuric acid, and, therefore, it is the custom with some bleachers to pass the material after washing through a dilute solution of bleaching powder, or (better) hydrogen peroxide, which at once oxidises the sulphurous to sulphuric acid. This is then removed by a further washing with water.

*Theory of the Process.*—Several theories have been put forward to explain the decolourising effect produced by sulphur dioxide upon the natural colouring matter of wool. It has already been mentioned that wool bleached by this process gradually re-acquires a yellow colour, and this fact gives support to the idea that the bleaching action consists in a *reduction* of the colouring matter to a colourless state, the original colour being restored by a slow *oxidation* of the same by the atmosphere. Another theory supposes that the sulphur dioxide forms a *colourless compound* with the colouring matter, but that the first-named supposition is correct is rendered additionally probable by the fact that other reducing agents—such as stannous chloride in acid solution—will also decolourise the fibre.

*Hydrosulphite of Soda Bleach.*—This substance has been practically employed as a bleaching agent for several years, the process being conducted as follows :—

(1) *Preparation of the Hydrosulphite.*—Into a 100-gallon cask place 70 to 80 gallons of bisulphite of soda solution at 60° Tw., and then add granulated or rod zinc, until the cask is nearly full. Cover tightly, and allow the reaction to proceed for about an hour, or until the liquid no longer smells of sulphurous acid, taking care that the temperature does not rise beyond 90° to 100° Fahr. The liquid is then syphoned off into another vessel, and allowed to stand for several hours, when it deposits zinc sodium sulphite. Immediately the liquid is syphoned off, the first cask should be filled up with fresh bisulphite, a small quantity of zinc being added to take the place of that dissolved during the operation. If not required at the time for another operation, the zinc should be well washed with water, and the cask also filled up with water.

(2) *The Bleaching Operation.*—The clear liquid from which the zinc sodium sulphite has crystallised out consists of a solution of sodium hydrogen hyposulphite (hydrosulphite), and this is used directly as the bleaching agent, being diluted with an equal bulk of boiled water. The wool is steeped in this solution for six to eight hours, when the bleaching operation should be complete. After squeezing, the wool should be washed in boiled water, since if water containing air is used the fibre is apt to heat and become greatly depreciated, on account of the great affinity of the hydrosulphite for oxygen. The reaction representing the formation of the hydrosulphite may be expressed as follows :—



A considerable economy, both in time and cost, is claimed for this method as compared with the gas bleach or the bisulphite process.

*The Hydrogen Peroxide Bleach.*—The fact that the natural yellow colouring matter of wool is decolourised both by the action of reducing and oxidising agents has already been alluded to. By oxidation the colouring substance appears



to be permanently destroyed, whereas by treatment with reducing agents, *e.g.*, in the case of the sulphur bleach, the original yellow colour is somewhat easily redeveloped. The recognition of this fact has led to the rapid adoption of hydrogen peroxide as a bleaching agent, although the process is somewhat more costly than the sulphur dioxide bleach.

Several oxidising agents have from time to time been proposed as bleaching agents for wool, hair, feathers, etc.; for instance, the manganates and permanganates of potassium, barium, and lead, and the peroxides of hydrogen, barium, magnesium, and sodium.

The disinfecting liquids known as "Condy's Fluids" consist of alkaline manganates and permanganates, and their efficacy lies in the fact that they readily give off oxygen in presence of organic matter. In the same way when wool, etc., is steeped in a solution of potassium permanganate, the liberated oxygen destroys the colouring matter, but simultaneously brown manganic oxide is deposited upon the fibre. In order to complete the bleaching process this has to be removed, and to this end a subsequent treatment with sulphurous acid is resorted to, whereby the brown insoluble dioxide is reduced to the white manganous oxide which readily dissolves in the acid.

This process was at one time frequently used for bleaching feathers, and is used to a very small extent in wool bleaching.

Much greater practical importance is attached to the use of peroxides as bleaching agents. Theoretically speaking, no novelty can be claimed for this process, since the process of "grass bleaching," which has been in use from the earliest times, largely depends for its action upon the fact that traces of hydrogen peroxide exist in the atmosphere, and more especially in dew, rain, and snow. Hoar frost frequently contains a considerable amount of peroxide, as much as 0.001 gr. per litre having been found. Practically, however, the employment of peroxides dates back about twenty years, although a great impetus has been given to the process

by the recent considerable reduction in price of hydrogen peroxide and the manufacture of sodium peroxide on a commercial scale.

*Hydrogen Peroxide.*—This substance was discovered by Thénard in 1818, and when pure is a syrupy, colourless, transparent liquid of the composition,  $H_2O_2$ . It is capable of evolving at the ordinary temperature and pressure 333 times its volume of oxygen gas. It dissolves in water in all proportions, and is usually sold commercially as a dilute solution, containing 3 to 6 per cent  $H_2O_2$ , and thus containing 10 to 20 volumes of available oxygen. When pure, hydrogen peroxide in either concentrated or dilute aqueous solution is comparatively stable, but in presence of organic matter, alkalies, metallic salts, or indeed of any solid matter, it is readily decomposed. \*The dilute commercial solution may be concentrated on a water bath in an ordinary dish at  $75^\circ C.$ , until it contains about 50 per cent  $H_2O_2$ . This may then be concentrated by distillation under reduced pressure, and after repeated fractioning, almost pure (99 per cent)  $H_2O_2$  is obtained; it boils at  $84^\circ$ — $85^\circ C.$ , is volatile in air, has a strongly acid reaction, and irritates the skin.

Two definite hydrates,  $H_2O_2 + H_2O$  and  $H_2O_2 + 2 H_2O$  have been prepared in a solid condition at very low temperatures.

Hydrogen peroxide found its first practical application as a cosmetic, and as a renovating agent for old oil paintings. For bleaching purposes it was first introduced by Messrs. Hopkin and Williams, of London, in 1875 or 1876, and afterwards in an improved form by E. de Haen, of Hanover. It may be prepared by decomposing barium peroxide ( $BaO_2$ ) by means of sulphuric, carbonic, or hydrofluoric acid. One process is carried out as follows:—Finely ground barium carbonate is moulded into bricks with sawdust and pitch, and heated in a furnace to about  $1,000^\circ C.$  The barium oxide thus obtained is cooled to  $500^\circ C.$ , at which temperature it absorbs oxygen from the

\* R. Wolfenstein Berichte, 1894, p. 3307—3312.—*Jour. Chem. Soc.*, 1894, Abs., p. 73.

atmosphere, and the  $\text{BaO}_2$ , being suspended in water, is decomposed by a current of carbon dioxide. The solution of hydrogen peroxide is then decanted off, and the precipitated  $\text{BaCO}_3$  used for another operation. The preparation of  $\text{H}_2\text{O}_2$  from sodium peroxide will be referred to later.

Commercially, hydrogen peroxide is sometimes known as "oxygenated water" or "ozonised water." It usually contains a notable amount of metallic salts—chiefly barium salts—in solution, and is therefore somewhat easily decomposed. It is, however, much more stable in acid than in neutral or alkaline condition, phosphoric acid having the most preservative action. A small addition of sodium phosphate is therefore of advantage when a mineral acid is present.

Kingzett\* states that its stability is further increased by the presence of 2 to 3 per cent of alcohol, a sample of  $\text{H}_2\text{O}_2$  which showed a 20 per cent loss of strength in 176 days remaining quite unaltered when  $2\frac{1}{2}$  per cent of alcohol was added. The deterioration of commercial samples is also reduced to a minimum by storage in a dark cool place.

