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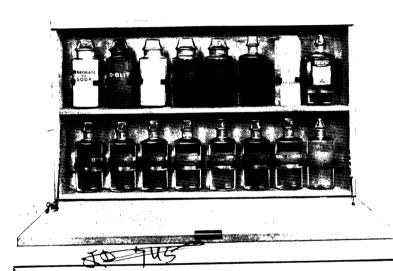
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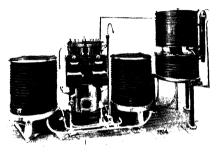
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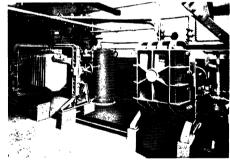


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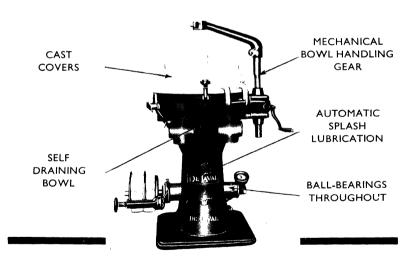
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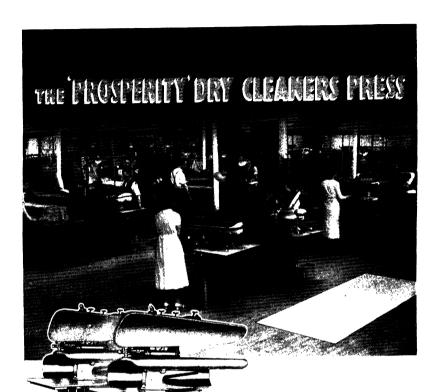
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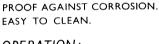
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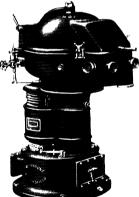
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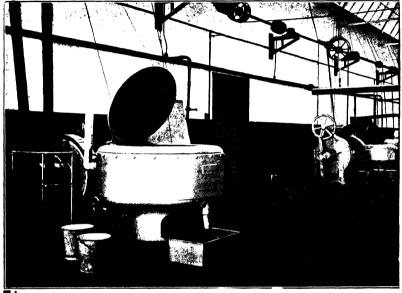
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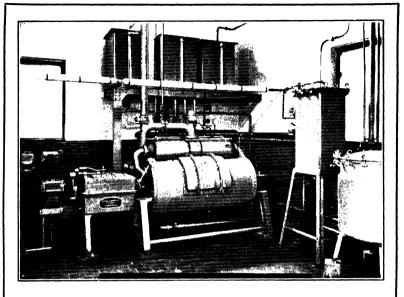
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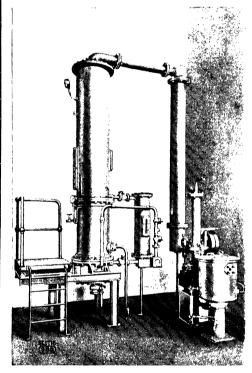
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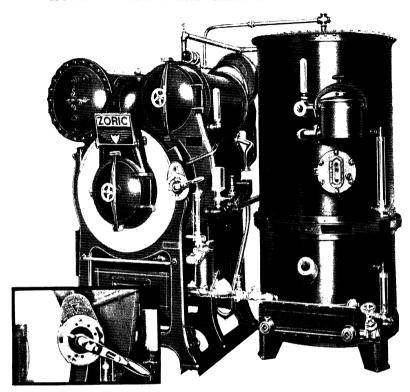
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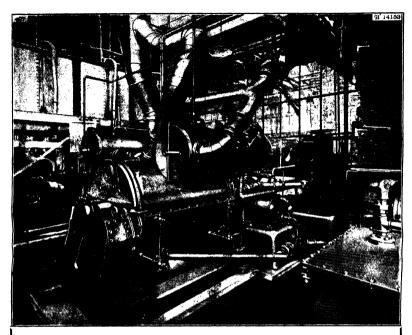
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#### **PREFACE**

No cookery book ever produced a good cook. No book can make a successful dyer, cleaner, or launderer.

Experientia docet is the only password to success. This book does not claim to open the door to success in what is one of the most skilful and certainly one of the most interesting and varied of the textile industries, but rather is written that the student, the trainee, and even the experienced dyer and cleaner may gain some understanding of the many apparently "rule of thumb" processes of garment treatment. Such understanding must definitely enhance the value of the experience gained in the practical school of the industry. While the emphasis must always be on the practical experience of the operative, there can be no doubt that guidance from a text-book is of great value.

During the past few years many changes have taken place in the dyeing and cleaning industry, of which the introduction of the non-inflammable solvents for dry cleaning is the most important. A considerable part of the book has been devoted to a careful study of the new solvents. The adoption of these solvents to practical dry cleaning has been a triumph for chemical engineers with the resultant decentralisation of the trade to a very large extent.

In the author's opinion, one of the most remarkable features concerning the dyeing and cleaning trade is the lack of knowledge of the characteristics of the textile fibres on the part of otherwise skilful operatives. This has prompted the allocation of the first chapter of the book to the consideration of the textile fibres, particularly those so often misnamed "artificial silk".

As new forms of textile fabrics appear on the market the difficulties of the dyer and cleaner will increase; it is therefore imperative that the successful dyer and cleaner should thoroughly understand the general trend in this direction. It is hoped that this book will supply some guidance, and as such be a contribution to the not too extensive literature dealing with the subject of dyeing and cleaning. If it does this it will fulfil its object.

WM. BROWN

London January, 1937

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#### Chapter I

#### THE TEXTILE FIBRES

THE production of textile fabrics from the various textile fibres entails a series of well-defined, highly specialised stages, each of which commands the skill, initiative, and intelligence of specially trained technologists. This specialisation is applied to the different textile fibres as well as to the different stages of manufacture. There are many examples of the various processes of manufacture being undertaken by separate firms, who are experts in that particular process on that particular fibre.

The acknowledged skill of the textile technologist offers an interesting comparison to that required of the garment dyer and cleaner. The garment dyer and cleaner who claimed that his was the most skilful of all the textile processes could certainly cite many factors in support of his claim. Not only is the dyer and cleaner called upon to process fabrics made from every known textile fibre and their mixtures, but also to handle these materials after they have been subjected to wear and other conditions which have considerable effect upon the original characteristics of the fabrics. The successful dyer and cleaner must have some knowledge of each textile fibre, its origin and manufacture into its various materials, and its physical and chemical characteristics. Because of this, the textile fibres are made the subject of the first chapter of this book.

Weaving was known long before writing, and consequently there are no records of early textiles. As far as can be determined flax was the first textile to be used by primitive man. Relics of flax garments have been discovered in association with utensils and weapons belonging to the Bronze Age. The cultivation of flax was extensive in the

time of Solomon. The Bible records in the first book of Kings, Chapter II, verse 28:

"And Solomon had horses brought out of Egypt, and linen yarn: the king's merchants received the linen yarn at a price."

The tombs of Egyptian kings have revealed linen fabrics probably 4,000 to 5,000 years old.

It is known that the Babylonian civilisation knew the uses of the wool fibre, so that it may be concluded that wool was the second textile to be employed by early mankind. The growing of cotton and its conversion into cloth have been known for many centuries. The original home of the silk industry was China, where the cultivation of the silk-worm was carried on during the dynastics of Yao and Shun, which flourished about 2500 B.C. The silk industry attracted the attention of Japan in the first or second centuries, and although silk was used in Rome about A.D. 500, it was not until the twelfth century that any attempt was made to introduce the silk-worm into Europe.

The history of cotton, linen, wool, and silk goes back far into the dim ages, and until recent times these fibres have been used almost exclusively in textile manufacture. The cultivation, spinning, weaving, and marketing of each have developed into highly specialised industries. In contrast to the older textile fibres, the advent of a new textile, loosely termed "artificial silk", is within living memory, but the adoption of the new fibre has been so rapid that a new industry as highly specialised as that associated with any of the other fibres has been created within the last twenty to thirty years.

The textile fibres resolve themselves into two distinct categories according to their origin. Those fibres which have been connected with plant life belong to the vegetable fibres, of which cotton, linen, jute, hemp, ramie, and sisal are well-known examples. In the other division, there are wool, silk, and hair belonging to the animal fibres, a classification which is obvious. The new textile "artificial silk", classified according to origin, would be included in the vegetable fibres, but because of certain well-defined differences in characteristics, it is usual to consider this fibre as a separate division.

#### THE VEGETABLE FIBRES

Of the vegetable fibres, cotton and linen are the most important, the remainder having only restricted applications.

#### COTTON

The cotton fibre is obtained from the white protective hairs which cover the seeds of the cotton plant. The cotton plant is a sub-tropical plant belonging to the botanical order of *Malvaceae*, or the mallow family. The botanical name of the plant is Gossypium, but there are many varieties of Gossypium, each with a geographical significance. Thus, Gossypium Barbadense includes the well-known Sea Island cotton valuable because of the length and strength of each individual fibre.

The cotton plant grows in well-defined countries where the climate is warm and humid. Thus the cotton areas are the sub-tropical states of America, India, Egypt, and the West Indies.

The cotton fibres are exposed when the seed-pods burst on ripening, and show the seeds embedded in a white fluffy ball resembling cotton wool. It is interesting to note that the ancient writings, which show that the use of cotton was known in India about 1500 B.C., describe the fibre as "tree wool". This term is still used in the German Baumwolle, which, translated literally, means "tree wool". The cotton is picked when the ripening stage has been reached, and in this condition is known as seed cotton. Seed cotton contains about two-thirds by weight of seeds, and the first process in the manufacture of cotton has the object of removal of the seeds, and is carried out in a machine called a cotton gin. Here the seed cotton is subjected to a somewhat harsh combing treatment which draws the fibres through openings too narrow to pass the seeds. The separated seeds find important applications in the manufacture of margarine and certain types of soap.

The ginning of the seed cotton is usually undertaken within the growing areas, after which the cotton is baled for export.

The stages of manufacture transforming the raw cotton first into yarn or thread, and finally into cloth and the many fabrics, are beyond the scope of this book, but it may be noted that the machinery connected with cotton manufacture is the practical outcome of inventions by many brilliant men, such as Hargreaves, Crompton, and Ackwright. The story of these men and their inventions is one of romance well worth reading.

#### CHARACTERISTICS OF COTTON

Seen under the microscope, the cotton fibre appears as a twisted ribbon. Cotton may be said to be curly, and this makes it easy for

the fibres to hold together, and to be twisted together and drawn out into fine long threads. In the raw state, raw cotton may have 1,100 to 1,600 fibres side by side in the space of one inch. The more fibres to the inch, the finer the cotton.

The length of the fibre or staple may vary from  $\frac{3}{4}$  inch to  $2\frac{1}{2}$  inches, Sea Island cotton being the longest. The number of twists may be from 300 to 800 in one inch of fibre.

The ribbon-like structure has thick rounded edges, but a cross-section of the fibre shows that it is really a collapsed tube or cylinder having an inner space or canal. This lumen, as the inner canal is termed, is of importance in that it enables the fibre to absorb liquids by capillary attraction, which causes the difficulty often experienced in rinsing out the last traces of alkaline or acid solutions. The spiral character makes it possible to spin exceedingly fine yarn, and also accounts for the elastic character of calicoes compared with linen, the fibres of which are stiff and straight. The microscopic appearance of cotton fibres serves to distinguish them from the other textile fibres.

Chemically cotton consists almost entirely of a compound known as cellulose. When in the raw state, the fibre is coated with a waxy covering, which is removed during the manufacture by boiling with a solution of caustic soda. The manufactured cotton may be taken as of pure cellulose. Cellulose belongs to the class of chemical substances known as carbohydrates, to which class belong starches and sugars.

The reactions of cotton with the various chemical reagents are highly important to the dyer and cleaner and launderer. Cotton will withstand fairly high temperature without decomposition, providing the time of heating is not prolonged; but it must be fully realised that there are very definite limits to the amount of dry heat to which the fibre can be submitted without permanently injuring it. When cotton is subjected to a temperature of 160°C., the cellulose loses water and becomes what might be termed "bone dry", accompanied by a definite weakening of the fibre. This fact has an important bearing on finishing processes where high temperatures are employed; for example, singeing, calendering, and other finishing processes. Below this temperature the cotton may lose slightly in strength and elasticity, but it quickly regains the moisture and strength on exposure to air. Above 250°C., cotton turns brown, or is scorched, from which there is no regain

either of moisture or strength. As is well known, cotton when ignited burns freely in air, leaving practically no ash.

Under normal conditions cotton may be said to be unaffected by cold or boiling water chemically, but may undergo certain physical changes. Boiling with water causes the fibre to become slightly plastic, a property which is made use of in certain finishing processes.

In the reactions caused by acids the vegetable fibres are in marked contrast with the animal fibres. In general, the mineral acids have a marked tendering action on cotton if allowed to dry on to the material. After any process involving the use of mineral acids, such as sulphuric, hydrochloric, or nitric acid, cotton fabrics must be thoroughly rinsed to ensure that the final traces of acid are removed. This will be readily understood by anyone, such as the dyer and cleaner and launderer, who frequently experience trouble due to the spilling of accumulator acid on to garments or articles sent to them for treatment. The extent of the damage to the cotton fibre depends upon the strength and temperature of the acid. The strength of the acid solution should not exceed 2%, nor the temperature 25°C. or 70°F. The tendering action of the mineral acid upon the cellulose produces a compound known as hydrocellulose. Hydrocellulose has no tensile strength, so that complete disintegration of the cotton takes place and the fibre becomes nothing more than a white powder.

This action of mineral acids upon cotton is made use of in the separation of cotton from mixtures with woollen rags or loose wool. The mixture fabrics are treated with sulphuric acid and after hydroextraction are dried at fairly high temperatures. When dry, the cotton portions, having been converted to hydrocellulose, crumble to dust and are removed mechanically, while the woollen portion remains intact. This process is known as "Carbonisation".

The action of mineral acids in concentrated form offers some slight contrast to that of the dilute acids. Concentrated sulphuric acid dissolves the cotton fibre, forming a thick syrupy liquid which, when diluted with water, precipitates a starchlike substance known as amyloid. This reaction is employed in the preparation of vegetable parchment. Unsized paper is rapidly passed through concentrated sulphuric acid, thoroughly washed and dried. This causes a layer of gelatinous amyloid to be formed on the surface of the paper, which, on pressing and drying, gives a tough membraneous surface to the

paper resembling true parchment. The treated paper becomes grease and waterproof, and has a considerably increased tensile strength. It is interesting to note that certain grasses are treated in this way to produce artificial horsehair.

Strong nitric acid has quite a different effect on cotton from that of the other acids. It completely decomposes the cotton, forming a variety of products. If the acid be heated, the cotton is converted into a compound known as oxycellulose, a friable substance which has an affinity for certain dyestuffs which will not in ordinary circumstances be absorbed by the fibre. The formation of oxycellulose occurs during bleaching and stripping processes, using such compounds as hydrogen peroxide, potassium permanganate, and, more than anything else, hypochlorite or chlorine bleaches, and the extent of the formation determines the extent of the weakening of the fibres during these operations.