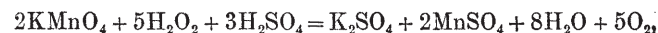
The strength of the 3 to 6 per cent solution of hydrogen peroxide which, as already stated, constitutes the commercial article, is usually indicated thus:  $\text{H}_2\text{O}_2$  (10 vols.),  $\text{H}_2\text{O}_2$  (12 vols.),  $\text{H}_2\text{O}_2$  (20 vols.), etc. This means that one volume of the liquid is capable of giving off 10, 12, or 20 times its volume of oxygen gas at the ordinary temperature and barometric pressure. Since, however, it is liable to considerable variation in strength, it is frequently necessary to make an actual estimation of the amount of  $\text{H}_2\text{O}_2$ . A 10-volume solution should contain (by weight) 3.04 per cent  $\text{H}_2\text{O}_2$  or 1.43 per cent available oxygen.

#### ESTIMATION OF HYDROGEN PEROXIDE.

Although hydrogen peroxide is essentially an oxidising agent, readily giving off one molecule of oxygen in presence of oxidisable substances, it behaves in an exactly opposite manner

\* *Jour. Soc. Chem. Indus.*, 1889, p. 3.

towards easily reducible substances. Thus by mixing together solutions of hydrogen peroxide and potassium permanganate—two powerful oxidising agents—in presence of acid, both substances are reduced, and a double volume of oxygen is liberated according to the equation



and upon this reaction the usual process for estimating samples of  $\text{H}_2\text{O}_2$  is based.

In carrying out the process a decinormal solution of permanganate is made use of, such a solution containing 3.156 grams  $\text{KMnO}_4$  per litre. From 10 to 20 grams of the sample of  $\text{H}_2\text{O}_2$  are weighed out and diluted with water. An amount of the solution equivalent to 1 gram of the original sample is then taken, about 3cc. of strong sulphuric acid is added, and  $\frac{N}{10}$  permanganate solution run in until a faint permanent pink colour is established. The amount of permanganate solution required is a measure of the strength of the original solution of  $\text{H}_2\text{O}_2$ , and by means of the following table the result may be at once read off without any calculation.

TABLE SHOWING STRENGTH OF AQUEOUS SOLUTIONS OF HYDROGEN PEROXIDE.

(When 1 gram  $\text{H}_2\text{O}_2$  solution is used for the estimation.)

Amount of $\frac{N}{10}$ $\text{KMnO}_4$ required.	Indicated percentage of $\text{H}_2\text{O}_2$ .	Corresponding oxygen strength in volumes at standard temperature and pressure.
1 cc. ....	0.17 per cent	0.56 volumes.
2 " .....	0.34 " "	1.12 " "
3 " .....	0.51 " "	1.68 " "
4 " .....	0.68 " "	2.24 " "
5 " .....	0.85 " "	2.79 " "
6 " .....	1.02 " "	3.35 " "
7 " .....	1.19 " "	3.92 " "
8 " .....	1.36 " "	4.47 " "
9 " .....	1.53 " "	5.03 " "
10 " .....	1.70 " "	5.59 " "
15 " .....	2.25 " "	8.38 " "
20 " .....	3.40 " "	11.18 " "
25 " .....	4.25 " "	13.97 " "
30 " .....	5.10 " "	16.77 " "
35 " .....	5.65 " "	19.56 " "
40 " .....	6.80 " "	22.36 " "

The second place of decimals is calculated to the nearest whole number.

If a solution of  $\text{KMnO}_4$  is employed containing 5 grams per litre instead of a decinormal solution (3.156 grams per litre); the results may be referred to the above table by multiplying the number of cc. of permanganate solution required by  $\frac{5.000}{31.56}$ .

An alternative process for determining the strength of solutions of  $\text{H}_2\text{O}_2$  by means of permanganate in which the use of a standard solution is not necessary, is as follows: A measured volume of the  $\text{H}_2\text{O}_2$ , say 5 cc., is placed in a graduated tube which is closed at one end (a nitrometer); then add a little sulphuric acid and read off the volume of air in the tube. A few crystals of potassium permanganate are now introduced, which immediately react with the peroxide, liberating a double volume of oxygen, as before explained. The gas mixed with the original air collects in the upper portion of the tube, and half the increased volume, when reduced to standard temperature and pressure, is the amount of oxygen liberated from the 5 cc. of  $\text{H}_2\text{O}_2$  used for the experiment; and working on the supposition that the 5cc. weighs 5 grams, which does not introduce any considerable error, by reference to the table, the percentage of  $\text{H}_2\text{O}_2$  may then be ascertained.

In the hands of a careful worker this process constitutes a very ready means of approximately estimating solutions of  $\text{H}_2\text{O}_2$  provided the necessary apparatus is at hand; but the method previously described should be, as a rule, preferably used.

The strength of solutions of hydrogen peroxide may also be estimated by mixing 10cc. with an equal amount of concentrated sulphuric acid (diluted) adding potassium iodide, free from iodate, and estimating the amount of liberated iodine in the usual manner by means of  $\frac{N}{10}$  thiosulphate of soda solution, using starch as indicator.

Notwithstanding the very excellent results obtained by bleaching wool with hydrogen peroxide, the use of this agent has never become general, on account of the expense of the process; and, in addition to the cost of production of the peroxide, the actual expenditure is largely increased by its comparative instability, and also, on account of the great

dilution of the commercial article, the considerable charges for carriage. The two last-mentioned defects have been surmounted by the introduction of *sodium peroxide*, which, being a solid body capable of liberating 13 times as much oxygen as an equal weight of hydrogen peroxide of 10 volume strength, is much less bulky and more easily stored and packed. It will also remain unaltered for an indefinite period if kept free from moisture and organic matter.

#### SODIUM PEROXIDE.

Potassium and sodium peroxides were first obtained by Gay-Lussac and Thénard in 1810, and were further studied by Vernon Harcourt in 1862, who gave the correct formulæ  $\text{K}_2\text{O}_2$  and  $\text{Na}_2\text{O}_2$ . They may be obtained by heating metallic potassium or sodium to  $200^\circ \text{C}$ . in a current of dry oxygen or air, or by dropping fragments of the metals into fused sodium nitrate. They may also be produced on an experimental scale by adding hydrogen peroxide to a solution of caustic soda or potash, and precipitating with alcohol, when the peroxide is deposited as a crystalline monohydrate.

When an aqueous solution of sodium peroxide is evaporated under reduced pressure, it crystallises out in plates having the composition  $\text{Na}_2\text{O}_2 \cdot 8\text{H}_2\text{O}$ . Under suitable conditions  $\text{Na}_2\text{O}_2 \cdot 2\text{H}_2\text{O}$  may also be obtained.

Commercially, sodium peroxide is of comparatively recent introduction. It is manufactured by the Aluminium Company of London, and by E. de Häen, of Hanover. The process made use of is similar to that originally devised by Vernon Harcourt, viz., metallic sodium is heated to a high temperature in aluminium vessels, and a stream of oxygen gas passed slowly through it. The composition of the commercial article is about as follows:—

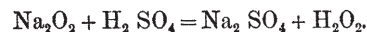
Sodium peroxide .....	93.0 per cent.
„ oxide .....	2.0 „
„ hydrate .....	2.5 „
„ carbonate .....	2.5 „
	100.0



The samples originally sent into the market contained a considerable amount of iron, but this has been largely eliminated by an improved method of manufacture.

The commercial article is a non-crystalline, anhydrous salt, of a pale yellow colour; which dissolves in water with considerable evolution of heat. As already stated, it is stable when dry, but in the moist condition it very readily parts with oxygen. In fact, so powerful is its oxidising action under these conditions, that it will readily ignite paper, and may even be the cause of serious explosions if when mixed with organic matter it is allowed to come into contact with water. Since it is very hygroscopic, the need of careful storage is therefore evident.

When a solution of sodium peroxide is heated, it gives off oxygen, and the solution becomes strongly alkaline; NaOH remaining in solution. When dissolved in dilute sulphuric acid, it is decomposed, sodium sulphate and hydrogen peroxide being produced, and both remaining in solution.