If cellulose be treated with a mixture of strong nitric and sulphuric acids, a series of products may be obtained according to the conditions. These products are known as cellulose nitrates, which are dissolved by certain solvents. Thus, the highest nitrated product is known as the explosive gun-cotton; less nitrated cellulose dissolves in a mixture of ether and alcohol to give collodion and, when dissolved in camphor, to give celluloid. Nitro-cellulose is important as the starting-point for the preparation of one form of artificial silk, named after its discoverer, Chardonnet (see Artificial Silks, p. 33).

The solutions of the organic acids do not have any apparent tendering action such as results from the mineral acids. The non-volatile acids, such as oxalic, tartaric, and citric acids, appear to act in a manner similar to the mineral acids if allowed to dry on the fibre, but it is generally considered that the action is mechanical rather than chemical, and is due to the acids crystallising within the fibre and thus breaking the walls of the fibre cells. Acetic acid, being completely volatile, has no such action, and is therefore safe and the most suitable for any process where the use of acid is necessary. Tannic acid is readily absorbed from its solutions in water by the cotton fibre, a property which is the basis of the procedure of mordanting cotton with tannic acid and tannins for the dyeing and printing with basic colours.

The action of alkalies on cotton is in direct contrast to that of the acids. It has already been shown that acids in general have a distinctly

njurious action on cotton. The action of the alkalies under normal circumstances is comparatively safe. Dilute solutions of even the caustic alkalies are not injurious to the tensile strength of the cotton fibre under ordinary conditions, even when the solution is allowed to dry on the fabric. It must be emphasised that in the case of white materials there may be a tendency to become yellow if traces of alkalies remain on the fibre after drying.

Even at high temperature, such as at the boil, very dilute solutions of caustic alkalies will not tender cotton if all air be excluded. When air is present, alkaline solutions produce a reaction very similar to that of the acids in that hydrocellulose may be formed with the result that the fibre is seriously injured. The prolonged action of alkalies in the presence of air is an important one to bear in mind in the operations of bleaching, dyeing, or mercerising.

Concentrated solutions of caustic alkalies have a distinct action on the cotton fibre. When cotton is treated with strong caustic soda solution, the twisted ribbon-like fibres become swollen, and as a result appear as smooth cylindrical semi-transparent fibres, possessing a remarkable lustre. There is considerable increase in weight and tensile strength, accompanied by a relative amount of shrinkage. At the same time, the treated cotton exhibits an increased affinity for dyestuffs. This reaction was noted and first patented by a Lancashire calico printer called Mercer, a name which is perpetuated in the modern process of mercerisation. The date of Mercer's patent was 1850, but it was only within recent times that it was discovered that if the fabric being treated is held under tension, not only can the shrinkage be counteracted, but a higher lustre is obtained. Without tension the effect is likely to be uneven, but under tension when shrinkage does not occur, a very smooth surface results, and the treated fabric takes on an almost silk-like lustre. The action of the alkali is very rapid. and the mercerisation is complete in a few minutes.

The mercerisation process is carried out in two ways. In the one the cotton cloth is held in tension during the whole of the operation, while in the other the tension is applied after the caustic soda treatment, but before the caustic has been rinsed out. If the tension is applied after rinsing the increase in lustre is not obtained. Mercerisation was at first only applied in order to produce crimp effects on cotton cloth according to distinct patterns. Certain preparations can

be printed on to the cloth so that the cotton is protected from the effects of the caustic or, to use the technical term, "resists" the effects of the caustic. By printing a resist of albumen or casein in stripes, and then passing the cloth through the mercerising bath, the caustic can only effect the cotton between the printed stripes. Shrinkage takes place between the stripes and causes the protected portions to pucker, thus producing the well-known crinkle or seer-sucker effect. It must be pointed out here that, originally, seer-sucker meant a silk fabric with flat and puckered stripes alternating across the fabric, but is now understood to apply to cotton dress fabrics of the better quality crimp styles.

Caustic potash solutions are considered by some investigators to give better lustre than those of caustic soda, and that the amount of shrinkage is not so great. It must be borne in mind that caustic soda is much cheaper than caustic potash, and it is doubtful whether the slight additional advantages are sufficient to justify the extra expense.

The strength of the caustic soda solutions employed during mercerisation may vary from 15°Tw. to 80°Tw., although investigations have shown that solutions of 60°Tw. appear to give the maximum effect. Solutions even as weak as 1°Tw. cause a certain amount of shrinkage, accompanied by an increased affinity for dyestuffs.

The reactions occurring during the mercerisation of cotton were studied by Hubner and Pope, and their observations have been published in the *Journal of the Society of Chemical Industry*, 1904, p. 404. The changes in the appearance of the cotton during the process were noted as follows:

	St	rength of		
	caustic	$soda \ solution$		Effect
То	15° Tw			No apparent change.
,,	16° Tw	. to 18° Tw.		Slight, but incomplete twisting.
,,	20° Tw			Initial untwisting followed by slow un-
				coiling of the twist.
,,	26° Tw			Rapid and slow uncoiling become one,
				lasting five seconds.
,,	40° Tw		••	Untwisting and uncoiling take place together.
,,	60° Tw	. to 80° Tw.	• •	Swelling precedes untwisting.

In his original process, Mercer contended that the cotton underwent a definite chemical change, and it has now been shown that the cellulose forms a loose chemical compound with the caustic soda known as alkali cellulose. It must be emphasised that the chemists who have conducted research in this direction are not in complete agreement as to the exact nature of the combination between the cellulose and the alkali, although in certain cases the experimenter has given alkali cellulose a definite chemical formula.

It is agreed, however, that the cellulose is capable of absorbing definite quantities of caustic soda from the mercerising bath. The rinsing process after the mercerisation is thought to replace the absorbed caustic soda with a corresponding amount of water to produce a compound cellulose hydrate. The alkali cellulose is a very important substance, as will be shown in the section dealing with artificial silk.

Examined under the microscope, mercerised cotton exhibits considerable differences from ordinary cotton. The flat ribbon-like structure is replaced by a smooth cylindrical fibre showing a more or less circular cross-section. The degree of mercerisation determines the extent of the removal of the twist, so that it may be measured by the loss of twist and irregularities in the treated fibre. The complete loss of the twist of the fibre is a rare occurrence.

Reagents other than the caustic alkalies have the property of producing a mercerisation effect on cotton. When patenting his process, Mercer described the mercerising effects obtained on cotton by the use of reagents other than the caustic alkalies. The patent shows that concentrated sulphuric acid, zinc chloride, and phosphoric acid could be used as mercerising agents. Certain salts of the metals, such as tin chloride or calcium chloride, have a mercerising effect of varying degree. In no case is the result obtained as effective as with caustic soda.

When treated with a solution of copper oxide in ammonia (known chemically as Schweitzer's reagent), cellulose is dissolved very slowly. The rate of solution can be increased if the cellulose has been treated previously with a solution of caustic soda strong enough to cause the fibres to swell. The solution obtained has two commercial applications, one for the production of cuprammonium silk, or Pauly silk, and the other for the production of Willesden canvas. The production of Pauly silk will be referred to in the paragraphs devoted to artificial silks.

For the production of Willesden canvas, the cotton fabric is treated with a solution of ammoniacal copper oxide, so that a partial gelatinising of the surface fibres occurs. The surface film is fixed by a process of hot pressing. In this way the canvas is coated with a substantial film which is waterproof and resists the effects of mildew and insects. It will be readily understood that this waterproof film makes it extremely difficult to wash or dye Willesden canvas.

Cotton may also be dissolved by concentrated solutions of zinc chloride after prolonged treatment at 100°C. As in the case of the copper-ammonium solution, the rate of solution is increased if the cotton has been treated with caustic soda, and complete solution is obtained at ordinary temperatures. The product of this treatment has many applications in industries. One of the most important is that of the preparation of vulcanised fibre. Vulcanised fibre is prepared by the treatment of paper (paper may be taken as a form of cellulose) with a concentrated solution of zinc chloride until the fibres are gelatinised. The gelatinous mass obtained is pressed and moulded into compact masses. Vulcanised fibre is quite hard with the consistency of horn, but by the addition of certain substances, such as glycerin or glucose, the product can be rendered pliable. As is well known, this product is used extensively as a substitute for leather. Nitration causes the fibre to become waterproof. Another application is for the production of a type of artificial silk and of filaments for incandescent lamps.

The reactions of the cotton fibre towards the various reagents employed in stripping of dyes are highly important. Those stripping agents, which are chemically classified as reducing agents, appear to have little or no damaging effect on cellulose. On the other hand, the oxidising agents readily convert cellulose into oxycellulose. The production of this compound has been shown in the description of the action of nitric acid on cotton. Reducing agents used in bleaching or stripping include sodium hydrosulphite and its zinc compounds, which are marketed under a series of trade names: formusol, oblit, hyraldite, hydros. Other reducing agents are sulphurous acid, sodium bisulphite, titanous chloride. Although titanous chloride is included among the reducing agents, it must be emphasised that this compound is used in relatively strong acid solution, and consequently tendering may arise from the action of the acid.

The oxidising agents include potassium bichromate with acid (sour chrome), potassium permanganate, and chlorine bleaches.

Cellulose has a very slight reaction with Fehling's solution—(Fehling's solution is made by mixing two solutions, one containing copper sulphate, the other Rochelle salt and caustic soda)—but oxycellulose has an appreciable effect. This effect is employed to estimate the amount of damage caused by bleaching the cotton or other cellulose fibres during manufacture or laundering. The result of the reduction of the Fehling's solution is the precipitation of an amount of copper equivalent to the amount of oxycellulose present in the fabric. The precipitate of copper can be determined accurately and a series of values obtained which are known as copper values. The following are examples of copper values:

Artificial silk (Glanzstoff)		 	1.1
Surgical cotton wool		 	1.6
Bleached mercerised yarn		 	1.0
Bleached sulphite wood pulp		 	3.8
Parchment paper		 	4.2
Hydrocellulose		 	5.2
Bleached cotton rag		 	6.5
Filter paper after bleaching pow	der	 	7.0
Over bleached wood pulp		 	19.9

Cotton exerts practically no attraction for the metallic salts, although in certain cases the metallic hydroxide may be loosely held by the fibre. For example, it has been shown that when treated in a solution of barium hydroxide or the basic salts of lead, zinc, tin, aluminium, iron, and chromium, cotton absorbs an appreciable amount of the basic oxide. The acid reacting salts, of which magnesium chloride is an example, may cause tendering of cotton, as in the case of the mineral acids, when allowed to dry into the material.

Cotton in contact with a flame readily takes fire, the flame spreading rapidly. This characteristic serves to distinguish cotton (and the other vegetable fibres) from the animal fibres and from the "cellulose acetate" artificial silks, but, at the same time, is a disadvantage with curtains, hangings, and garments made of cotton. The impregnation of the fibre with certain metallic salts causes cotton materials to become

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fireproof. Compounds of ammonia have been frequently employed to fireproof cotton. A mixture of ammonium chloride (sal ammoniac) and ammonium phosphate in equal proportions is quite effective, but as these salts readily dissolve in water, the fireproofing is removed if the material is washed or treated with water in any way. Cotton can be rendered permanently fireproof by precipitating oxide of tin on the fibre. The process has been the subject of several patents. The cotton is first impregnated with a solution of sodium stannate and then dried. A second treatment with a solution of sulphate of ammonia is given and the cotton again dried. Finally, the fabric is thoroughly rinsed. The reaction between the sodium stannate and ammonium sulphate causes the fibre to be impregnated with stannic oxide, while ammonia is liberated and sodium sulphate dissolved in the rinsing water. It is claimed that the process does not impair either the feel or strength of the treated fibre.

#### LINEN

Linen is obtained from the stem of the flax plant. It is considered that linen was the earliest vegetable fibre. Egyptian mummy cloths, probably 4,500 years old, are of linen. Flax was a product of Egypt in the time of the Pharaohs. Linen ranks next to cotton in importance as a textile fibre.

While cotton is a seed fibre, linen is known as a bast, or stem fibre. It is the fibrous portion of the stem held together in the plant by woody tissue and plant membranes.

The flax plant, although grown almost all over the world, is cultivated extensively in Russia, Belgium, Italy, Ireland, and America. The seeds of the flax plant are used for the preparation of linseed oil. The flax of the northern climates provides the better fibres for textile purposes, while tropical flax produces the better seeds.

The leaves and seeds of the flax plant are removed from the harvested stalks by a somewhat drastic combing operation known as rippling. The next stage is the separation of the fibres from the woody portions of the stem. This separation process, known as retting, causes the decomposition of the vegetable tissues of the stalks, after which the linen fibres are ready separated mechanically.

For the retting process (sometimes called rotting, a term which probably describes the process literally), the gathered plants, after

rippling, are packed in bundles, placed in crates, and steeped in water. Retting may be carried out in several ways: in stagnant water, in running water, by exposing the flax to the action of dew in open fields, or by chemical means.

During retting, fermentation sets in and results in the formation of soluble organic matters. The fermentation takes place in two stages: in the first of which the liquor is acid, and in the second when the condition of the liquor changes to alkaline solution. Since the presence of acid retards the progress to the second stage, sodium bicarbonate is added to neutralise any acid which is formed. Sodium carbonate is not suitable, as this not only softens the tissues, but also retards the progress of the fermentation. In stagnant water retting there is a danger of over-retting, which is detrimental to the subsequent fibres. To prevent this the crates are lifted out of the liquor and the flax spread out on the fields for 5 to 10 days. This is known as grassing. The fibre obtained by stagnant retting is of a somewhat dark colour.

With running water retting, the crates are steeped by being lowered into running streams. After a few days the flax is taken out of the crates, allowed to dry, and then returned to the crates. This process may take from 10 to 20 days. The flax obtained by running water retting is lighter and of better quality than that obtained by any other method.

Dew retting consists of about ten weeks' exposure to dew and atmosphere, but the flax obtained is often of a dark, uneven colour. Retting by chemical means is not in general use. Chemical retting may be carried out by submitting the flax plant to alternate treatment with 3% hydrochloric acid solution and an alkaline solution, or to treatment with warm water for a few days, or to high-pressure steam.

After retting, the fibres freed from adhesion to the woody part of the stem are separated by means of many mechanical operations, which remove the woody and glutinous matters. Thus, squeezing through rollers removes the soft glutinous portion, passing through fluted rollers breaks the woody matters, beating or scutching removes them, and, finally, combing or heckling, during which the flax is passed through a series of graduated combs, so that all the fibres are placed parallel and in a condition ready for spinning.

In the case of cotton, the longest fibres obtained have a length of about 2 ins., but the linen fibre may have a length up to 5 ft. Good

linen fibre should average 20 ins. in length and be free from fibres less than 12 ins. in length.