If care is taken to keep the solution cool during the reaction, no evolution of oxygen takes place.  $\text{Na}_2\text{O}_2$  thus forms a very convenient source of  $\text{H}_2\text{O}_2$ .

#### BARIUM PEROXIDE.

This substance ( $\text{BaO}_2$ ) has also been proposed as a source of hydrogen peroxide, which is readily obtained by treating it with an acid. It is formed by exposing baryta ( $\text{BaO}$ ), heated to dull redness, to a stream of air or oxygen gas; and is a grey substance only slightly soluble in water. At a very high temperature it loses the second atom of oxygen, being reconverted into the monoxide, and this double reaction is used for the preparation of oxygen on the large scale.

Weight for weight, sodium peroxide is much richer in available oxygen than either barium peroxide or hydrogen peroxide, the exact comparison being as follows:—

	Available oxygen by weight.
Sodium peroxide (pure).....	20.51 per cent.
"    "    (compl. 93%).....	19.07    "
Barium peroxide.....	9.48    "
Hydrogen peroxide (12 volumes)...	1.72    "

One pound of sodium peroxide (93 per cent) is thus equivalent to 11lbs. ( $1\frac{1}{10}$  gallons) of hydrogen peroxide of 12 volumes strength, or  $13\frac{1}{3}$ lbs. ( $1\frac{1}{3}$  gallons)  $\text{H}_2\text{O}_2$  (10 volumes).

#### THE BLEACHING PROCESS.

It is very necessary that wool which is intended for bleaching should be previously very thoroughly scoured. And this for two reasons: Firstly, in order that the bleaching solution may thoroughly penetrate into every portion of every fibre, which is impossible if the fatty matter is not removed; and secondly, because any greasy matter is liable to give rise to yellow oxidation products, which destroy the purity of the white.

The usual process for applying the peroxide is to steep the wool for from 12 to 24 hours in a bath containing a dilute solution of  $\text{H}_2\text{O}_2$ , made distinctly, but not strongly, alkaline with ammonia or other alkali. When the process is conducted in this manner, the strength of the solution may be such that it contains one volume of available oxygen, and a suitable alkalinity is obtained by adding about half per cent of the volume of the bath of strong ammonia. Such a bath would contain—

90 gallons water,
10    "    hydrogen peroxide (10 vols).
$\frac{1}{2}$ gallon ammonia, 0.008 sp. gr.

Sometimes silicate of soda is preferred to ammonia.

The amount of liquor used need only be sufficient to readily saturate the material operated upon, and at the end of the operation the excess of liquor should be allowed to drain into the bath, which may then be made up to its original strength and used again.

Another plan is to use several baths of gradually increasing strength, passing the wool through the weakest bath first. By this means each bath is in rotation quite exhausted, and then, being freshly made up, becomes in turn the strongest.

The character of the wool has some influence upon the speed at which the oxygen is liberated from the peroxide, and thus the method of conducting the process should vary somewhat according to the material treated; but by the help of the following

notes the necessary modifications will readily be made :—The speed at which the bleaching takes place may be increased by (1) raising the temperature of the bath ; (2) increasing the alkalinity of the solution ; (3) exposure of the material to light, especially sunlight, during the process. It is, however, difficult to conduct the process quickly without a great loss of oxygen, and, therefore, considerations of economy place a practical limit to its curtailment. What may be termed the safe temperature varies according to the substance treated and the alkalinity of the bath ; but it may be considered that 35°C. (95°F.) is the highest temperature that should be employed, although with a minimum of alkali 60° C. (140°F.) may be used if great care is exercised. The bleaching may even take place in a boiling bath if a large amount (25 to 30 per cent of the weight of wool) of certain metallic salts—notably magnesium sulphate—is added to the bath ; but the increased cost does not compensate for the saving in time. It should be always borne in mind, however, that whatever process is used the loss of oxygen may be very great, and the cost thus much increased, if the process is unskillfully or carelessly conducted.

The amount of ammonia or silicate of soda which is added to the bath should vary according to the character of the material and the temperature which is to be employed. If the material is of such a nature that a rapid evolution of oxygen takes place, the amount of alkali used should be small ; but, on the other hand, the amount may be increased in cases where the bleaching would otherwise be too slow, the presence of the alkali tending to accelerate the process. By saturating suitable brown wool with a strong solution of peroxide and then exposing to ammonia fumes, the bleaching action may even be followed by the eye.

The most beautiful white is obtained by exposing the wool to sunlight after steeping some time in the bleaching solution, but of course this is frequently impracticable.

If a bath containing a useful amount of peroxide is not required for some time, it should be acidified with sulphuric, or

better, with phosphoric acid (sodium phosphate + sulphuric acid), in order to prevent, as far as possible, loss of oxygen. Such a bath may be again rendered available by adding alkali.

After the bleaching, the wool should be squeezed, washed in water, then in acidified water, and finally in water again. It may then be treated with a dilute solution of soap or glycerine to soften the fibre, and dried at a low temperature.

#### SODIUM PEROXIDE.

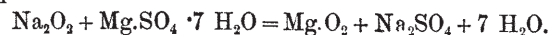
An aqueous solution of sodium peroxide cannot be used directly as a bleaching agent, on account of its strong alkalinity. It is, however, readily converted into either hydrogen or magnesium peroxide, and may be used in these forms ; but the method employed in the two cases is quite distinct.

If used as a source of hydrogen peroxide, the bath is prepared as follows :—The necessary amount of the solid  $\text{Na}_2\text{O}_2$  is added to dilute sulphuric acid until the latter is neutralised, the solution, which then contains  $\text{H}_2\text{O}_2$  and  $\text{Na}_2\text{SO}_4$ , being employed in exactly the same way as hydrogen peroxide. If the  $\text{Na}_2\text{O}_2$  is not pure, it is better, when neutralising the acid, to add an excess of peroxide, since the impurities are precipitated in alkaline solution and may be removed by decantation. The solution should then be slightly acidified with sulphuric acid to remove caustic alkali before making up the bath with ammonia or silicate of soda in the usual way. Since much heat is generated by the decomposition of the  $\text{Na}_2\text{O}_2$ , and loss of oxygen will occur if the temperature rises at all high, it is better to dilute the acid with the whole quantity of water ultimately required for the bath, in order to prevent undue heating. Each pound of  $\text{Na}_2\text{O}_2$  requires 11b. 6oz. of strong sulphuric acid (1.84 sp. gr.) for complete decomposition.

A bleaching bath that will contain one volume of available oxygen may be made up as follows :—To 90 gallons water add 10lbs. 5oz. D.O.V. ( $\text{H}_2\text{SO}_4$  1.84 sp. gr.), and then add gradually, with constant stirring, 7lbs. 8oz. sodium peroxide.

The necessary amount—say  $\frac{1}{2}$  gallon—of ammonia being then added, the bath is ready for use.

The use of magnesium sulphate in conjunction with sodium peroxide, as proposed by de Häen, renders it possible to use the bleaching bath at a high temperature, and thus greatly accelerates the process. The real bleaching agent in this case is probably magnesium peroxide, produced in accordance with the equation—



It is recommended that the substances be used in the proportion, 3 parts  $\text{Mg}.\text{SO}_4 \cdot 7 \text{H}_2\text{O}$  to 1 part  $\text{Na}_2\text{O}_2$ , and this approximates closely to the proportion theoretically required by the above equation, which is 3.16 parts  $\text{Mg}.\text{SO}_4 \cdot 7 \text{H}_2\text{O}$  to 1 part  $\text{Na}_2\text{O}_2$ .

The process is carried out as follows: Thoroughly wet out the wool in a solution containing 30 per cent of its weight of magnesium sulphate, then raise the wool, add 10 per cent  $\text{Na}_2\text{O}_2$ , and stir till dissolved; re-enter and raise the temperature of the bath to  $60^\circ$ — $70^\circ$  C ( $140^\circ$ — $160^\circ$  F.), maintaining that heat for one hour, when the process should be complete; then lift, squeeze, wash thoroughly, first in water, then in dilute hydrochloric acid, and finally in water.

On adding the  $\text{Na}_2\text{O}_2$ , the bath becomes milky, on account of the insolubility of the magnesium peroxide, and the washing in dilute acid after the process serves to remove any  $\text{MgO}$  which may be present on the wool.

The saving in time by this as compared with the ordinary process is very great, but the cost is considerably more.

The peroxide bleach is not only useful for goods which are to remain white, but also for wool which is to be dyed in pale delicate tints. After bleaching in this manner the wool dyes much more readily than before treatment, and the cost of the process is more than compensated for by the advantage gained. On the other hand, wool which has been bleached by the sulphur process cannot be dyed in light colours, on account of its destructive effect upon the latter.

#### IV.—WATER.

One of the primary requisites in a dyehouse is a plentiful supply of good water, but in judging of the suitability or otherwise of any source of water for the purposes of a dyer, one must, of course, be guided by different considerations to those which come into force, *e.g.*, when examining a water for potable purposes; since impurities which are very injurious in the one case may be quite harmless, or even beneficial, in the other. In many dyeing operations the use of absolutely pure water would be an advantage, but this is never obtainable in sufficient quantity. The impurities existing in water obtained from natural sources of supply vary greatly both in quantity and kind, and it is very important that dyers should know what are the impurities in the particular water they use, what influence these substances will exert in the various processes employed, and if injurious, how they may be removed or their effect counteracted.

In dealing with this subject, therefore, we shall first explain the method of detecting the common water impurities, and afterwards deal with the other points mentioned above.

##### SOURCES OF WATER.

From the surface of the ocean, and all bodies of water, a constant process of evaporation is taking place, the speed of the evaporation depending upon the heat and pressure of the atmosphere and the amount of aqueous vapour which it already contains. For a given temperature and pressure there is a certain definite maximum amount of moisture which can be retained in suspension; a rise either in temperature or pressure will increase the possible maximum amount, while a fall will have an opposite effect. It is evident, therefore, that a reduction of pressure or decrease in temperature of air



saturated with water vapour will cause a deposition of a part of the moisture, thus giving rise to rain or dew.

Aqueous vapour, when condensed, forms perfectly pure water, and therefore *rain water*, when intercepted and collected before reaching the ground, constitutes the most pure natural form of water; since it will contain only those impurities which it has absorbed during its passage through the air. In the usual course of things, however, the rain or dew falls on the ground, in which case a portion of the water simply drains over the surface, flowing to the lower levels, and thus forming and feeding the *streams* and *rivers*. Another portion sinks into the ground, through the soil and permeable rocks, until it reaches an impervious stratum. If the surface of this is not level, and is inclined in a suitable direction, the water may flow along it until it again reaches the surface of the ground at a lower elevation, in which case it forms a *spring*. On the other hand the water may collect upon the surface of the impervious rock or in some underground cavity; when, by boring down to this point from the surface of the ground, the water, which is then termed *well-water*, may be obtained by pumping. Excluding *rain water*, which is of little technical importance, we thus find that *rivers*, *springs*, and *wells* form the sources of our water supplies.

#### CAUSE OF IMPURITIES IN WATER.

Water exerts a more general solvent action than any other known substance, and therefore it is not surprising that in its passage through the ground it dissolves some of the vegetable and mineral substances with which it comes into contact. *River water*, frequently known as "*top*" water, has, for the most part, simply run over the surface of the ground, with which it has been in contact for only a short space of time.

It does not therefore, as a rule, contain so much dissolved impurity as *spring* or *well* water, but on the other hand is frequently muddy by reason of solid vegetable or mineral matter held in suspension.

River water is also in many places contaminated by the refuse from works situated higher up the stream, and as this class of impurities is the most variable and uncertain, it is as a rule the most difficult to deal with. As will be readily imagined, the purity of river water is also much more quickly and extensively affected by heavy rains or continued drought than is that of springs or wells.

*Spring* or *well water*, however, which has necessarily passed through a greater or less thickness of rocky formation, always contains in solution a quantity of mineral salts which it has dissolved during the passage. The composition and amount of the dissolved salts is of course determined by the character and extent of the strata through which the water has passed. Spring and well water are, as a rule, free from mechanically-suspended impurities, and appear quite clear and bright. Water obtained by boring operations, *i.e.*, *well* water, usually contains more mineral matter than a *spring* water from the same district. This is explained by the fact that the presence of a spring is in itself a proof that the water composing it has some means of circulation, otherwise no flow could be produced.

On the other hand, well water may have remained stagnant for a long period of time, saturating some rock, from which it gradually dissolves a soluble constituent.

If the strata composing the earth's crust were distributed uniformly and parallel to the surface, rain water would, of course, in all places penetrate to the same depth before reaching an impervious stratum. In such a case it would always be possible to ensure a supply of water by boring down to that point. The actual state of things is, however, very different from this. The various layers of rocks, shales, clays, gravels, etc., of which the earth is composed, have been twisted, bent, broken, disarranged and rearranged by natural forces in a most remarkable and irregular manner, and it is impossible for any one who is not a skilled geologist, possessing a special acquaintance with the locality, to predict whether, and at what depth, water is likely to be found in any particular place.

Indeed, even with the best available information, such opinions are subject to great uncertainty.

It will be seen, therefore, what a very risky proceeding it is for a manufacturer to commence to sink a well in expectation of finding water at a certain depth, simply because a neighbouring works is supplied from such a source. This is, however, by no means uncommon.

In the case of well water, the character of the impurities, as well as the amount of water supplied, may alter considerably after the pumping has been continued for a few days, weeks, or months, this being due to the exhaustion of some underground cavern or reservoir which had been tapped.

#### CHARACTER OF IMPURITIES.

As has been indicated above, the impurities in water may be of two kinds, viz., those which are merely mechanically mixed with, or suspended in, the water, and those in a state of solution. Water containing suspended impurities will, of course, appear more or less turbid or muddy, while if dissolved impurities only are present it will be quite clear and probably colourless. Suspended impurities are thus visible to the eye, and may be got rid of by mechanical means, whereas dissolved impurities can only be detected by chemical reactions, and require a chemical treatment for their removal.

#### MECHANICAL IMPURITIES.

These consist principally of mineral matter, in the form of mud and sand, organic matter from the banks of the stream or the gathering ground, and solid matter turned into the river by works or towns situated higher up the stream. The composition and amount of these is very variable, not only in different places but also at any given place at different times. Generally speaking, however, they do not cause much trouble to the dyer, unless present in large amount; although, if oxide of iron is found, the water cannot be considered fit for use in dyeing and scouring operations until the iron has been removed.

All suspended impurities may be got rid of by means of settling tanks or reservoirs, or by filtration. In works where sufficient ground space is available a very good arrangement is as follows:—The water is pumped from the river into large shallow reservoirs placed at a sufficient elevation which it slowly traverses, depositing most of the solid matter during its passage. It then passes down a slight incline so formed that the water runs in a shallow, broken stream; the consequent aeration being of special value when an iron salt is present, since the iron is oxidised to ferric oxide, which readily deposits. From the incline the water may run into storage reservoirs and from thence by gravitation into the works.

It frequently occurs that from want of space, or other cause, it is impracticable to carry out this system, in which case a simpler but less effectual plan may be adopted, viz.:—The water is pumped into large tanks or cisterns placed on the roof of the works, running from these through fine cloth filters into the water mains.