#### CHARACTERISTICS OF LINEN

Under the microscope, linen appears as a composite fibre, being composed of long cylindrical vessels or filaments. These filaments end in characteristic cone-like points. The cell walls, in comparison with those of cotton, appear very thick, with a correspondingly small inner canal or lumen. The lumen in certain cases is so small as to appear only as a dark line. A distinguishing feature of the linen fibre is the more or less regular joints or nodes which give a bamboo-like appearance. In many instances this bamboo structure is the only certain test for linen in contra-distinction to cotton.

The linen fibre is stiffer and straighter than the cotton fibre. It is also of greater lustre and of greater tensile strength than cotton. Linen is a better conductor of heat than cotton, and in consequence linen goods always feel cooler than those of the other material. The greater tensile strength of linen is dependent upon the exact degree of retting of the flax. Over-retting causes considerable deterioration in the strength of the fibre obtained.

The amount of moisture retained by the linen fibre is similar to that retained by cotton, that is, from 6% to 8%. This percentage of moisture is contained approximately in all the vegetable fibres.

Chemically, linen is similar to cotton. The ultimate filaments which compose the linen fibre have been shown to be of pure cellulose, as in the case of cotton. These filaments are held together by a kind of resinous gum known as pectose, and it may be said that the flax fibre is a chemical combination of cellulose and pectose. The pectose can be separated by boiling with 2% solution of caustic soda and then removing certain fatty and waxy compounds with boiling alcohol. The cellulose obtained from flax is chemically indistinguishable from that obtained from cotton. It therefore follows that the chemical reactions of linen are similar to those of cotton, any difference being of degree only.

Linen is attacked by mineral acids if these are allowed to dry into the fibre from lack of proper rinsing. Linen is tendered by exposure to sunlight due to the formation of oxycellulose as with cotton. Oxycellulose is also formed when linen is bleached with any of the oxidising

reagents. When treated with Schweitzer's reagent, the linen fibre becomes swollen, but does not dissolve completely. The swelling shows under the microscope as irregular, characteristic blisters.

In its reactions towards mordants and dyestuffs, linen exhibits much less affinity than cotton, and in consequence its treatment in dyeing is correspondingly more difficult. In general, however, the dyeing and laundering of linen is for all practical purposes the same as for cotton. Caustic soda solutions do not produce a mercerising effect on linen, but may, in the case of hot solutions, cause disintegration of the fibre. Linen is readily disintegrated by the action of boiling alkaline solutions and solutions of oxidising agents. The bleaching of linen after retting is a somewhat complicated process and is much slower than in the case of cotton. Linen is marketed in various stages of bleaching: unbleached, quarter bleached, half bleached, and full bleached. The characteristic specks which often appear in unbleached linen are due to woody particles left after retting.

#### JUTE, HEMP, AND RAMIE

The remainder of the vegetable fibres are of much less importance than cotton or linen. Jute is a bast or stem fibre of a plant cultivated in India and the East Indian islands. The stalks of the plant are retted in slow-running streams, as in the retting of flax, followed by scutching to remove woody matter. Jute fibres are specially suitable for spinning because of their length and fineness. In addition, jute has a silk-like lustre. On the other hand, the fibre tenders on keeping, and the colour darkens. It rapidly disintegrates with bleaching, and on this account cannot be given a full bleach. Coarse jute is employed in the production of sackcloth, matting, paper, twine, and small ropes. Finer jute may be used in carpets, in plush fabrics, and for adulteration with more valuable fibres. The fibre has an affinity for the basic dyestuffs without mordanting.

Chemically, jute consists of a combination of cellulose with a compound known as lignin. It is, therefore, a modified form of cellulose known as ligno-cellulose.

Hemp is obtained from a shrub-like plant grown in many temperate and tropical countries. (The seeds of the plant are narcotic and are used for the preparation of the Indian beverage "Hasheesh".) The preparation of the fibre follows closely the methods used with flax.

The plants are subjected to rippling, dew retting, breaking, scutching, and heckling. The hemp fibre produced in Italy is of a light colour, soft, pliable, with a characteristic lustre, and may be used as a substitute for flax. Hemp is used for sacking, sailcloth, rope, string, and ships' hawsers. It is durable and does not rot with water, as in the case of jute. The chemical reactions of hemps are those of a ligno-cellulose.

Ramie and China grass are frequently confused, in spite of their separate origins. They are obtained from two species of nettle plant, the one grown in tropical countries, and the other in more temperate climates. The fibres obtained are very similar and consequently may be considered alike for textile purposes. The isolation of the fibre is difficult, since retting is not practicable. Retting results in the separation of the ultimate fibrils, which become hopelessly entangled. For the preparation of the fibre, the outer bark is removed from the stalks by mechanical means. The gummy matters, which hold the fibres together, are removed by treatment with soap solutions under pressure. Ramie fibre is naturally colourless, with high lustre, and resistance to mildew. It is much more durable than jute; its tensile strength is three times that of hemp, but it has the disadvantage of being difficult to spin.

The fibre is a composite one, consisting of many fibrils, and is difficult to distinguish from linen when examined under the microscope. Ramie fibre gives the chemical reactions of pure cellulose. It finds application as a substitute for linen and mohair.

The other vegetable fibres used as textiles include straw and wood chip, and certain types of grass. Their applications are very limited, but, in general, they may be considered as being of various forms of cellulose, and as such react similarly to the more important vegetable fibres.

#### PAPER

During the War period (1914-18) use was made of paper in the manufacture of textile materials, particularly in Germany and Japan. Although paper fabrics cannot be successfully treated in dyeing and cleaning, owing to the complete loss of tensile strength when wet, the relation of paper to the vegetable fibres is such that the manufacture of paper may be described at this stage without being considered out of place. Paper is chemically pure cellulose, so that any difference between the cellulose of paper and that of the textile fibres is a physical

one. In the textile industry, the longer the fibre the better the product, but in making paper the fibres must be short. For this reason textile waste may be used. Paper may be made from any of the vegetable fibres, but, generally, linen, wood pulp, jute, and certain grasses or straw are used in a state of fine division. The textile waste is subjected to a series of operations with the object of separating the cellulose content as individual fibres. The finely-divided fibres are treated with size and filling materials and beaten into a pulp. The pulp is then dried over heated rollers and pressed to give paper.

#### THE ANIMAL FIBRES

#### WOOL

While the hair-like coverings of many animals may be included under the term "wool", it is generally accepted as strictly applying only to the covering of sheep. For practical purposes the soft hairs of certain animals may be considered as wool; for example, cashmere from the Cashmere goat of India; mohair from the Angora goat of Asia Minor; alpaca from the llama and alpaca of South America; and the hair of the camel. As is well known, sheep are bred and reared entirely for the production of wool. The greater portion of wool in commerce is fleece wool, which is obtained by shearing from the living animal. The character and quality of the wool depends upon many factors, which include the breed of the sheep and the part of the body from which the wool is taken. The finest wool, known as lamb's wool, is the clippings from the lamb about eight months old. The classification of the wool fibre is according to the length of fibre; more than 11 ins. to 2 ins. in length long staple, less than 11 ins. short staple. Pulled wools are obtained from the fleeces of dead sheep. The wool is removed from the skin by chemical processes. Such wool is inferior to fleece wool. Yet another form of wool may be termed "recovered wool"; this form is known by the rather derogatory name of "Shoddy", but it must be emphasised that certain grades of re-worked wool or shoddy are of better quality than some types of "virgin" fleece wool. The employment of mineral acids to remove the vegetable fibres from wool rags has been described under "Carbonisation" of Cotton (p. 11). This "shoddy" consists entirely of wool. In the best qualities of shoddy the fibres may have an average length of 1 in.

The raw wool, as sheared from the sheep, contains many impurities, which must be removed by a scouring (or washing) process in order to obtain the pure fibre. These impurities consist of relatively large amounts of grease, dried-up sweat, and dirt. The grease is considered to act as a covering to the growing fleece, protecting the wool from mechanical injury and preventing the matting of the fibre. The grease or wool fat forms a waterproof coating, being insoluble in water, but is soluble in certain volatile solvents, such as benzene, ether, or carbon bisulphide. On the other hand, the wool fat is readily emulsified during a close-controlled washing process. The dried-up sweat, or suint, consists of potash compounds with various fatty acids, and is readily soluble in water. The suint is removed after the emulsification of the wool grease. Dust, sand, and burrs make up the third impurity. The adhering dust and sand are removed in suspension during the scouring process, while the vegetable burrs are removed by carbonising (see p. 11).

#### THE CHARACTERISTICS OF WOOL

The wool fibre has a characteristic appearance when observed under the microscope. It appears as a rod-like structure covered with scales. The arrangement, shape, and size of these scales vary considerably with the origin of the wool. These scales play an important part during any wet process, as it is considered that the interlocking of the scales produces shrinkage in woollen fabrics. It will be readily seen that friction or rubbing together of the fibres must be reduced to an absolute minimum in order to avoid this interlocking.

The wool fibre has three distinct portions: the scaly covering, or epidermis; a layer of fibrous substance or cortical tissue, on which the strength and elasticity of the fibre depends; the marrow of the fibre, or medulla; the character of any one or all of these portions may vary with the origin of the fibre. The medulla may also contain pigment matter, to which the wool owes its natural colour.

Wool fibres may be said to be "wavy" rather than "curly", and this waviness varies considerably with the quality of the wool. Again, the tensile strengths of wool exhibit wide variations, but it is interesting to note that cotton is considerably stronger than the majority of wools.

Chemically, wool is classed as keratin, a protein-like substance. The proteins are a class of chemicals connected with living animal

tissues. The exact chemical structure of keratin has not been determined, but it has been established that it contains a portion which has a basic character and another portion which has an acidic character. This dual character explains why wool has an affinity for basic dyestuffs and chemicals, on the one hand, and an affinity for acid dyestuffs and chemicals, on the other. The basic portion is closely allied to ammonia.

Wool is very hygroscopic, which means that it is capable of absorbing and retaining large quantities of moisture without feeling damp. When heated at 100°C., wool loses this hygroscopic water, becoming bone dry. On cooling much of the moisture is re-absorbed from the atmosphere, although, if the high temperature has been unduly prolonged, the wool does not completely return to its original degree of softness. In moist heat, at temperatures approximately 100°C., wool becomes plastic and can be shaped or stretched, such shape being maintained when the wool cools. This property is the foundation of the many finishing processes on wool yarn, piece goods, and even garments. In contrast to cotton, wool only burns with difficulty, emitting the peculiar smell associated with burning feathers, and leaving a black, charcoallike bead. The flame is not transmitted along the fabric as in the case of the vegetable fibres. The flame test is generally employed to distinguish wool from cotton and linen.

Normally, wool is unaltered by cold or hot water, but prolonged boiling tends to decompose the fibre, which would cause loss of strength and elasticity. This property must be considered when it is necessary to strip or re-dye wool garments.

Yet another contrast with the vegetable fibres is shown by the reactions of wool with the various acids. The dilute mineral acids have little appreciable effect on the wool fibre, so that the strength, lustre, and felting properties are unaltered. Yet it has been shown that the acids are readily absorbed and tenaciously held by wool. When wool has been treated with dilute sulphuric acid, some acid is still retained, even after repeated rinsing in boiling water, until the rinse-water shows no trace of acid. Such wool can be dyed with various acid dyes without the addition of acid to the dye-bath. It is evident that the wool is able to form a chemical combination with the acids. If the acid treatment is carried out at high temperatures and prolonged, a harsh feel may be imparted to the fibre. Since

hydrochloric acid is not retained by the wool to the same extent as sulphuric acid, the handle is much softer after treatment. Because of this, the use of hydrochloric acid is often preferred for the process of carbonising. Chromic acid is also readily absorbed by the wool fibre, and this property is the foundation of the mordanting of wool by means of acid solutions of chrome (sour chrome).

The reaction of nitric acid solutions on wool is markedly different from those of the other mineral acids. Except when the acid is below 4°Tw. strength and at low temperature, nitric acid turns wool a yellow shade. The formation of this yellow shade is similar to the yellowish brown stains produced on the skin by nitric acid. The yellow colour is due to the conversion of the fibre into a compound known as xanthoproteic acid.

In solutions of nitrous acid (sodium nitrite and hydrochloric acid), wool is diazotised and then can be developed to give rise to a range of shades. The diazotisation of wool increases its affinity for the basic dyes, but decreases its affinity for the acid dyestuffs (for diazotisation of dyes, see p. 196). As is usual with organic substances, the concentrated mineral acids destroy and dissolve the wool fibre. Wool readily absorbs the organic acids, oxalic, lactic, tartaric, and acetic, but tannic acid shows an exception. Tannic acid is only absorbed from hot solutions, and is only fixed by an after-treatment with a metallic salt such as tartaric emetic or stannous chloride. Tanned wool has the peculiar property of resisting dyeing with the acid, direct and mordant dyes, while the attraction for basic dyestuffs is obviously increased.

In contrast to its reaction with acids, wool is highly sensitive to the reaction of the alkalies. While the effect at low temperature may be slight, even extremely dilute solutions of the caustic alkalies will cause permanent injury to the fibre, producing loss of handle and tensile strength. A 5% solution at 100°C. will completely and rapidly dissolve wool. It will be seen that soaps containing free alkali are definitely unsuitable for woollen articles. The alkaline carbonates (e.g. soda ash), although not as injurious as the caustic alkalies, exert a destructive action on wool, especially if used above moderate temperatures.

The so-called mild alkalies (borax, sodium bicarbonate) have much less action and therefore can be used in laundering of woollen articles

which are too dirty to be treated with soap alone. The oxidising agents usually employed for wool bleaching are hydrogen peroxide, sodium perborate, and potassium permanganate. Solutions of hydrogen peroxide or acidulated solutions of sodium perborate may be used without deleterious effect on wool. (It must be pointed out that sodium perborate contains caustic soda, which must be neutralised in order to prevent damage to the fibre; hence its use in acidulated solution.) Bleaching of wool by means of potassium permanganate solutions may be safely carried out by careful control of the concentration and temperature of the solution. During the treatment the wool becomes a dark brown colour, due to the precipitation of the manganese oxide on the fibre. This manganese compound must be afterwards removed by treatment with sulphurous acid, sodium bisulphite, or oxalic acid. The result obtained is a good permanent white, but the oxidising action on the outer scales of the fibre produces a harsh feel.

The reducing agents are not harmful in themselves, and under normal conditions are valuable for bleaching and removal of stains from wool. The disadvantage arising from their use is that in many cases the oxygen of the air gradually oxidises the natural colouring matter to its original colour. A well-known example of this is the so-called "sulphuring" of blankets. In order to produce blankets of a good white colour, the wet blankets may be hung on a rail in the sulphur stove. The sulphur stove consists of a chamber in which sulphur is burned in order to produce sulphur dioxide. The sulphur dioxide is absorbed by the wet blanket and by chemical reduction of the colouring matter produces a good white. The effect is not permanent and a sulphured blanket will quickly become yellow.

The action of the chlorine bleaches on wool is extremely interesting. Except in very weak solution the hypochlorite bleaches first turn wool yellow then completely destroy the structure of the fibre; on the other hand, wool may be treated with weak solutions of hypochlorite under certain well-controlled conditions. It is found that the wool in these circumstances has undergone considerable alteration. It becomes somewhat harsh, loses strength, but increases in lustre and acquires a "scoop" like silk, while the affinity for dyes in general is greatly enhanced. But the most important effect is that the tendency to shrinkage and felting almost disappears. This action is the basis

of the process for the manufacture of "unshrinkable" woollen fabrics. Microscopic examination of the treated wool shows that the original scales of the fibre have been removed, either wholly or partially, according to the extent of the chlorination.

The mixing of chlorinated wool with untreated wool gives rise to a variety of fancy effects; for example, in dyeing, two colour effects may be obtained from the same dye-bath, since the chlorinated wool dves to a much deeper shade; or, in finishing, where the fabric may be "fulled", so that the untreated wool is caused to shrink while the chlorinated wool does not, producing crepon effects. the neutral metallic salts are inactive, wool is very reactive with salts of chromium, iron, copper, tin, and aluminium, which are acidic. When boiled with these solutions, the wool absorbs considerable amounts of the metallic portion, but the exact nature of the combination is not certain. It has been thought that the metallic oxide or hydroxide combines with the wool, but, in the case of copper, this can be definitely shown as not the case, as the colour of wool treated with copper salts does not give the colour reactions of copper hydroxide. The property of absorbing metallic compounds is the basis of the important process of mordanting wool as the preliminary treatment before dyeing with certain dyestuffs. These dyestuffs, which of themselves have no affinity for the untreated wool, produce extremely fast colours when dyed on mordanted wool.

If wool is left in a moist condition in a warm, closed place, mildew rapidly develops on the fibre. Mildew causes the fibre to become tender and eventually rot. The tendency to mildew is greater in wool than cotton, and is not dependent on the presence of size and starch, as in the case of cotton.

The minor hair fibres, such as mohair, alpaca, cashmere, and camel-hair, are similar to wool, any difference being of degree only. Many of them are coloured brown or black and are difficult to bleach white. Each hair fibre has the characteristic scaly appearance under the microscope.

#### SILK

The silk industry originated in China approximately 3000 B.C., and throughout all the ages silk has been prized for its beauty and lustre.

Silk is a thread excreted by a genus of caterpillar, "the silkworm", in forming its cocoon. This thread is the simplest textile fibre, but has the greatest strength and lustre of all the textile fibres. Silk may be divided into two classes: true silk, which is obtained from cultivated silk-worms, and wild silk from wild silk-worms. True silk is obviously the most important and valuable. Silk-worms are extensively cultivated in China, Japan, Italy, Greece, and south of France. The principal specie of silk-worm is known as the mulberry silk-worm, from the fact that the caterpillars are reared on mulberry leaves. The silk-worms, after hatching from specially selected eggs, feed upon the mulberry leaves until the rate of growth ceases. At this stage they begin to spin their cocoons. Silk is produced from glands, which communicate with a central orifice in the head of the caterpillar. The liquid excreted from the glands coagulates and solidifies in contact with the air to form the silk thread. (It is interesting to note that the appliances used in the artificial silk formation are an imitation of the spinneret of the silk-worm, and that there is a striking analogy in the coagulations of the natural and artificial silks.) When the cocoons are completed, they are collected and killed by boiling water, steam, or stoving at 75°C.

The thread is obtained from the cocoons by a process known as reeling. This consists of soaking a number of cocoons in warm water, so that the silk glue, which holds the fibres together, is softened. The threads from a number of cocoons are reeled by passing through a porcelain "eye", and through suitable guiders onto a reel, so that the threads are twisted together during the process. The product obtained is known as raw silk, or grege. Damaged cocoons are collected and subjected to fermentation. After a treatment with sodium carbonate, the cocoons are washed and dried. The dry cocoons are torn to shreds and the torn fibres combed, carded, and spun, as in the case of cotton. The product in this case is known as spun silk. Organzine is the name given to the thread obtained by combining a number of silk threads and twisting them together. Organzine is mainly used in weaving as warp. In the case of a few threads slightly twisted together, the result is known as tram silk, which is used as weft. (In weaving, the warp is the thread running the length of the fabric, while the weft is the thread running across the fabric.)

#### THE CHARACTERISTICS OF SILK

Under the microscope, raw silk appears as a double cylindrical rod with no cell structure. It consists of two fibres, held together by a glue or cement-like compound, known as "silk gum". Silk gum is readily removed by treatment with hot soap solution. This process goes by the name of "boiling off", and the liquor obtained is known as "boiled-off liquor". Boiled-off liquor has an important application as a levelling assistant in dyeing with acid dyestuffs. With silk dyeing it helps to preserve the lustre of the fibre. Raw silk, or silk in the gum, is a dull, dark-coloured, harsh fibre, but the removal of the gum reveals the true lustre and softness associated with silk. The microscope shows "boiled-off" silk as a smooth transparent rod-like fibre, apparently without cellular structure. The fibre is actually composed of a number of fibrils. These fibrils can be made more pronounced by treatment with certain chemicals. The separation of these tinv fibrils, following drastic or rough treatment, is the cause of the so-called "face" marks on silk.

Silk is very hygroscopic, retaining as much as 30% moisture, and still feeling dry to the touch.

Silk, especially in the raw state, has exceptional tensile strength and elasticity. Its tensile strength is nearly equal to that of a steel wire of the same diameter, while it may be stretched to increase its length by one-seventh. After boiling off, the strength and elasticity is reduced. The silk fibre may vary from 500 yds. to 1,500 yds. in length.

Silk is a non-conductor of electricity, and can be electrolised by friction. Because of this generation of electricity, silk is kept moist during manufacture in order to avoid danger of sparking. In dry cleaning with inflammable solvents, the treatment of silk garments calls for special care and attention. Raw silk consists of two chemical compounds—fibroin and sericin. Fibroin is the fibre proper, while sericin is the silk gum, or external covering of the fibre. The silk gum resembles gelatine in appearance; it is readily dissolved by solutions of weak soap or alkali solutions, or even in boiling water. Fibroin belongs to the same class of chemical compounds as the keratin of the wool fibre, i.e. it is a proteid. It therefore resembles wool in its many reactions.

Silk is extremely sensitive to heat. White silk, heated at 230°F. for 15 minutes, becomes discoloured and acquires a yellow colour. Under similar conditions, wool and cotton would be unaltered. On the other hand, silk will withstand higher temperatures than wool before its fibres are disintegrated. In contact with flame, silk burns with difficulty, as in the case of wool, emitting the odour characteristic of the animal fibres.

The effect of caustic alkalies is considerably less on silk than on wool. Dilute solutions of caustic alkalies have the main effect of deteriorating the lustre of the silk fibre. Silk is dissolved by strong caustic alkalies only when hot. The minor alkalies may be said to have no effect on the silk fibre, although it must always be borne in mind that prolonged treatment may cause "facing". Silk readily absorbs the dilute acids and retains them tenaciously. This causes an increase of lustre and imparts a property which is known as "scroop". Scroop is the well-known rustle of silk garments. It may be produced by treating the silk with dilute solutions of acetic, tartaric, or sulphuric acid and drying without rinsing out the acid. In contrast to wool, silk is capable of absorbing relatively large amounts of tannic acid, a property which is exploited in the weighting or loading of silk, and also in mordanting.

Concentrated sulphuric and the mineral acids dissolves silk when the immersion is prolonged. If the treatment is only for a short time, shrinkage and loss of lustre takes place. The extent of the shrinkage depends upon the concentration of the acid, the temperature and duration of the treatment. The reaction of concentrated hydrochloric acid in dissolving silk is fairly rapid, and is used in the removal of silk from admixture with wool and cotton. Nitric acid on silk produces a yellow coloration similar to that obtained on wool. It is peculiar, however, that the yellow colour is not developed if the acid is pure, being free from nitrous acid.

The organic acids have no disintegrating action on silk, except at high temperatures, and in such concentrated form, as glacial acetic acid or molten oxalic acid, which obviously would not be used in practice.

The chlorine bleaches in very dilute solutions have a similar effect on silk as on wool, in that the treated fibre shows an increased affinity for dyestuffs. This is used in producing two-colour effects from the

same dye-bath by mixing treated and untreated silk. In solutions such as are normally used on cotton the silk fibre is entirely destroyed. Hydrogen peroxide solution and the hydrosulphite solutions are recommended for cases where silk has to be bleached. Silk is dissolved by Schweitzer's reagent and ammonium nickel hydrate. Zinc chloride solutions and basic zinc chloride solutions dissolve silk to form a thick resinous solution, and since this reaction does not take place on cotton or wool, it serves to separate silk from mixtures with the other two fibres.

Towards the metallic salts, silk has a remarkable affinity, so that considerable increase in weight results. Since silk was originally sold by weight, the reaction is often employed for the purpose of increasing the weight of silk, or of making up for loss, which occurs during boiling-off and subsequent manufacturing processes. Solutions of tin chloride are used. The silk is immersed in the cold solution so that it absorbs about 10% of the tin weighting. If the tin is then fixed by a further treatment with sodium phosphates or sodium silicate, the silk is able to take up more tin, so that by repeated treatment as much as 100% weighting can be obtained on the silk.

Silk weighting, if carried beyond definite limits, causes deterioration of the silk. The strength of the fabric is much reduced. Weighted silk is rotted very quickly by perspiration, as every dyer and cleaner knows. Further, weighting of the silk often causes considerable deterioration during storage, so that silk may become rotten without having been subject to wear at all.

The typical example of wild silk is that known as Tussah silk. Tussah silk is cultivated in China. It is much coarser than real silk and has a characteristic brown shade. Tussah silk has a much greater resistance to chemical action than real silk, and, in consequence, requires a more drastic treatment for the boiling-off process. It is very difficult to dve evenly.

## THE ARTIFICIAL SILKS

Artificial silk is a misnomer. The name conveys the impression of silk produced by other than natural means. In general, it may be said that the so-called artificial silks are produced from cellulose, and thus are closely related to cotton and the vegetable fibres. The textile industry has endeavoured in many cases to find better names for this

new fibre, such as "lustra cellulose", or "rayon", but, unfortunately, the original term "artificial silk" still persists.

Artificial silk is not marketed or employed as a substitute for silk. It has a market of its own, and is one of the most important of the textile materials.

There are four kinds of artificial silk: (1) Chardonnet silk; (2) Cuprammonium silk; (3) Viscose; (4) Cellulose acetate. All these are also known by a variety of names. The first three are very closely related in that they all belong to the class of regenerated cellulose. In their preparation, cellulose, in the forms of short stapled cotton or wood pulp, is treated by various chemicals so that a solution is obtained. This solution is then forced through a tiny orifice or spinneret in such a manner that the cellulose is regenerated to form a long continuous thread of very high lustre.

Chardonnet silk is also called nitro silk, pyroxylin silk, collodion silk, or by the trade-name of "trubize". It was first prepared by Count Hilaire de Chardonnet in 1884, but it was not until some 15 years later that it was produced on a commercial scale. The starting-point of the process is cotton linters, that very short fibred cotton as obtained by combing or ginning raw cotton. The linters are converted into nitro-cellulose by the action of nitric acid with sulphuric acid. Nitro-cellulose will dissolve in a mixture of alcohol and ether to give a thick syrupy solution. Such a solution is prepared and forced through fine jets, so that the alcohol and ether evaporate and leave the nitro-cellulose as a fine thread. The evaporating solvents are recovered for re-use. The regenerated nitro-cellulose is highly inflammable, but can now be made safe by denitration. Denitration is carried out by treatment with ammonium sulphide or sodium hydrosulphite. By denitration, a loss of strength takes place, but a stable thread of high lustre is obtained. Chardonnet silk is cellulose containing a small amount of nitrogen, the presence of which serves to distinguish it from other forms of artificial silk. The thread is bleached with chlorine bleaches. Chardonnet silk has the disadvantage of being very weak when wet, a factor which is of great importance to the launderer, dyer, and cleaner. Wood pulp may be used in place of linters in Chardonnet silk, but the product is much inferior.

Cuprammonium silk. Mercerised cotton is readily soluble in a solution of ammoniacal copper hydrate, and such a solution is employed in

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the preparation of cuprammonium silk. The solution of cellulose is coagulated by forcing through orifices into a solution of acetic acid, sulphuric acid, or caustic soda. The thread is stretched during coagulation, and afterwards thoroughly washed. The copper is removed by passing the thread through an electrified acid bath. Cuprammonium silk has a high lustre, and when spun by special methods resembles real silk very closely in softness and draping qualities. It is known by such names as Brysilka, Glanzstoff, Pauly silk, Parisian silk, Thiele silk, and Cuprate silk.

Viscose silk is without doubt the most important of the artificial silks. It is extensively manufactured at the present time, and its commercial manufacture has created a new textile industry within recent time. It has the great advantage of having a comparatively cheap and plentiful raw material. The viscose process employs cellulose in the form of wood pulp. The bleached sulphite wood pulp (usually in the form of standard-sized sheets) is cut up and treated with caustic soda to form alkali-cellulose. The excess caustic soda is squeezed out and the product ground in a machine. The ground, finally divided alkali-cellulose is termed "crumbs", from the crumb-like appearance of the mass. The crumbs are stored at a fixed temperature for three or four days. After this maturing process, the crumbs are treated with carbon bisulphide in iron vessels fitted with mechanical stirrers. As a result of this process an orange-coloured, jelly-like product is obtained. This product is known as cellulose xanthate. Cellulose xanthate is soluble in water, or in weak caustic soda, to give a syrupy solution. The xanthate is dissolved in caustic soda solution and, after filtration, is allowed to ripen until the solution reaches a certain stage of fluidity or viscosity. The viscose solution is coagulated by forcing the filtered solution through spinnerets into a bath containing a mixture of sulphuric acid, ammonium sulphate, and glucose. The viscose obtained is of brown yellow colour and has little lustre. It is passed through a solution of sodium sulphite, to remove sulphur, and is then bleached with sodium hypochlorite. The thread is finally washed and dried under tension, in order to increase the lustre.

Acetate silk differs from the usual types of artificial silk, in that, while they are simply regenerated cellulose, it is a chemical combination of cellulose with acetic acid. Acetylated cellulose was first produced in the form of dope for making wings of aeroplanes completely waterproof.