#### DISSOLVED IMPURITIES.

These are much more important from a dyer's point of view than the suspended matters; and since by far the greater portion of the soluble matter forming the earth's crust consists of salts of lime and magnesium, these are the chief impurities found in solution; in fact, natural waters, almost without exception, contain more or less calcium or magnesium compounds. Other substances, also, are frequently found, and for technical purposes the dissolved impurities may be classified as follows:—

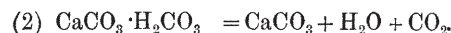
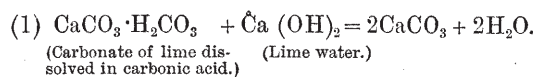
- (1) Lime and magnesium salts.
- (2) Compounds of iron.
- (3) Alkaline impurities.
- (4) Free acids and acid salts.

(1) *Lime and Magnesium Salts.*—These are generally present, either as *carbonates* (held in solution by free carbonic acid) or as *sulphates* or *chlorides*; and since, in dyeing operations, the

action of carbonates differs greatly from that of the last-named salts, every dyer should be able to make such simple tests as are necessary to determine the character and approximate amount of these impurities.

*Lime Salts* may be detected by adding to a small portion of the water in a test tube a solution of ammonium oxalate, a white precipitate indicating the presence of calcium. *Magnesium* is not precipitated under these conditions, but on evaporating the liquid to a small bulk after removing the lime salts, an addition of ammonia and ammonium phosphate gives a white precipitate in presence of magnesium.

The next step is to determine in what form the calcium and magnesium are present. *Carbonates* of these metals may be detected in one or two ways. (1) A solution of clear lime water gives a white precipitate; (2) on boiling the water the carbonates are precipitated. Both these reactions depend upon the fact that calcium and magnesium carbonates are insoluble in pure water, but dissolve in water containing free carbonic acid, which is present in almost all natural waters. If the free carbonic acid is removed, the carbonates thereby held in solution are precipitated, and this is effected by either of the reactions above described, in accordance with the following equations:—



In the first case the calcium hydrate which is added combines with the carbonic acid which is acting as a solvent, being converted into carbonate of lime, which is precipitated along with that originally present in the water. In the second reaction the carbonic acid is gradually driven off by the boiling and the carbonate thus precipitated.

In the case of most samples of water the lime and magnesium are not present entirely in the form of carbonate, but partly in combination with sulphuric, hydrochloric, or nitric acid. These salts also may be detected in a simple manner.

The presence of *chlorides* is indicated, if a white precipitate is obtained by adding nitric acid and silver nitrate to the water, while *sulphates* produce a white precipitate of barium sulphate on addition of hydrochloric acid and barium chloride. *Nitrates* are less frequently found, and, if present, contamination with sewage is probable; and although this may not be of much importance as regards dyeing operations, it, of course, renders the water quite unfit for domestic use. For the detection of nitrates a quantity of the water must be evaporated to small bulk, a little ferrous sulphate solution added, and then concentrated sulphuric acid carefully poured down the side of the test tube to form a separate lower layer of liquid. Nitrates are indicated by the formation of a brown ring at the junction of the two layers.

#### DETERMINATION OF AMOUNT OF LIME AND MAGNESIUM SALTS.

The method usually adopted for the estimation of lime salts in water is based upon a fact of everyday experience, viz.: that "hard" water will not form a lather with soap until a certain amount of the soap has been rubbed away. At first the soap produces merely a curdy scum or precipitate, but by continuing the rubbing a lather is eventually obtained.

The explanation of this fact is as follows:—Ordinary soap consists of a fatty acid, say oleic, stearic, or palmitic acid, in combination with either sodium or potassium. These fatty acids will, however, also combine with other metals, such as calcium, magnesium, iron, etc., the resulting compounds or soaps being insoluble in water, and of a sticky or curdy nature. The scum produced when soap is employed in conjunction with "hard" water consists, indeed, of the insoluble lime soap, and until the whole of the lime, etc., present in the water is thus combined, no lather is produced.

The amount of soap destroyed by a given quantity of the water before the lather is produced may thus serve as a measure of the amount of lime, etc., present in the water; although for reasons which will be touched upon later, the determination is by no means an accurate one.



## TEMPORARY AND PERMANENT HARDNESS.

It will be remembered that on boiling a water containing carbonate of lime in solution, the carbonic acid, which holds the carbonate in solution, is driven off, and the latter is precipitated. If, now, the water is filtered, the lime originally present as carbonate is entirely got rid of. On this account, a water containing carbonates only, is said to possess "removable" or "temporary" hardness; while a water in which sulphates or chlorides are present is said to be "permanently" hard, because the "hardness" is not removable by boiling. Most samples of water possess both "temporary" and "permanent" hardness, the sum of the two giving the "total" hardness.

In making an estimation of the hardness of water by means of soap, the "total" hardness is first determined, and then another portion of the water is boiled and filtered (thus removing the carbonates) and the "permanent" hardness estimated, the difference between the two giving the "temporary" hardness which was lost during the boiling.

This method of estimating the amount of lime and magnesium salts in water is known as "Clarke's Soap Test." For carrying out the process, two solutions are required, (a) standard soap solution and (b) hard water of known composition with which to determine the value of the soap solution.

The following details (abstracted from Thorpe's "Quantitative Analysis") indicate the method of procedure.

(a) *Preparation of Standard Hard Water.*—Weigh out into a porcelain dish exactly 0.2 grms of finely powdered and dried calcite, and dissolve in dilute hydrochloric acid, covering the dish meanwhile with a watch glass. Heat the basin on a water bath, and when no more carbonic acid is evolved rinse the underside of the watch glass into the basin and evaporate to complete dryness. Add a small quantity of water, and again evaporate to dryness, to ensure the complete removal of excess of hydrochloric acid, then dissolve the calcium chloride in distilled water, and dilute to 1,000 cc.

(b) *Preparation of Standard Soap Solution.*—Pound in a mortar 3 parts of lead soap (lead plaister) and 1 part dry potassium carbonate; mix thoroughly, add a small quantity of methylated spirit, and triturate until a thin creamy mixture is obtained. After standing for some time pour off

the clear solution of potash soap through a filter, and exhaust the residue repeatedly with fresh quantities of methylated spirit. The white insoluble residue is lead carbonate.

Now dilute the strong soap solution thus obtained with a mixture of 2 parts methylated spirit and 1 part water, proceeding as follows: Transfer 50 cc. of the standard hard water to a stoppered bottle of about 250 cc. capacity; fill up a burette with the soap solution, and add the latter gradually—about 1 cc. at a time—to the stoppered bottle, shaking vigorously after each addition. The process is finished when a uniform lather is obtained, which remains permanent for at least three minutes, and which is reproduced on again shaking the liquid. Having thus determined the strength of the strong soap solution, it is diluted with the mixture of alcohol and water until exactly 14.25 cc. are required to produce a permanent lather with 50 cc. of the standard hard water.

*The Process.*—Transfer 50 cc. of the water under examination to the 250 cc. bottle, shake vigorously and suck out the air by the aid of a glass tube in order to remove the  $\text{CO}_2$  expelled on agitation. Fill up the burette with the soap solution and then add the latter, 1 cc. at a time, to the water; after each addition inserting the stopper and shaking vigorously. As soon as there is an indication of the formation of a lather, add the soap solution in smaller amounts until the point of permanent lather is reached. The weight of calcium carbonate in 100,000 parts of water corresponding to the number of cc. of soap solution required for 50 cc. of the sample water is given in the following table (*see page 88*), in which column I = volume of soap solution required, and column II = corresponding amount of  $\text{CaCO}_3$ . If it is desired to express the result in the form of grains per gallon, the figures given in column II. must be multiplied by  $\frac{1}{175}$ .