The controlled action of a mixture of glacial acetic acid with acetic anhydride and sulphuric acid on cellulose produces an acetylated cellulose, which is soluble in certain organic solvents. These solvents may be chloroform, acetic acid, or acetone. The solutions are coagulated by water, or by evaporation of the solvent. Many compounds of acetic acid with cellulose are known with properties varying according to the acetyl content. Since acetate silk is an acetate acid compound, it is easily decomposed by alkalies, particularly caustic alkali. Acetate silk is resistant to dyes and must be specially treated. It differs from other artificial silks in that it does not lose tensile strength on wetting, rather increases strength, and, also, it is practically non-inflammable. Cellulose acetate silks, in contact with flame, give a hard, black bead.

Dyers will be familiar with immunised tape, as used for marking. Immunised tape remains undyed by normal methods in that it may be used for ink-marking without risk of the marks being covered or obliterated in the dyeing process. Acetate silk belongs to a chemical class known as "Esters", i.e. it is an acetic ester. Immunised cotton is obtained by treating the cotton tape with a limited quantity of paratoluene sulphochloride in presence of caustic soda. In this way a sulpho-ester is formed on the surface of the tape, and the cotton is "immunised". It will be readily understood that, since immunised cotton belongs to the same chemical class as cellulose acetate, it will also be affected in any process for dyeing or otherwise treating this type of artificial silk.

In the beginning, artificial silk possessed an extremely high, almost metallic lustre, which eventually was considered rather a disadvantage. Recently special treatments have been devised, in order to de-lustre artificial silk and so impart appearance and handle more like that of real silk. In some cases the solution is treated before spinning, and in others special after-treatment is applied. Special care is necessary in many cases in handling de-lustred artificial silk, in order to avoid disturbance of the de-lustring agent.

# MODERN AND DYEING CLEANING PRACTICE

hot

dissolves

Yellow, insoluble

Dissolve rapidly with

yellow coloration

Insoluble

Yellow, insoluble

Brown, insoluble

Insoluble

Insoluble

NITRIC ACID

slowly

slowly

rapidly

Yellow,

# Dissolves Soluble Burn slowly with characteristic smell ANIMAL FIBRES Insoluble Soluble Black Wool ploo Acetate Silk Forms beads swells Fibre ARTIFICIAL FIBRES Rapidly dissolve grates and partly dissolves IDENTIFICATION OF TEXTILE FIBRES Char-donnet Burn rapidly with Disinte-1 pungent smell Un-changed Viscose Dissolves slowly Yellow, insoluble Insoluble Ramie Dissolves slowly Burn rapidly with pungent smell Hemp VEGETABLE FIBRES Brown, insoluble Dissolves Jute Insoluble Insoluble Dissolves Linen Dissolves Cotton : SULPHURIC ACID 168° Tw. ALKALINE LEAD CAUSTIC SODA 76° Tw.

BURNING

Silk

AMMONIAGAL COPPER SOLUTION	Soluble	Soluble	Insoluble	Insoluble	Soluble Insoluble Insoluble Insoluble	Swell, disintegrate and are partly dissolved		Un- changed	Un- Insoluble Soluble changed cold	Soluble cold
ANILINE SULPHATE			Yellow	Yellow	1					1
ACETONE						Un- Un- Dissolves changed changed rapidly	Un- changed	Dissolves rapidly	1	I
IODINE AND SULPHURIC ACID	Blue	Blue	Yellow	Yellow	Blue		I	I	I	
DIPHENYLAMINE AND			1				Blue	1	1	J

# Chapter II

#### DRY CLEANING

The first mention of cleaning by means of a dry process on a commercial scale appears as recently as 1848, when there is evidence that a method of cleaning with "essence" was being attempted in France. This original employment by the French led to the process being termed "French cleaning", a term which still persists. The "essence" used by the French was camphene, an oil of turpentine, which was specially distilled at that time for use in lamps. It is difficult to imagine any great success with this new process, and it is not surprising that the French cleaners began to look round for more suitable cleansing agents. After about ten years, solvent naphtha was employed and, almost simultaneously, petroleum benzine. The dry-cleaning process appears to have been practised exclusively by the French for nearly twenty years, until the process was introduced into the United Kingdom by Messrs. J. Pullar & Sons, of Perth, in 1866.

The dry cleaning industry in this country is, therefore, just seventy years old. The methods and men employed in the Perth works were imported from Paris. Solvent naphtha and petroleum benzine were the solvents used. These two solvents, or slight modifications of them, have been used exclusively in dry cleaning from 1866 up to recent times, when their "monopoly" has been challenged by the introduction of the non-inflammable solvents.

The liquids or essences used in dry cleaning belong to that category of chemicals known as solvents. The chemistry of solution and the functions of solvents have by no means been thoroughly and exhaustively investigated, and out of the present state of knowledge no logical explanation of the widely variable action of solvents on different substances is forthcoming.

The essential property required of a dry cleaning solvent is that it must be an efficient grease solvent. Any chemical that will dissolve grease would serve as a dry cleaning agent, and the number of such

chemicals is legion, but, in the main, the vast majority of them are ruled out by the eliminating process of commercial conditions. These conditions can be indicated by even a sketchy outline of what comprises a dry cleaning process.

The goods to be treated are immersed in the solvent for a prescribed length of time, which varies, not only with the type of goods, but also with the solvent used and the style of machinery employed. The second stage is that of removing the solvent from the articles. This is usually carried out by removing as much as possible by centrifugal extraction, and then evaporating what still remains in the goods.

The third, but by no means the least important stage is that of recovery of the solvent for use again. In this stage the final and only certain method of recovery and purification is distillation. The more efficient the recovery of solvent, the greater the commercial success of the operation. No dry cleaning process could be worked profitably without the recovery of the solvent.

The three stages of a dry cleaning process are: cleaning, drying, and recovery. It will be seen from this that the cleaning medium passes through a series of changes, first as a liquid, secondly as a vapour due to evaporation of the liquid, and, finally, as a liquid due to condensation of the vapour from the evaporation process.

The ease, or otherwise, with which these changes can be brought about is connected with one peculiar characteristic of the solvent, known as volatility. The second essential property of a dry cleaning solvent is that of being volatile.

To these two essentials must be added many more limitations in the characteristics of a solvent suitable for dry cleaning purposes. The complete specification of the ideal solvent would be as follows:

- (1) Must be an efficient solvent for greases, oils, and fats.
- (2) Must be a volatile liquid.
- (3) Must be chemically inert, that is, without action or effect on all kinds of textile fibres.
- (4) Should be free from offensive odour.
- (5) Should not be too volatile.
- (6) Should not be too non-volatile.
- (7) Should be non-toxic, that is, without injurious effect on operatives.
- (8) Relatively cheap.

- (9) Should be chemically stable.
- (10) Not miscible with water.
- (11) Colourless.
- (12) Flash point as high as possible.
- (13) Close distillation range.

The importance of the first two conditions has already been indicated, and the significance of the remainder calls for explanation.

The third condition of being chemically inert is actually the source of the term "dry" in "dry cleaning". Many trade terms appear to be misnomers. Perhaps the most recent is that of "artificial silk", a name applied to a textile which is not "silk" in any form, artificial or otherwise. Similarly, "dry cleaning", being a process of immersing articles in a liquid, would also appear to be a misnomer. It is somewhat difficult to explain to a layman how such a process can be termed a "dry" one, but it should be quite clear to technical men that in dry cleaning the immersion in the solvent does not cause "wetting", such as immersion in water does. Water is one of the most potent of chemicals and its characteristic chemical properties are such that, in addition to "wetting" textile fibres, it causes them to become limp, to swell, and lose shape and size. This effect on textiles is always exercised by liquids containing water. A process from which these effects are absent is termed "dry" in contra-distinction to a water process.

The wide adoption of cellulose acetate for the manufacture of textiles has narrowed the range of dry cleaning solvents, for many liquids which otherwise might find application in dry cleaning have a dissolving action on this material. This applies to some of the synthetic solvents, especially certain of the chlorhydrocarbons. The ideal solvent must be above suspicion in its inertness to cellulose acetate.

The fourth condition of being free from offensive odour would almost go without saying, but it must be pointed out that certain dry cleaning fluids would impart a definite odour to garments cleaned in them. At one time it was commonly supposed that a certain smell was associated with dry cleaning, and the writer has knowledge of a complaint being made of incorrect cleaning, because of the absence of the odour of cleaning. While the odour sometimes found in dry cleaned garments has been due to the incomplete removal of the solvents, there have been cases of smell due to impurities, particularly the presence of sulphur compounds. Apart from the question of solvent left in the goods, the ideal solvent must not transmit any odour. Much emphasis is to be placed on the point of odourless cleaning.

The conditions (5) and (6), (5) specifying the desirability for the solvent not to be too volatile, and (6) for the solvent not to be too nonvolatile, appear to cancel each other. It is agreed that a dry cleaning solvent must be a volatile fluid, but it will be appreciated that a highly volatile fluid would evaporate readily at ordinary temperatures and consequently loss by evaporation would be difficult to avoid. The ideal solvent would have a very slow rate of evaporation at ordinary temperatures, and so conform with the condition (5) of not being too volatile. The apparently contradictory condition (6) is necessary when considering the drying stage of the cleaning process; once the actual cleaning process has been completed, the removal of the solvent has to be brought about by evaporation, and should the solvent be too non-volatile, too large a consumption of steam for heating purposes will be required, in addition to prolonging the time of drying. A further point is that the presence of a portion of solvent difficult to evaporate entails the danger of some solvent remaining in the goods. As steam consumption is a factor in the cost of recovery by distillation, too great a degree of non-volatility is all the more undesirable, not only because of the greater amount of steam necessary, but also because of the increase in "tailings" (that is, unrecoverable residue) from distillation which would result.

The ideal solvent should be non-toxic, according to condition (7). This condition has important bearing upon the newer forms of dry cleaning, and many new solvents are open to criticism because of the suspicion that they have an injurious effect if inhaled by operatives. It is obvious that the ideal solvent would be a harmless one. The question of toxicity covers such a wide range, that it can only be treated adequately when taken as a subject by itself.

Condition (8) of being relatively cheap calls for no comment. The dry cleaning industry is not different from any other industry in its demand for cheap, easily obtainable raw material.

The condition of being chemically stable (9) also comes under review with more prominence because of the adoption of the

non-inflammable solvents for dry cleaning. Many of the new solvents have a tendency to decompose under certain conditions, and the result of the de-composition is the production of hydrochloric acid and the highly poisonous gas, phosgene. For example, carbon tetrachloride, in presence of moisture and in contact with iron, decomposes with the liberation of hydrochloric acid, which leads to corrosion of metallic parts of machinery. Apparatus for the use of carbon tetrachloride must be constructed from special metals: copper, nickel, and monel. It is customary to use monel whenever possible. Trichlorethylene is stable to the action of moisture in presence of metals, but tends to decompose under the action of direct sunlight, and become acidic in which condition its action on cellulose acetate is increased.

Any tendency to decomposition or alteration during storage and repeated distillation is undesirable, and should be absent in the ideal solvent.

The term "miscible" can, perhaps, best be transcribed as "mixable", so that non-miscible with water means that when the solvent is shaken up with water, the two liquids do not mix, but immediately, on standing separate from each other, form two distinct layers. In dealing with dry cleaning solvents, the characteristic property of being non-miscible with water has great importance in connexion with the purification and recovery of dirty solvent. For example, it is usual in certain processes of recovery to treat the used solvent with soda lye, which has the effect of removing fatty compounds and neutralising acids. Soda lye treatment is a recognised method of benzine and white spirit purification, and is also used with some of the new non-inflammable fluids, particularly trichlorethylene.

This treatment consists in passing strong solutions of soda, either caustic soda or carbonate of soda, into the solvent, and, after agitation, allowing the liquids to separate into layers. In the case of the petroleum solvents, the solvent floats on the layer of water, but with all the non-inflammable solvents the water forms the top layer. The drawing-off of either layer is effected by a very simple mechanical device, known as a separator. The property of being non-miscible is yet another adjunct to the ideal solvent.

The eleventh item in the ideal specification, that which relates to colourlessness, is obvious and needs no comment.

The twelfth item refers to a technical term, "flash point", which may require some explanation. When a liquid is said to be inflammable, it is generally the vapour from the evaporation of the liquid that is inflammable. It would be more correct to say that the liquid gives off an inflammable vapour. The flash point may be taken to indicate the lowest temperature at which the vapour will take fire and continue to burn on the application of a flame or spark. The older solvents, the petroleum and aromatic hydrocarbons, give off inflammable vapours at all temperatures. The railway companies classify inflammable liquids in two classes:

- (1) Those flashing below 73°F.
- (2) Those flashing between 73°F. and 150°F.

Both classes are considered to be dangerously inflammable. Benzine, benzols, and solvent naphtha are in class (1), and white spirit is in class (2). The chlorhydrocarbons have no flash point, as they are absolutely non-inflammable, which gives them a big advantage over the petroleum solvents. With solvents such as benzine or white spirit, the danger from fire cannot be said to be absolutely absent, in spite of all precautions in the way of special equipment and machinery, while the chlorhydrocarbons fulfil this twelfth condition exactly. The ideal solvent would be non-inflammable, that is, without a flash point.

The last item refers to another technical term, "distillation range". This term is best explained by reference to the distillation of the petroleum products. With all the commercial petroleum solvents, distillation begins at relatively low temperatures, and once the initial distillation temperature has been reached, definite percentages of solvent distil within the limits of every 10° of temperature. The final point is reached when all the solvent has been distilled. Between the initial distillation temperature and the final end point, there may be a large range of temperature, the extent of this range varying with the source of the petroleum. Thus, in the case of benzine from Borneo petroleum:

4% distils below 80°C.

74% distils between 80°C. and 130°C.

22% distils above 130°C.

Most of the benzines have an end-point between 120°C. and 140°C., while, only in rare cases, is the extent of boiling-point range less than 60°C.

A dry cleaning solvent with a long distillation range would require greater consumption of steam in distilling, and would entail a longer time to ensure complete drying-out from the cleaned goods than one with a short range. Yet another point is that the chemical nature of each portion may vary according to its position in the distillation range, so that variability of solvent is difficult to avoid. A solvent with a very short distillation range is nearer the ideal than one with a long range, and one with no range at all (i.e. all the solvent distils at the same temperature) is ideal.

The specification of the ideal solvent can be interestingly compared with the actual specification for "Stoddard solvent", which, according to the United States Bureau of Standards, is as follows:

- (1) Appearance shall be clear and free from suspended matter and undissolved water.
- (2) Colour shall be water white or not darker than 21 by the Saybolt chromatometer.
- (3) Odour shall be sweet.
- (4) Flash point not lower than 100°F.
- (5) Corrosion test: a clean copper strip shall show not more than an extremely slight discoloration, when submerged in solvent for three hours at 212°F.
- (6) Distillation range: not less than 50% at 350°F. The dry or end point not above 410°F. No tolerance shall be allowed above 410°F.
- (7) Acidity: the residue after distillation shall not show acid reaction.
- (8) Doctor test shall be negative.
- (9) Sulphuric acid absorption test: not more than 5% of solvent absorbed by sulphuric acid (1.835 Sp. gr.).

There is no one solvent in use at present that conforms to every one of the requirements suggested in the specifications of the ideal solvent. Where a solvent fits exactly to several of the items, it invariably fails in one or more of the others. It can be definitely said that the "perfect solvent" is not yet known.

The solvents which find application in the dry cleaning industry at the present time are classified into two sharply defined groups, according to whether they are inflammable or non-inflammable.

While the inflammable solvents lend themselves to a further classification, based on their chemical constitution, the non-inflammable solvents all belong to the same chemical class, the chlorhydrocarbons. The complete classification of dry cleaning solvents would, therefore, be as follows:

- (a) The inflammable solvents—
  - (1) The petroleum hydrocarbons.
  - (2) The aromatic hydrocarbons.
- (b) The non-inflammable solvents—
  - (1) The chlorhydrocarbons.

The line of demarcation between these groups is quite definite, for the solvents in any one division have marked differences in their characteristic properties from those in either of the other divisions.

The Petroleum Hydrocarbons.—The solvents included in this division are those belonging to the paraffin or aliphatic series of hydrocarbons. In its wider sense, the term "petroleum" is an omnibus one, and embraces all the inflammable solvents. For example, in the Petroleum (Consolidation) Act, 1928, petroleum and petroleum spirit are defined as follows:

"'Petroleum' includes crude petroleum oil, oil made from petroleum, or coal, shale, peat, or other bituminous substances, and other products of petroleum. 'Petroleum spirit' means such petroleum as when treated in the prescribed manner gives off an inflammable vapour at a temperature of less than 73 deg. F. Petroleum spirit includes benzine, benzole, benzoline, carburine, gasoline, motor spirit, naphtha, pentane, petrol, and other similar substances."

The petroleum hydrocarbons are the most widely used of all the dry cleaning solvents. The two representatives of this group, benzine and white spirit, have been employed almost universally until recent times. Petroleum benzine has been used for dry cleaning since the beginning of the industry.

An unfortunate vagueness in nomenclature must be pointed out. Two terms—BENZINE and BENZENE—while denoting two absolutely different liquids, have the same pronunciation, and consequently the two are often confused. The author has known instances where it has been difficult to decide which liquid a speaker has referred to.

The production of solvents for dry cleaning forms a very small part of the petroleum industry, being merely incidental to the major object of the production of motor spirits, oils, and oil fuels. Yet the application to dry cleaning was one of the first instances of the commercial application of petroleum other than for heating and lighting. It is rather surprising that the potentialities of petroleum products had not been recognised earlier than they actually were, especially in view of the fact that they have been known from the earliest ages. Reference to petroleum products can be traced in the most ancient records, and there is evidence of their use as far back as 3000 B.C. The Bible refers to "oil out of the flinty rock" (Deut. xxxii, 13), which was probably crude petroleum.

The natural sources of crude petroleum are of three distinct classes: natural gas, crude petroleum oil, and shale oil. The term "natural gas" is yet another example of indefinite nomenclature. Natural gas could be the name assigned to any escape of gas from below the crust of the earth, and many cases of the outburst of gases, such as carbon dioxide, sulphur dioxide, and nitrogen are known, especially in connexion with volcanic action. But "natural gas" refers to the emission of petroleum gases. These escapes from fissures in the earth's crust probably account for the "sacred fires" known to the Tartars around the Caspian Sea. The "natural gas" yields petroleum "distillates" by processes of alternating compression and expansion, or by means of chemical absorption. The actual yield of benzine suitable for dry cleaning is very small, as such benzine would be extremely volatile, and, in consequence, its use would entail heavy and unavoidable loss by evaporation.

The main source of cleaners' benzine is crude petroleum oil. This crude oil has been found in practically every country in the world. Some years ago, a well was bored in Derbyshire and a considerable yield of good oil obtained. In spite of the almost universal occurrence of petroleum, the world's supply of crude oil is, however, derived from a few well-defined areas—the U.S.A., Mexico, Russia, Malay Peninsula, and Roumania contributing about 95% of the total output.

The technology of petroleum is a branch of applied science in itself and demands a long period of specialised training and study. The "working up" of the crude petroleum oils is the isolation of certain portions or fractions, which have specific applications in the various

industries. The separation into fractions is achieved by a process of distillation, which is essential in the refining of all crude oils. The crude oil is heated in retorts to convert the volatile constituents into gas, the lighter or more volatile being converted into vapour first, followed by the heavier fractions in turn as the temperature of the retort increases. The gases then pass through cooling condensers, where they are converted into liquid. The condensed liquid is collected in receivers, which are changed as the temperature of distillation rises beyond certain points. Thus, a series of fractions are obtained, the nature of which may vary not only with the crude oil, but also with the method of distillation.

Three methods of distillation are adopted:

- (1) Distillation at atmospheric pressure.
- (2) Distillation under vacuum.
- (3) Distillation under pressure.

A modification of the first method is obtained by the introduction of live steam into the oil during the later stages of the process.

The distillation of crude petroleum oils at atmospheric pressure, or under greater pressure, is usually employed when larger yields of benzines and motor spirit are desired. Distillation under vacuum gives higher yields of lubricating oils.

The first fraction obtained during the preliminary distillation of the crude oil is usually taken up to a boiling point of 220°C., and gives a product suitable for benzine or motor spirit. The next fraction will probably be a mixture of benzine and kerosene (paraffin oil for burning), the exact composition depending upon the temperature at which the receiver is changed. In the petroleum industry the changing of the receiver for the distillate is termed "making a cut". The point at which a cut is made is decided by laboratory tests. This preliminary distillation gives only a rough separation of the constituents of the crude oil. No well-defined fractions are obtained; for example, there is no sharp division between benzine and kerosene.

To effect a clearer separation each fraction is further distilled and fractionated. The complete working up of crude petroleum may include distillation, refrigeration, filtration, and chemical treatment before the various products are ready for the market. The benzines, having been separated from the kerosenes, may be further divided to

give four fractions. The first and lightest fraction is used for aviation spirit, the second for motor spirit or petrol, the third for cleaning spirit or benzine, and the fourth and heaviest for white spirit or turpentine substitute. This description of the working up of petroleum crude oil, in spite of its sketchy character, clearly indicates the relationships between benzine and white spirit.

The third source of petroleum crude oil is oil shales. The occurrence of oil shales in the earth and the method of mining are comparable to the occurrence and mining of coal. By a process of destructive distillation, the oil shales yield a crude oil, which is then refined in a manner similar to natural crude petroleum. Oil shales have been mined in several places in the British Isles, particularly on the Lothian Hills in Scotland. The oil shale industry is handicapped in its competition with the more cheaply refined oils from natural petroleum owing to the additional cost of mining and distilling the shales. On the other hand, it is helped by the fact that ammonium sulphate is obtained as a by-product. This assistance is, however, negligible at present, owing to the state of the sulphate market. Scotch shale oil produces a benzine suitable for dry cleaning.

The benzines obtained from American and Borneo crude oils and, more recently, from Roumanian oil, are largely employed for dry cleaning. The finer points among the properties of benzine may vary according to the source of the crude oil, but, in general, benzine is a light, mobile spirit of specific gravity between 0.70 and 0.78, and is an excellent solvent for grease, oils, and fats. It is comparatively volatile, and precautions must be taken to prevent loss by evaporation. The main constituents of benzine belong to the series of hydrocarbons known as the aliphatic, or paraffin, series. Benzine is a mixture of paraffin hydrocarbons, hexane, heptane, octane, and nonane.

The term "paraffin" denotes "without affinity", a name particularly applicable to the series, because of their stability or inertness to chemical change.

As a dry cleaning solvent, benzine is absolutely harmless to every kind of textile material. It is without action or effect on all fibres.

The odour of benzine is distinctive, though generally not offensive. The presence of sulphur, probably in the form of mercaptans, would impart an unpleasant smell to the cleaning spirit. During the process of refining, sulphur compounds are removed by agitating the petroleum

with concentrated sulphuric acid, or by a treatment with a solution of sodium plumbite. A simple test for sulphur compounds in cleaning spirit is an imitation of the refining process. The sample of benzine to be tested is treated in a test tube with a tenth of its volume of concentrated sulphuric acid, thoroughly shaken, and then allowed to stand. A layer of acid will form in the bottom of the tube and will be coloured from pale yellow to a brown shade, according to the sulphur content. The average commercial benzine is practically free from sulphur compounds, and therefore without unpleasant smell.

Being a mixture, benzine boils over a considerable range of temperatures. Some samples begin to distil about 60°C., and varying proportions distil between every 10° of temperature until a dry or end point is reached about 150°C. An initial boiling point below 80°C. would tend to serious losses from evaporation, while the less volatile portion, above 130°C., would entail lengthening the time of drying, with increased consumption of steam, as well as the risk of some residue of spirit being left in the goods after cleaning.

For these reasons, a spirit showing a larger portion distilling between 80°C. and 130°C., is usually considered the best and most economical. It is impossible to lay down any hard-and-fast rule, since the suitability or otherwise of benzine depends upon the system of cleaning in operation. The use of open washers involves conditions differing from those of the closed circuit systems. A totally enclosed system, such as the Barbe, entails a large initial outlay, and to offset this the maximum number of loads per day must be maintained. In this system the drying takes place in the machine, and hence, the presence of any high boiling fraction would mean increasing the time of the process, and so reducing output and adding to the consumption of steam.

While many would state that, for practical purposes, benzine is non-toxic, it must be emphasised that this solvent can cause violent headache, and with some people a state of intoxication. "Women are more readily intoxicated with benzine fumes than men. Headache, giddiness, and vomiting are symptoms commonly developed. Workpeople who become quarrelsome or intoxicated should be taken outside into the open air until they recover." A Home Office Memorandum on Dry Cleaning states: "Good ventilation is necessary in order to mitigate the intoxicating effect of benzine vapour. When this is not considered, fainting and hysterical symptoms are not uncommon,

especially in women under 25 years of age." Other symptoms have been irritation and pain in the eyes; headache, dizziness, excitability; followed (late in the afternoon) by drowsiness and a sense of exhaustion.

The asphyxiating effect of benzine varies in its intensity with the persons concerned. A new-comer may be affected considerably, but it is generally found that a worker becomes acclimatised, so that an operative who has spent any length of time among benzine fumes is rarely conscious of any ill-effect. The real danger with benzine cleaning can only occur when tanks are being cleaned.

Benzine is certainly the ideal solvent if we consider only the facts that it is easily obtainable and relatively cheap. The present-day price of dry cleaners' benzine is 15.  $3\frac{1}{2}d$ . per gallon, the lowest price of all the dry cleaning solvents. The low price of benzine is even more pronounced when it is recognised that the price quoted includes a Government tax of 8d. per gallon, the tax actually being 100% ad valorem on the benzine.

Another satisfactory characteristic of benzine is that it is quite stable to storage and repeated distillation. Owing to its inert nature, it has no tendency to corrode machinery and pipe-lines, and consequently no special precautions of this kind are necessary, which is not the case in the construction of installations for other solvents. The ideal solvent must withstand repeated distillation without chemical change, and benzine fulfils this condition exactly.

Yet another ideal property of benzine is its non-miscibility with water. This property is of the greatest importance, not only in certain processes of distillation, but also in the alternative processes of purification and recovery for re-use by chemical means. Perhaps the most common method of distillation is that of heating the still by means of closed steam coils in order to boil off the more volatile portions, and then completing the distillation by blowing live steam into the spirit. The open steam carries with it the higher boiling fractions, enabling these to evaporate at a temperature lower than normal. The distillate from this process is a mixture of benzine and water. It will be clear that if the benzine and water were miscible, the dry cleaning properties of the spirit would disappear entirely. Fortunately, the opposite is the case, so that two separate layers are formed, the benzine floating on the surface of the water.

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The parting of the two layers is brought about by means of an arrangement known as a separator. The principle of the separator is that a column of water in one arm of a U-tube will support a higher column in the other arm, consisting partly of water and partly of benzine (benzine having a specific gravity less than that of water, for example, benzine sp. gr. 0.70 to 0.78). Thus, benzine can be drawn from the top of the composite column at a level above that of the water in the other arm of the U-tube.

The same property of non-miscibility with water is employed in the more recent methods of recovery by chemical treatment. All processes of chemical settling depend upon the removal of minute amounts of moisture which, adhering to the surface of the dirt particles, cause these to remain in a state of suspension. By agitating the benzine with dehydrating agents, which may be certain acids on the one hand, or certain alkalies on the other, the moisture is gathered into an aggregate layer, which sinks to the bottom of the receptacle along with the particles of dirt, while the clarified benzine floats on the top.

The flash point of benzine is far from ideal, and is the cause of most of the restrictions imposed on its use by the Home Office. For instance, there is the regulation that a licence must be obtained by anyone who wishes to store or use benzine. The flash point is so low that benzine gives off a vapour at ordinary temperatures, which is not only highly inflammable, but is also liable to form explosive mixtures with air. In order to reduce the risk due to the highly inflammable nature of benzine, the granting of a licence is accompanied by many conditions, contravention of which makes the offender liable to heavy fine.

The danger from fire with benzine is very real at all stages connected with its use, even without the accentuating nature of its conductivity to electricity. The electrical conductivity of the petroleums is so exceedingly low that for practical purposes they may be considered as non-conductors. This adds to the danger of fire and explosion, since the movement of garments, particularly silk goods, in a dry cleaning machine, may give rise to a sufficient charge of static electricity to cause sparking. It has been found that a charge of electricity can be formed by passing benzine through pipes from one tank to another. The danger from the accumulation of these static charges is the "bogey" of every cleaner using benzine.

It is fortunate that the electrical conductivity of benzine can be increased considerably by small additions of certain compounds. Particularly striking is the influence of small amounts of soaps. In his book, *Petroleum and its Products*, W. A. Cruse, of the Mellon Institute of Industrial Research, states that 0.05% of magnesium oleate increases the conductivity 100 times.

In 1910, a special inquiry by a departmental committee of the Home Office resulted in several precautions being recommended to be observed in dry cleaning works. Most of the precautions then suggested are now compulsory within the London County Council area. The report of the committee, issued as a *Memorandum on Dry Cleaning*, states:

"The principal cause of explosions in such works is that textile fibres moved about in benzine, either in the cage of a revolving washing machine, or when being rinsed by hand in a tank, become charged with static electricity which may cause sparking and so ignite the mixture of air and vapour in the machine. The production of electricity is more likely to occur when the articles being cleaned are perfectly dry, as on a hot day."

The reference to the dryness of the cleaned goods is interesting because of a custom that many cleaners once followed, of drying their work before cleaning in order to remove moisture. Records of fires in cleaning establishments in America show that in one particular year every fire investigated proved to be at a works where pre-drying was practised.