It will be noticed that although (*e.g.*) 6 cc. soap solution in Col. I. correspond to 7.43 degrees of hardness in Col. II, 12 cc. soap solution do not indicate double this amount, but 16.43 degrees of hardness. This irregularity is indeed the reason why the table is necessary, since if the numbers in the two columns bore a definite relationship to each other a simple formula would suffice.

This soap test for determining the hardness of water, although very useful, cannot by any means be considered an accurate analytical process for the estimation of the lime and magnesium salts, since the result obtained is influenced by the presence of many other substances. For instance, the presence of free acid or alkali gives a reading correspondingly too high or

CLARKE'S SOAP TEST.—TABLE OF HARDNESS.

I.	II.	I.	II.	I.	II.	I.	II.
cc.		cc.		cc.		cc.	
0.7	0.00	4.6	.43	8.5	11.05	12.4	17.06
0.8	0.16	.7	.57	.6	.20	.5	.22
0.9	.32	.8	.71	.7	.35	.6	.38
1.0	.48	.9	.86	.8	.50	.7	.54
.1	.63	5.0	6.00	.9	.65	.8	.70
.2	.79	.1	.14	9.0	.80	.9	.86
.3	.95	.2	.29	.1	.95	13.0	18.02
.4	1.11	.3	.43	.2	12.11	.1	.17
.5	.27	.4	.57	.3	.26	.2	.33
.6	.43	.5	.71	.4	.41	.3	.49
.7	.56	.6	.86	.5	.56	.4	.65
.8	.69	.7	7.00	.6	.71	.5	.81
.9	.82	.8	.14	.7	.86	.6	.97
2.0	.95	.9	7.29	.8	13.01	.7	19.13
.1	2.08	6.0	.43	.9	.16	.8	.29
.2	.21	.1	.57	10.0	.31	.9	.44
.3	.34	.2	.71	.1	.46	14.0	.60
.4	.47	.3	.86	.2	.61	.1	.76
.5	.60	.4	8.00	.3	.76	.2	.92
.6	.73	.5	.14	.4	.91	.3	20.08
.7	.86	.6	.29	.5	14.06	.4	.24
.8	.99	.7	.43	.6	.21	.5	.40
.9	3.12	.8	.57	.7	.36	.6	.56
3.0	.25	.9	.71	.8	.52	.7	.71
.1	.38	7.0	.86	.9	.68	.8	.87
.2	.51	.1	9.00	11.0	.84	.9	21.03
.3	.64	.2	.14	.1	15.00	15.0	.19
.4	.77	.3	.29	.2	.16	.1	.35
.5	.90	.4	.43	.3	.32	.2	.51
.6	4.03	.5	.57	.4	.48	.3	.68
.7	.16	.6	.71	.5	.63	.4	.85
.8	.29	.7	.86	.6	.79	.5	22.02
.9	.43	.8	10.00	.7	.95	.6	.18
4.0	.57	.9	.15	.8	16.11	.7	.35
.1	.71	8.0	.30	.9	.27	.8	.52
.2	.86	.1	.45	12.0	.43	.9	.69
.3	5.00	.2	.60	.1	.59	16.0	.86
.4	.14	.3	.75	.2	.75		
.5	.29	.4	.90	.3	.90		

too low; the former increasing and the latter decreasing the apparent hardness as indicated by the soap test. To take an extreme case: If carbonic acid gas is led into pure distilled water, the latter may show 10 or 12 degrees of hardness by the soap test, although, of course, no lime whatever is present.

Again, in the case of samples of water that possess more than about 15 degrees of hardness, the test is very unreliable. A sample may, for instance, show 25° hardness if 50cc. is titrated in the usual way, but if 25cc. is taken, diluted with 25cc. of pure water, and then titrated, the number found on multiplying the result by 2 may be 35° or even 40° hardness. It is evident, however, that for practical purposes the supposed sample is only 25° hard as regards soap-destroying power, since in practice it would not be diluted. In other ways the relative proportion between soap-destroying power and the actual amount of Ca. and Mg. present, varies in an erratic and unexplained manner.

A much more accurate method of determining the actual amount of lime, etc., present as carbonate and as sulphate is the "acidmetric" test. In this process the temporary hardness is estimated directly by titrating a known volume of the water with standard  $\frac{N}{100}$  acid, using methyl orange or lacmoid as indicator. If any free alkali is present the result will, of course, be too high, but free carbonic acid does not influence the result. The total hardness is determined by evaporating a known volume of the water to dryness in presence of carbonate of soda, which changes the sulphates, etc., of lime into carbonates. The precipitated carbonate is then collected on a filter, washed free from excess of soda, dissolved in a known amount of standard acid, and the excess of the latter determined by titration with standard alkali. The amount of acid necessary to neutralise the whole of the lime, etc., present in the water is thus arrived at, and if the "temporary" hardness is deducted from this, the "permanent" hardness, due to sulphates, etc., is obtained.

The most direct way of arriving at a fairly accurate estimation of the true hardness of a water is to estimate the "permanent" hardness by the soap test, and the "temporary" hardness by acidmetry, in which case the inaccuracy of the former and the more complicated nature of the latter process are in great measure eliminated.

## IRON AS AN IMPURITY.

Iron is frequently present in water which is obtained from beds of shale or from iron or coal mines, and from a dyer's point of view is an even more objectionable impurity than calcium and magnesium. When iron is present in a considerable amount the water is termed a *chalybeate* water. Examples of such are found at Spa, Tunbridge Wells, and in one of the Harrogate springs, but waters of this type are not employed industrially.

The presence of iron in smaller quantities is, however, frequently demonstrated by the appearance of brown ferric oxide on half-covered stones in a river or stream.

Iron is usually present in water either as carbonate, held in solution as free carbonic acid, or as sulphate. The latter is produced by the atmospheric oxidation of naturally occurring ferrous sulphide (iron pyrites). The ferrous sulphate thus produced readily undergoes further oxidation and dissociates, ferric oxide being precipitated and sulphuric acid remaining in solution. On account of this decomposition, a water which contains iron, or from which the iron has been precipitated, frequently also contains free sulphuric acid.

## DETECTION OF IRON.

The presence of any considerable amount of iron is usually rendered self-evident by the formation of the brown deposit of ferric hydrate before referred to, but definite information as to the presence or absence of iron is obtained by applying the following test. A quantity of the water (say 200cc.) is made slightly alkaline with carbonate of soda and then evaporated to small bulk. If a deposit is obtained, it should be collected on a filter, washed, and dissolved in a small amount of pure hydrochloric acid. The acid solution is then boiled with a few crystals of potassium chlorate, in order to oxidise any ferrous salt into ferric salt. A few drops of potassium ferrocyanide (yellow prussiate of potash) solution is added, which at once produces a blue precipitate if iron was present in the original water.

A simpler, but less conclusive, test is to add to a sample of the water a few drops of a freshly made decoction of logwood (not logwood extract). In pure water a straw colour only is given, whereas the presence of even a trace of iron results in the development of a grey or black colour. Lime or magnesium salts produce a claret or purple colouration. A solution of tannic acid (from gall nuts, sumach, or myrobolans) also furnishes a delicate test for the presence of iron (ferric) salts, extremely dilute solutions of the latter giving a blue or black colouration.

## ESTIMATION OF IRON.

The amount of iron present in a sample of water may be determined as follows:—Evaporate 1 litre of the water to dryness (500cc. if much iron is present), acidify with hydrochloric acid, filter, add to the filtrate a few drops of hydrochloric acid, and boil for a few moments. Now add a slight excess of ammonia, allow the precipitated ferric hydrate to settle, then filter it off and dissolve in a very little hydrochloric acid. Again add ammonia, collect the precipitate on a weighed filter paper, dry and weigh the  $Fe_2O_3$ .

## FREE ACIDS AS IMPURITIES.

The source of free sulphuric acid, as a water impurity, has already been alluded to. Ferrous sulphide is oxidised to ferrous sulphate, which being dissolved by the water is gradually decomposed, ferric oxide being deposited and sulphuric acid liberated; and in the absence of carbonates, the free acid remains in the water.

Sulphuric acid is the only mineral acid which is at all commonly met with, but certain organic acids, derived from decomposing vegetable matter, are of frequent occurrence in surface water; these so-called *peaty acids* being an objectionable impurity on account of their great solvent power for iron. Free carbonic acid is also generally present in water, but since it is expelled on boiling, it does not cause any trouble during dyeing processes, although it will decompose a certain amount of soap in scouring operations.



The presence of peaty acids imparts a yellow or brownish colour to the water, and the amount may be approximately estimated by noting the amount of a standard solution of potassium permanganate destroyed by a given volume of the water. Sulphuric acid may be estimated by treating with  $\frac{N}{100}$  alkali using methyl orange as indicator.

#### ALKALINE IMPURITIES.

Water from wells which tap the lower coal measures not unfrequently contain sodium carbonate in considerable amount, as much as 10 parts  $\text{Na}_2\text{CO}_3$  in 100,000 parts water (= 1 lb. per 1,000 gallons) being by no means rare. Any such quantity as this would of course exert a great effect in many dyeing operations, but fortunately its action, if injurious, can be neutralised by adding the corresponding amount of sulphuric acid.

The presence of free alkali may be detected by means of a suitable indicator, such as red litmus paper, phenol phthalein, etc., and the amount estimated by titration with  $\frac{N}{10}$  or  $\frac{N}{100}$  acid, after evaporating the water to small bulk in order to precipitate any carbonate of lime, etc., which is removed by filtration.

#### LEAD AND COPPER IMPURITIES.

Salts of these metals are of much less frequent occurrence than calcium, magnesium, or iron compounds, although if present they are equally objectionable. Lead may be detected by evaporating 1 litre of water to about 50cc., adding a few drops of acetic acid and then passing sulphuretted hydrogen gas through the solution, a brown colouration being obtained if lead is present. If a little potassium ferrocyanide (yellow prussiate) solution is added to the concentrated water a brownish-red colouration indicates that copper is present.

#### EFFECT OF WATER IMPURITIES DURING VARIOUS DYEING, ETC., OPERATIONS.

In discussing the influence exerted by these substances it will be convenient to take the processes in the order in which they

are practically employed. The first to be considered will therefore be

#### (a) WOOL SCOURING.

When soap is one of the scouring agents employed, the use of water containing calcium or magnesium salts in notable amount is very objectionable, because, as already mentioned, this leads to the formation of sticky insoluble Ca. or Mg. soaps, which are not only quite useless as detergents, but become fixed upon the wool in such a permanent manner that they cannot be removed by washing with water. This results in a waste of soap corresponding to the amount of metallic salts present, and there is also the probability of trouble in the subsequent dyeing operations. In the case of some dyestuffs—the azo scarlets for example—the lime soap acts as a *resist*, preventing the fixation of the dye, and thereby causing light-coloured or white spots. With other colouring matters it plays the part of a *mordant*, thus producing darker coloured irregularities, e.g., when dyeing with magenta, the *fatty acid* of the lime soap acts as a mordant, while in the case of alizarin the *calcium* or *magnesium* is the combining body.

Iron, lead, or copper salts act in a precisely similar manner in this case to calcium or magnesium, but with iron the defect is greatly accentuated, because the iron soap possesses a brown colour, and iron mordant gives dark, dull shades with most colouring matters.

It should be remembered that an amount of soap is destroyed equivalent to the *total* amount of metallic salt present, and not merely an amount corresponding to the carbonates.

Free acid, if present in the water, will also decompose its equivalent of soap, but, since some alkali is usually employed with the soap, no bad effect results.

The presence of free alkali in the water may, of course, be neglected in scouring operations.

Frequent defects of the kind known as “white lists” or “dark lists” are the result of using a hard water for the purpose of washing off piece goods after scouring, mordanting, or dyeing.

The defect arises from the capillary flow of the water into the selvages caused by the latter becoming the most quickly dry when the cloth is rolled up in a wet condition. As it evaporates, the water deposits all the solid matter it contains in solution, and there is thus a continual deposition of the lime, etc., at the edges of the material, which, as indicated above, acts as a mordant or a resist.

(b) MORDANTING.

If the precaution is taken to neutralise with sulphuric acid any carbonates present in the water, salts of lime, magnesium or sodium do not exert a noticeable injurious action in ordinary mordanting processes. When oxalic or tartaric acid, or cream of tartar, is used there is, however, undoubtedly a loss of these substances through the production of calcium oxalate or tartrate which will exert much less action than the free acids or potassium salts; although by neutralising the carbonates with sulphuric acid the effect is even here diminished.

Aluminium and tin mordants are employed only for bright colours, whereas iron, copper, and lead produce dull, dark shades. Therefore if the latter metals are present as impurities in the water, the beauty and brilliancy of the aluminium and tin shades will be diminished in proportion to the amount of impurity present.

(c) DYEING.

The effect of the different water impurities during dyeing operations varies greatly according to the colouring matter employed. With some dyes the character of the water used has very little effect indeed, whilst in other cases it is quite impossible to produce a satisfactory result when a hard water only is available.

In *vat indigo* dyeing the presence of any of the common water impurities may be quite neglected, since the amount of lime present would constitute but a fraction of the amount added in setting the vat; other impurities are also without action. Even in this case, however, the washing off, after cleansing with acid, should certainly be done with soft water.

*Basic Coal-tar Colours.*—Most of the basic coal-tar colours are readily precipitated by metallic salts, some, such as *auramine* and *Victoria blue*, being very sensitive in this respect. The use of hard water in the application of this class of dye is therefore very wasteful, since the precipitated colour is not only useless, but, adhering to the surface of the wool, causes the defect of “rubbing off.”

*Acid Colouring Matters.*—This class of dyes, which includes the acid coal-tar reds, yellows, blues, greens, blacks, etc, and indigo extract, is, generally speaking, less affected by water impurities than any other. It is, however, necessary to add to the bath such an amount of sulphuric acid as will neutralise all carbonates present, in addition to the amount normally required in the process.

*Mordant Dyes.*—It is not possible to make any general statement dealing with the action of the impurities when dyeing with mordant dyes. In some cases it is quite essential that some lime salt should be present in the dye bath; for instance, when dyeing logwood blues (alum mordant), alizarin reds (alum mordant), alizarin purples (iron mordant), etc. Even in such instances, however, there is a certain definite amount of lime salt which gives the best result, and it is, therefore, preferable to use pure water and, in each instance, add the amount of lime salt necessary.

The action of the lime, where beneficial, is usually twofold. One portion, and indeed the larger part, acts as a neutralising agent for the acid which is inevitably carried over from the mordant bath into the dye bath when acid mordants are employed. In many cases another portion of the lime enters into the composition of the colour lake which is obtained by the combination of the mordant and dyestuff; thus, alizarin red has been shown to be a triple compound, containing calcium as an essential constituent—viz., an alizarin-aluminium-calcium lake.

With many other mordant dyes, the presence of lime in the water used for dyeing exerts a very injurious action, *e.g.*, in the

case of logwood blacks (chromic acid mordant), cochineal scarlets (tin mordant), alizarin blues (chrome mordant), etc.

It is not improbable that with these dyes also, calcium compounds are formed; but that as these lakes do not possess a pleasing colour the action of the lime is considered to be injurious.

The action of lime, when dyeing with logwood, is quite a special one. Before being used by the dyer, logwood undergoes the preliminary operation of "ageing" or "maturing," which is essentially a development of the colouring matter by oxidation. The oxidation takes place much more rapidly in presence of lime or other alkali, and the colouring matter is easily destroyed by "overageing." By the use of a water containing lime the oxidation is therefore still continued in the dye-bath, with consequent destruction of colouring matter if the wood was previously fully matured.