Within the London County Council area, all cleaning machines, extractors, and drying tumblers must be electrically earthed. In the case of extractors, the rubber cushion on which the spindle housing rests would make a perfect insulator for the revolving cage, and thus increase the possibility of heavy static charge on the cage, so that it is essential to earth the spindle housing. In the case of a charge having accumulated on an article, the earthing would not reduce the danger of sparking when the goods are being removed from the machine. "It would appear that the only way of getting rid of such a charge safely is by allowing the machine to stand for some minutes, so as to give the charge time to leak away before opening the cover, and where this is done, the efficient earthing of the machine is an additional advantage."

The precautions taken to prevent fires or explosions in benzine works include the use of humidifiers in all rooms in which spirit is used. It has been proved that in an atmosphere of more than 70% humidity no electrical charges can be obtained. The term "humidity", or, more correctly, "relative humidity", refers to the amount of water vapour in the atmosphere. It is expressed in the form of percentage, indicating the percentage present in the atmosphere, 100% being the atmosphere saturated. This saturation point varies with the temperature, and hence the word "relative". The London County Council insists on the provision of wet- and dry-bulb thermometers, and the maintenance of a humidity not less than 70% saturation. The air is humidified by jets of steam or water should the humidity fall below the required level. Systems of automatic humidification are strongly recommended. This provision of artificial humidity is considered to be the most effective of all methods for preventing fires.

Another precaution is the use on all machines of heavy lids with counter-balance weights held by easily fusible links, which would melt by the heat of a fire and allow the lids to close. The cutting off of air supply from a fire is assisted by means of spring buffers, which would throw back the lid of any machine blown open by the force of an explosion. Many machines are now fitted with explosion doors, that is, with small spring lids which operate a steam valve if opened and admit steam into the machine. An American suggestion is that steam should be blown into drying tumblers for five minutes immediately the goods are placed in the machine.

With the object of eliminating the danger from fire in benzine cleaning, enclosed cleaning systems have been designed. One of these, the Barbe, takes the special precaution of replacing the air in the machine with carbon dioxide, and so reduces the fire risk to an absolute minimum. Some German works use carbon dioxide to fill the free space in the benzine storage tanks and have the spirit pipe-lines enclosed in outer pipes containing carbon dioxide under pressure, so that in the event of leakage in the spirit pipe, the pressure of the carbon dioxide would force the liquor back to its storage tank.

The restrictions due to the low-flash point of benzine are very numerous. In addition to those mentioned, there are others applied to the design of work-rooms, the arrangement of plant, the clothing and boots worn by operatives, the ventilation and lighting of rooms,

the equipment for extinguishing fire, the use of gauge glasses on storage tanks, the position of storage tanks, and the prevention of leakage of inflammable spirit. Nor is this list complete, for the following come under the heading of general precautions:

"Materials should never be dried directly before being cleaned, since a static charge is more likely to be formed if the materials are free from moisture.

"No person should be allowed to smoke or to take into any part of a dry cleaning works any naked light, lamp, matches, or any apparatus for producing a naked light or spark.

[Notices to this effect are invariably found posted up in dry cleaning establishments.]

"No person should be allowed to work alone in a dry cleaning process."

The low-flash point of benzine is definitely a handicap, and is the chief reason why many cleaners favour petroleum products of higher flash points, and why the newer non-inflammable solvents have a big advantage over the older ones. The insurance companies indicate the fire danger from benzine by demanding high premiums on all buildings where it is used.

The distillation range of benzine usually covers about 100° on the Centigrade scale, from 60°C. to 160°C. Many petroleum concerns market "close range" benzine specially prepared for dry cleaning. The nearest to ideal in a benzine would be obtained with a spirit showing a maximum percentage distilling between 80°C. and 120°C. It rarely occurs that this percentage approaches 80%.

The following is the complete specification of a spirit sold under the name of "special cleaning benzine", which shows a satisfactory percentage distilling between the limits indicated:

Specific gravity at 60°F.					0.738
Flash point—probably be	elow				32°F.
					52°C.
Distilling 10% at 85°C.		Disti	lling 6	io%	113°C.
" 20% " 93°C.		•	, 7	o%	118°C.
" 30% " 99°C.		9:	, 8	60%	124°C.
" 40% " 104°C.		9:	, 9	0%	133°C.
" 50% " 108°C.					

Dry point 164°C.
Total distillate 97.5%
Residue in flask 2 c.c.
Loss 0.5 c.c.

Colour W.W. (water white)

Odour Sweet

It has been stated that the ideal petroleum benzine for dry cleaning would have a constant boiling point of 95°C. This statement, which was made in an article in the Journal of the Society of Chemical Industry in 1906, has an added significance in these days when comparisons between the petroleum solvents and the chlorhydrocarbons are so often made. The nature of the petroleum benzines, being mixtures of hydrocarbons, precludes the possibility of a constant boiling point.

The second representative of the petroleum series is white spirit. This solvent is very closely related to benzine, the main difference being that its essential temperatures are much higher than those of benzine. The introduction of white spirit for dry cleaning was the result of a demand for a cleaning solvent with the same efficiency as benzine, but without its disadvantages, particularly the danger due to inflammability.

Mr. Dixie Stoddard, in an article in the American journal, Chemical Markets, July 1932 (reproduced in The Dyer, August 19, 1932), points out that previous to 1907 dry cleaning in the United States was very crude, a poor grade of gasolene being used. Between 1907 and 1917 experiments were made with solvents to improve the quality of dry cleaning. "Much was accomplished. Special cleaners' naphtha was produced by refineries, eliminating much of the rancidity, corrosion, and other difficulties caused by the use of gasolene. One of the first problems was the development of a new solvent, from which all of the objectionable features had been removed. Gasolene (petrol) was out of the question, because no attempt was made by the refineries to remove unsaturated hydrocarbons, sulphur compounds, colour, and other objectionable factors. Naphtha (benzine) was a very great improvement, but still no real effort to remove the unsaturated and sulphur compounds was made by the manufacturers. great objection was the extreme fire hazard in the low boiling hydrocarbons."

Following some years of research, a heavy petroleum solvent was produced and adopted in America. It was named "Stoddard Solvent", after its discoverer. The solvent in the British Isles analogous to Stoddard solvent is white spirit. The adoption of white spirit on this side of the Atlantic was probably due more than anything else to a search for a cheaper solvent, at a time when the demand for the lighter petroleum products as petrols for the motor trade made the cost of the benzines somewhat high, while white spirit was in the nature of a by-product. One outlet for this surplus product was found in the need for a substitute for turpentine in the paint industry, and consequently white spirit is also called "turpentine substitute", or "painters' naphtha".

White spirit is a heavier spirit than benzine, with a specific gravity 0.80 (benzine 0.70 to 0.78). It is obtained during the refining of the gasolene distillate from the fractionation of crude petroleum oil, and is that portion obtained after the benzine fraction. It is an efficient solvent for grease, oils, and fats, as required by the specification of the ideal solvent. It is much less volatile than benzine, which means that evaporation losses are proportionately reduced. As with benzine, its main constituents belong to the aliphatic series of hydrocarbons, so that white spirit satisfies the condition of being inert and therefore above suspicion in its action on all kinds of textile materials.

The odour of white spirit is characteristic of the petroleum solvents, but it must be pointed out that the presence of impurities, such as sulphur compounds, are particularly undesirable, owing to the high temperatures required to remove them in the drying operation. In practice, the actual odour of the solvent is of little importance, provided the solvent can be entirely removed from the goods being cleaned without residual odour. Efficient dry cleaning does not transmit odour. Again the emphasis is on odourless cleaning.

White spirit conforms exactly to the condition of not being too volatile, and is an ideal solvent in so far that it has a very slow rate of evaporation at ordinary temperatures. In this respect, white spirit is the best of all the solvents in use for dry cleaning at the present time. White spirit hardly agrees with the desirable property of "not being too non-volatile". There is no doubt that many samples of white spirit have contained large proportions of constituents boiling at high temperatures, which are consequently insufficiently volatile

to be evaporated by use of reasonable heat. Many "white spirits" exhibit final boiling points above 200°C. It will be seen that the presence of such high boiling portions would entail considerable lengthening of the time of drying, with increased consumption of steam for heating purposes. The danger of residual odour due to the solvent itself left in the goods is very real when cleaning is being done with white spirit. Another point connected with the use of white spirit is that unless the final traces of solvent are removed entirely, an oily film may remain on the surface of the fibres, imparting a peculiar handle to the material. Recently there has been an endeavour on the part of the refiners to market a white spirit with an end or dry point much lower than 200°C.

Because of its slow rate of evaporation at ordinary temperatures, only minute quantities of vapour of white spirit are to be found in the atmosphere of rooms where it is used, and these quantities are insufficient to exert any physiological effect on operatives. The danger of asphyxiation with white spirit is very remote, although it must be realised that it can have definite physiological effects when its vapour is inhaled in sufficient quantity. Like benzine, white spirit can cause headache, giddiness, and sometimes sickness. Perhaps the most outstanding effect on one who was not acclimatised to it would be what, for want of a better description, might be called "taste", that is, the person effected imagines that a "taste" of the spirit is imparted to all food. Asphyxiation may take place during operations in enclosed spaces, such as when storage tanks are being cleaned. Because of this, it is recommended that storage tanks should be arranged so that the necessity for workmen entering them in order to clean out is avoided, and the tanks are cleaned by means of tools worked from the outside. In cases where this is not practicable, and a workman must enter the tank, the safest arrangement is for the top of the tank to be removed entirely; failing this, the manhole of the tank should be at least 27 ins. in diameter.

As already indicated, white spirit had an initial advantage of being much cheaper than benzine or solvent naphtha, the two solvents previously employed in the dry cleaning industry, but the margin between it and benzine has diminished until, at the present time, benzine is the cheaper by a fraction of a penny per gallon. In common with benzine and other petroleum products, it is subject to a

Government tax, which represents 100% ad valorem on the actual price of the spirit.

Nevertheless, the price of white spirit can be said to comply with the ideal condition of cheapness. The price of white spirit—1s.  $4\frac{1}{2}d$ . per gallon—is one advantage which can be claimed for this solvent in the comparison with the new non-inflammable solvents, which cost on an average 4s. 6d. per gallon.

There is no tendency on the part of white spirit to decompose or alter during storage. It might be said that some alteration is liable to take place in the hydrocarbons, which form the high-boiling fraction in white spirit. The effect of distillation may be to cause "cracking", or breaking down, of the higher members of the paraffin hydrocarbons into the lower members, but the products of this action would not effect the efficiency of the spirit for practical purposes.

White spirit is non-miscible with water, so that the methods of separation used with benzine may also be applied here. It will be found during this discussion on dry cleaning solvents that all these solvents are alike in being non-miscible with water. As the name implies, white spirit is colourless, or, to use the technical description, water white.

The flash point of white spirit is above the "legal danger" limit of 73°F. for petroleum spirit within the scope of the Petroleum (Consolidation) Act, 1928. This exemption from the restrictions placed on the use of petroleum can be taken as a measure of the safety, as regards fire, during the employment of white spirit. It is usual for white spirit to have a flash point in the neighbourhood of 100°F. The specification of Stoddard solvent shows a flash point of not less than 100°F., and the discoverer claims that this solvent is practically non-inflammable. The railway companies, however, classify white spirit as dangerously inflammable.

The crux of the matter is that the flash point is so high that the danger of fire from electric sparking is almost eliminated during the cleaning process, but danger may arise in any process involving the use of heat. Thus, in the modern drying tumbler where garments impregnated with spirit are moved about in a current of hot air, static electricity is likely to be generated in sufficient quantity to cause a spark, which would ignite the mixture of hot vapour and air within the machine and so cause an explosion. Because of this, every part

of a drying tumbler must be efficiently earthed and the machine fitted with explosion doors. As previously explained, these explosion doors operate valves on a steam line when opened, and so allow steam to be blown into the tumbler. In comparison with benzine, white spirit is considerably freer from risks of fire and explosion, but not absolutely immune from them, and therefore all the precautions applicable to benzine must also be observed with white spirit.

The distillation range of white spirit constitutes a disadvantage in this solvent's competition with other solvents, for not only does white spirit have high initial boiling point, but the presence of fractions of low volatility means that the spirit only becomes dry at high temperatures. The distillation range usually extends from 150°C. to 200°C., and consequently redistillation is more costly than with benzine, owing to heavier steam consumption. The use of white spirit entails the highest efficiency in extractors and drying arrangements. Redistillation can only be carried out economically by means of vacuum distillation, for the recovery of white spirit by distillation at ordinary pressure is often incomplete, owing to the large amount of "tailings" (unrecoverable residue in the still) which remain even after long use of live steam.

The Scott vacuum still, manufactured by Messrs. Scott & Son, Ltd., is typical of the plant especially suitable for the distillation of white spirit. The temperature at which any liquid boils is influenced by the pressure of the atmosphere on the surface of the liquid. The boiling points of liquids are usually expressed as the temperatures at which they boil under atmospheric pressure. The boiling point can be raised by increasing the pressure or lowered by decreasing the pressure. The lowering of the boiling point of white spirit by decreasing the pressure is the main feature of the Scott vacuum still. The Scott vacuum still is particularly applicable to the recovery of white spirit, but may be used for benzine or solvent naphtha.

The apparatus consists essentially of two vertical cylindrical vessels connected by means of a wide tube at the top. One vessel functions as the retort or still, while the other acts as the condenser. The still has a lower end in the form of an inverted cone, while the top is in the form of a flat dome, forming a space for vapour. The section of the retort cylinder just below the centre is tubular, and so arranged that steam circulates around the tubes, while the solvent, being

distilled, fills the tubes, which form communication between the cone bottom and the vapour space. There are a number of small evaporating tubes and evenly spaced between these are a few tubes of larger diameter. The larger ones act as down-take tubes, while the spirit passes up the smaller ones. Thus, a vigorous circulation of the liquor is obtained, which ensures the maximum heating effect.

The vapours from the still pass through a centrifugal spray arrestor (which prevents the spray of the liquor from splashing over out of the still), and from thence into a tubular feed heater. This appliance serves as an economiser of heat, in that the hot vapours pass over tubes containing the dirty solvent on its way to the still, and in so doing the heat of the vapours is transferred to the incoming liquor. The uncondensed vapours then enter the top of the second cylindrical vessel, which is the main condenser, and passes down small-bore vertical tubes, around which cooling water circulates, by entering at the bottom and being drawn off near the top of the cylinder. The cooling, of course, causes the vapours to condense to liquid. A pump in the series has the double function of exhausting the air from the apparatus and of withdrawing the solvent and delivering it to storage tanks. As the air exhausted from the still may contain uncondensed vapour, this is passed through a small vertical scrubber, wherein the solvent spray is trapped and returned to the storage tank.

One important accessory is an automatic feed control which controls the supply of solvent and maintains the liquor in the still at a fixed level. This has the advantage of reducing the amount of attention necessary to a minimum, while ensuring that the plant is working under optimum conditions.