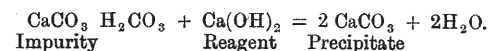
*General Remarks on the Action of Water Impurities.*—Sufficiently detailed notes have been given above to show that a water containing any considerable amount of impurity is quite unfit for general use in a dyehouse. The amount of colouring matter, soap, and other wares which is wasted in a dyehouse where a hard water is employed is very great; and if to this is added the cost and annoyance entailed by bad work due to the same cause, it will be seen that a works is very seriously handicapped thereby. The general level of the work is, indeed, to some extent, deteriorated, and falls short of the standard which would be attained if a more suitable water supply were available.

#### CHEMICAL PURIFICATION OF WATER.

For general use in a dyehouse water should not contain more than 4 or 5 parts of dissolved solids per 100,000 parts water—say, 3 grains per gallon. If, therefore, this amount is much exceeded, it is necessary, or at any rate very desirable, to submit the water to a chemical treatment before use.

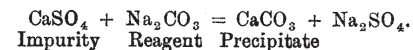
The reactions involved in a technical purification of water are comparatively simple. It has already been noted that the

impurities consist mainly of sulphates of lime and magnesium and carbonates held in solution by free carbonic acid. The carbonates are eliminated by precipitation with clear lime water, the re-agent combining with the free carbonic acid, and being precipitated as carbonate of lime along with the carbonate already present in the water. The reaction is represented by the following equation:—



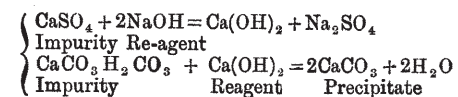
It is evident, therefore, that the amount of re-agent which should be added should be governed by the amount of free carbonic acid present, and does not bear any fixed ratio to the amount of calcium carbonate in the water, because in some cases the free acid may be only just sufficient in quantity to hold the chalk in solution, while in others it may be present in large excess.

The sulphates or chlorides are also precipitated as carbonates by adding the necessary amount of sodium carbonate:—



The equation indicates that sodium sulphate remains in solution in the water. The small amount thus produced is, however, quite without action in most dyeing processes.

Most waters contain both carbonates and sulphates, and if the latter and the free carbonic acid are present in equal molecular amounts, the purification may be simplified, one re-agent—caustic soda—replacing the lime-water and carbonate of soda; since, reacting with the sulphates, it produces from that impurity the lime-water (calcium hydrate) necessary for the precipitation of the carbonates thus:—



If both carbonates and sulphates are present, the latter in excess, the purifying agent should then consist of a mixture of caustic soda and carbonate of soda, while if carbonic acid



is in excess of the amount indicated by the equation, a mixture of caustic soda and lime-water should be added.

It is, of course, absolutely essential that the amount of re-agents used should be in exact accordance with the amount of impurity present. If added in excess the re-agents themselves constitute impurities. It is therefore necessary to possess an exact knowledge of the character and amount of the impurities in the water before attempting its purification, and also to control the purifying process by frequent analyses of the water, since it may vary in composition from day to day.

The following simple method of analysis gives all the necessary data. It involves three estimations, viz.:—(a) Free carbonic acid; (b) amount of carbonate of soda necessary to change the sulphates, etc., into carbonate; (c) amount of lime, etc., present as carbonates.

(a) The free carbonic acid is determined by titrating a known volume of the water with clear saturated lime-water, using phenol phthalein as indicator.

(b) The amount of carbonate of soda necessary for the removal of the sulphates, etc., is estimated by titrating with a standard solution of carbonate of soda, again using phenol phthalein as indicator.

(c) The carbonate of lime is estimated by titration with dilute standard sulphuric acid  $\left(\frac{N}{100} \text{H}_2\text{SO}_4\right)$  with methyl orange as indicator, and from these three results the proper proportion of purifying agent is readily calculated.

*Water Purifying Apparatus.*—The purifying process consists in mixing together the purifying agents and the water to be purified in proper proportion, and then removing the precipitated carbonates. The simplest possible process is to allow the water and a solution of the re-agents to run in regulated amounts into a small mixing tank and from thence into large settling tanks or reservoirs, from which it is afterwards drawn for use. The settling tanks may be replaced by filter beds, in which case a smaller area will give an equal capacity; or, as an alternative,

one of the many systems of high pressure filtration may be adopted and thus the necessary area largely diminished, since under a head of about 15ft. a high pressure filter may have a capacity of 50 to 60 times as great as that of an ordinary sand filter.

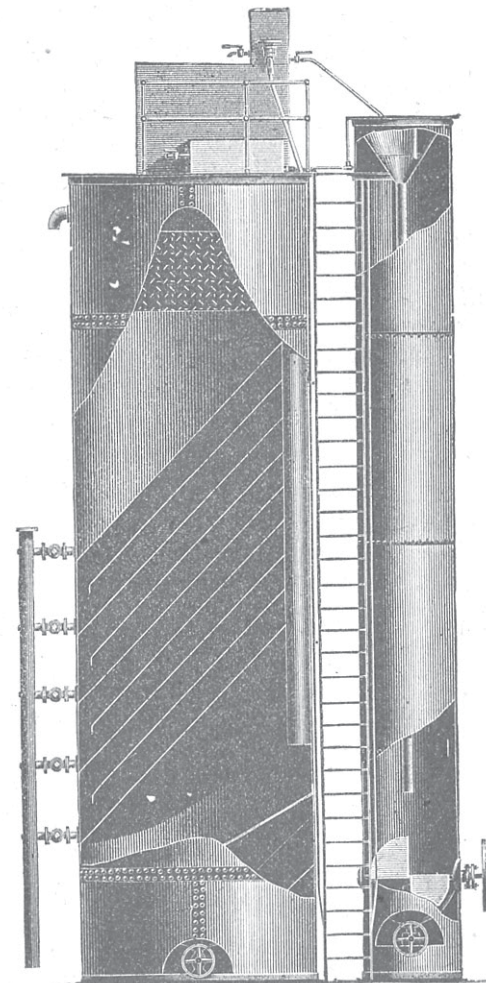


FIG. 14.—WATER SOFTENING APPARATUS.

It is, however, usually found most advantageous to adopt a so-called water softening apparatus when a sufficient ground area for settling tanks is not available.

These consist essentially of a series of small tanks for the preparation of the re-agents, and one or more larger tanks which serve for the removal of the precipitated carbonate. The large tanks are usually fitted internally in such a manner that they are practically equivalent to a very shallow reservoir of considerable area; and the precipitate having never more than a few inches to fall before it finds a resting place upon shelves placed for its reception, the clarification of the water is very quickly effected.

There are many forms of water-softening apparatus, a typical example being illustrated in Fig. 14 (p. 99). In this case the larger vessel (to the left) is the settling tank; the narrow tank serving for the preparation of the lime-water, and the small cisterns situated above these containing the soda solution. The lime-water is prepared by agitating freshly-slaked lime with water in the lower portion of the lime cylinder, the muddy liquid thus obtained gradually depositing the excess of lime and eventually passing out of the top of the lime cylinder as perfectly clear saturated lime-water.

The water which is to be purified is mixed with caustic soda solution and carbonate of soda or lime-water, in amount determined by the preliminary analysis, the mixture being led into the lower portion of the settling tank and gradually rising up the latter in the very circuitous manner rendered necessary by the internal divisions. The carbonate of lime is gradually deposited upon the inclined shelves, from which it is removed when necessary by means of the sludge cocks seen to the extreme left of the illustration.

By means of some such apparatus, water may be reduced from 16° hardness to 3°, at a cost of about a penny per 1,000 gallons.

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