The vacuum pump is usually driven by a directly connected steam engine, although in smaller plant it may be belt-driven. The heating of the still is by steam up to 120 lbs. per sq. inch pressure in the case of the heavy petroleum solvents, but the still would operate on exhaust steam in the case of benzine. The water from the condenser has value, where a supply of hot water is required, e.g. for boiler feed or dye-house supply.

Messrs. Scott & Son, Ltd., make vacuum stills of capacities from 25 gallons distilled spirit per hour up to 1,200 gallons per hour, and they claim the following advantages from the use of their equipment, as compared with ordinary wet distillation plant:

- (1) The distillation takes place without use of open steam. This gives a dry, bright distillate with a greatly reduced steam consumption, particularly when dealing with higher boiling point solvents, now coming into general use. The standard plant is designed so that the steam chest of the still is suitable for any steam pressure up to 120 lbs. per sq. inch., and can satisfactorily deal with white spirit having an end point up to 220°C., when using much lower pressure than this, actually 50 to 80 lbs. pressure.
- (2) The reduction in the amount of steam is also accompanied by an equivalent reduction in the volume of water required for condensing the solvent.
- (3) The Scott still, by reason of the fact that the steam does not come into contact with the spirit being distilled, gives practically dry distillate and the simple device adopted makes certain that there is no trace of water in the spirit as it leaves the plant.
- (4) The distillate is delivered from the plant at a low temperature (within about 1°C. of the temperature of the incoming condensing water). This minimises loss by evaporation from the solvent as it leaves the plant.
- (5) The vacuum still is not so difficult to control, and there is not the same liability to frothing troubles. With the Scott-Bennett feed control, the attention necessary is still further minimised, and when once the still is started up, it is possible to leave the still to work itself, except for occasional glances by the operator, until the time comes to discharge the sludge, which collects in the cone bottom.

It is of interest to note that the Scott still will work on benzine, white spirit, solvent naphtha, and trichlorethylene.

A performance test on a Scott still gave the following results:

Spirit distilled per hour				1,000 gall.
Steam used				1,200 lbs.
Water used				675 gall.
Temperature of water en	tering o	condens	er	52°F.
Temperature of water lea	ving co	ondense	r	184°F.
Temperature of distillate				54°F.

Specification of White Spirit.—The following is the actual specification of a white spirit, the distillers of which stress the high flash temperature

as a selling point, emphasising that "it is not so inflammable as petrol, so that one should save money on insurance".

Specific gravity	· · ·			• •		0.800
Flash point						100°F.
Colour					Wa	ter white
Initial boiling p	oint				• •	159°C.
Distillation 100	% at 16	S2°C.	Disti	illation	70% a	ıt 169°C.
	% " 16	-		,,		, 173°С.
	% ,, 16	-		,,	90%,	, 179°С.
	% " 16			,,	95%,	, 182°C.
	% ,, 16			,,	Dry ,	, 187°C.
" 6o°	% ,, 16	67°C.				

The Aromatic Hydrocarbons.—The second chemical group in the classification of dry cleaning solvents is the aromatic hydrocarbons. The two representatives of this division which find application in dry cleaning are benzol and solvent naphtha. These two solvents have many similarities to the petroleum solvents—benzine and white spirit—not only in their properties, but also in the method of refining from crude sources.

The main source of the aromatic hydrocarbons is the tar obtained during the distillation of coal in the manufacture of coal gas or coke. The distillation of coal gives rise to four products: (1) coal gas; (2) coal tar; (3) ammonia; and (4) coke.

The first patent for the destructive distillation of coal was obtained as long ago as 1681, the object being the production of coal tar and pitch, while Murdoch's patent for the distillation of coal, in order to produce coal gas, was dated 1792, at which time the condensed tar was regarded as more or less a waste product. In 1822, the distillation of coal tar was carried out in order to produce naphtha, which was used as an illuminating oil and as a solvent for rubber, this second application being the reason for the name "solvent naphtha". The possibilities of solvent naphtha as a grease solvent in dry cleaning do not appear to have been recognised until about 1856, when its introduction as a dry-cleaning agent coincided with that of petroleum benzine.

The working up or refining of the aromatic solvents from coal and coal tar is analogous with the refining of benzine from Scotch shale

and crude shale oil. As with the crude shale oil, the coal tar is redistilled and fractionated.

Coal tar is a thick black viscous fluid with a characteristic smell. The composition of the tar is influenced by many conditions, such as the type of retort used in the distillation of the coal; the temperature of distillation; and, naturally, the type of coal being treated. The tar still is usually an iron cylindrical vessel with a concave bottom and a dome-shaped top, from which an outlet pipe conducts the volatile gases into a condensing worm. The still may be direct fired. but more frequently the gas obtained in the previous distillation of the coal is used. The volatile constituents of coal tar are divided into four fractions, according to the temperatures at which they boil. Thus, the first fraction, known as the light oil, contains the oils which distil up to 170°C.; the second fraction, the middle oil, the constituents which distil up to 230°C.; the third or heavy oil fraction, those distilling up to 270°C.; and the last fraction, known as anthracene oil, the components of which distil up to 400°C. Each fraction is then subjected to redistillation and further fractionation. Benzol and solvent naphtha are obtained during the working up of the light oil fraction. The chief constituents of this oil are the aromatic hydrocarbons, benzene, toluene, and xylene. Other constituents are smaller quantities of certain aromatic compounds, such as pyridene bases and phenols.

The phenolic compounds form water soluble compounds with caustic soda, and in order to remove them, the light oil is washed with dilute soda solutions, while the pyridene bases are extracted by taking advantage of the fact that they form water soluble compounds with acids. Following the washing with caustic soda, the light oil is therefore treated with a dilute solution of sulphuric acid. As a small amount of compounds known as cumarones still remain, a final washing with concentrated sulphuric acid is given, and then the oil is ready for redistillation and fractionation.

The number of fractions into which the light oil is divided may vary according to the object in view. One German authority shows eight fractions, but four fractions are the average practice. The first fraction is known commercially as "90s. benzol"; the second as "50s. benzol"; the third as "heavy benzol"; and the fourth as "solvent naphtha". The 90s. benzol, 50s. benzol, and solvent naphtha are all employed in dry cleaning. The figures 90 and 50 indicated the

percentage of the solvent distilling at 100°C. The 90s. benzol is the most suitable for dry cleaning.

The benzols are light liquids of specific gravity approximately 0.88. They are excellent solvents for grease, and are highly volatile. Loss by evaporation can only be kept within economical limits by use of special apparatus, such as totally enclosed circuit systems. The benzols satisfy the conditions of being chemically inert and without action on all kinds of textile fibres. The odour of the benzols is quite pleasant. With regard to volatility, the benzols are perhaps too volatile, while, at the other end of the scale, the occurrence of compounds of low volatility is rare, so that the end point is reached about 120°C. The toxicity of the benzols is a source of danger. It has been shown that, owing to the large amount of benzene present, both 90s. and 50s. benzols are definitely toxic and have a distinct poisoning effect on the blood and the brain centres. Female workers are less resistant than males, and the vapours of benzol, even in small doses, may lead to chronic anæmia, especially in the case of young female workers. Large doses may have fatal results.

The price of the benzols, 1s. 8d. per gall., places them at a disadvantage, in comparison with the petroleum solvents, 1s.  $3\frac{1}{2}d$ . and 1s.  $4\frac{1}{2}d$ . per gall., and is probably the chief reason why the aromatic solvents have fallen into disuse within recent times.

The benzols are quite stable to storage and repeated distillation. They are also non-miscible with water and are water white, as required in the specification of the perfect solvent.

The flash point of the benzols must be classed as a disadvantage. Both the 90s. and 50s. benzols give off highly inflammable vapours at all temperatures, their flash points being below o°C.

Benzols usually show quite a close distillation range, distillation taking place between 80°C. and 120°C., so that they present no difficulty in drying out and distillation.

Solvent naphtha stands in the same relation to the benzols as white spirit does to benzine. The differences in properties are of degree only. As a grease solvent, it is equally as efficient as benzol, but, being a higher distillation fraction, it is less volatile, and consequently the loss incidental to evaporation is reduced. It is inert towards all kinds of textile fibres. It has quite a sweet odour.

Owing to its comparatively high boiling point, solvent naphtha cannot be considered as being too volatile, but may be at a slight disadvantage from the presence of compounds of low volatility, so that the complete drying out and deodorising entail longer time and greater consumption of steam than with benzol. The boiling point varies between 120°C. to 170°C. Solvent naphtha is usually sold as solvent naphtha 90% at 160°C., indicating that 90% of the solvent distils below 160°C. The toxicity of solvent naphtha is considerably less than that of the benzols, partially due to the lower volatility, but mainly due to the fact that the percentage of benzene is lower, while that of toluene and xylene is increased, the two latter aromatic compounds being much less poisonous than benzene. The vapours of solvent naphtha are practically harmless.

The cost of solvent naphtha 90% at  $160^{\circ}$ C. is 1s.  $6\frac{1}{2}d$ . per gall., so that it is considerably cheaper than the non-inflammable solvents, but slightly dearer than white spirit. Solvent naphtha satisfies the condition of being stable to storage and repeated distillation. It is non-miscible with water, so that recovery by chemical means is easy and economical. It is also without colour.

The flash point of solvent naphtha is much higher than that of 90s. benzol, but is still too low for an ideal solvent. Its flash point is about 20°C., and consequently solvent naphtha gives off an inflammable vapour at all temperatures. The distillation range for solvent naphtha is what might be termed close range, usually extending over 40°C. The position of the distillation range on the centigrade scale is somewhat high, the average being between 125°C. to 165°C. As a result of the moderately high final boiling point, this solvent can only be efficiently and economically distilled under vacuum.

## THE NON-INFL'AMMABLE SOLVENTS

The third group in the classification of dry cleaning solvents includes all the recently introduced non-inflammable solvents. The classification due to non-inflammability coincides with a chemical one, as all these solvents belong to the chemical class of chlorhydrocarbons. The chlorhydrocarbons, so clearly differentiated from the two other classes reviewed, by absolute freedom from the risk of fire, exhibit another difference. The inflammable solvents of the petroleum series and aromatic series are obtained from natural sources, but the

chlorhydro-carbons are synthetic products, having to be manufactured in chemical works. This difference in origin is the cause of the great hiatus between the costs of the two kinds of solvents.

The chlorhydrocarbons in use as dry cleaning agents at the present time are carbon tetrachloride and trichlorethylene principally, and perchlorethylene to a much lesser degree. They are derivatives of the paraffin series of hydrocarbons, but belong to two different branches of them. The parent substance of carbon tetrachloride is methane and the family relationship is indicated by the alternative and more strictly correct chemical name of tetrachloromethane. The chemical comparison with chloroform, which is trichloromethane, explains the similarity between the two compounds. Trichlorethylene belongs to another branch of the paraffins, and is a derivative of the olefine or unsaturated hydrocarbons, the parent substance of which is ethylene. These differences in the compositions of carbon tetrachloride and trichlorethylene are mentioned here because they afford some indication of the cause of many variations in the properties of these two solvents, which will be brought out by comparison with the "ideal solvent".

# CARBON TETRACHLORIDE

This synthetic solvent has been used in Germany since 1890 as a spotting agent, under the trade-name "Katharin". Its preparation was the subject of many patents between 1903 and 1912, and it was marketed under several names during that period. It is known industrially as phoenixine, spectral, tetra, tetracol, tetraform, katarine, econol, asordine, and zoric fluid.

Carbon tetrachloride is synthetically prepared by the action of sulphur chloride on carbon bisulphide. Any impurity would most probably be sulphur, but the commercial product is generally a pure compound, a marked contrast to the properties of previous solvents. It is a clear spirit of specific gravity 1.6. This high specific gravity must be borne in mind when comparisons of solvent consumption are being made. It is usual to express solvent consumption as a percentage of the weight of goods cleaned. Thus, equal percentage losses for white spirit (sp. gr. 0.8) and carbon tetrachloride (sp. gr. 1.6) would mean that the actual quantity in gallons of white spirit used would be twice that of the carbon tetrachloride.

The "ideal solvent" is required to be an efficient grease remover,

5 [65]

and carbon tetrachloride fulfils this condition completely. It is an excellent and rapid solvent for all kinds of grease, fats, oils, and resins. Its solvent action is greater than that of the inflammable solvents, and because of this, the time of processing with carbon tetrachloride is shorter.

In comparison with the inflammable solvents, carbon tetrachloride is extremely volatile and rapidly evaporates even at ordinary temperatures. This high volatility has been the peculiarity which has delayed the commercial application of carbon tetrachloride for dry cleaning. It must be remembered that this solvent has been produced commercially for more than thirty years, yet its adoption for dry cleaning is quite recent. The problem of its successful application was one for chemical engineers, and within recent years there has been almost a glut of installations designed specially for the use of carbon tetrachloride. The fundamental principle throughout these new plants is that of totally enclosed cleaning systems, in which cleaning, drying, deodorising, and recovery are carried to completion with the system kept entirely vapour tight. It is only by employment in such an installation that carbon tetrachloride can be used on any commercial scale. The author's reference to the "ideal solvent" for dry cleaning explained that the cleaning medium passes through a series of changes: first, it is a liquid; secondly, a vapour due to evaporation of the liquid; and, finally, a liquid due to condensation of the vapour from the evaporation process. Because of its high volatility, carbon tetrachloride passes easily and rapidly through the varying phases as required in the dry cleaning process.

The next point of comparison with the ideal specification refers to the requirement of chemical inertness, that is, without action or effect on any kind of textile fibre. During the consideration of dry cleaning solvents, especially the chlorhydrocarbons, it will be seen that the necessity of being chemically inert to textile fibres has an increased importance, not only in connection with the action of the solvent on the various fibres, but also in reference to solvent action on colours, which are peculiar to certain textile material.

Dyestuffs of the vat class, or of the azoic class, may be slightly soluble in certain solvents, and yet be completely fast to water. It is doubtful, however, whether these occur sufficiently often enough to be noted. Should such sensitive dyestuffs be used on a fabric it would be safe to say that they would be affected by any dry cleaning solvent to a more

or less extent. In general, it may be said that carbon tetrachloride is above suspicion in respect of its action on all kinds of textile fibres and colours, including dyed cellulose acetate, a property which is in contrast to that of trichlorethylene, and which causes the displacement of the latter solvent by carbon tetrachloride under certain circumstances.

The odour of carbon tetrachloride is peculiarly sweet, resembling that of chloroform. All the chlorhydrocarbons possess the same characteristic odour. Carbon tetrachloride satisfies the specification in being free from offensive odour. Users of carbon tetrachloride rightly claim that their method of dry cleaning is odourless. Apart from the fact that the odour of carbon tetrachloride is quite pleasant, the high volatility makes the complete removal of the solvent by evaporation easy, so that there is no fear of a residual portion remaining in goods after they have been treated.

Carbon tetrachloride has a fixed boiling point of 76.8°C. Commercial samples usually boil within 1° of this. It is, however, extremely volatile at all temperatures, and consequently does not agree with the condition of "not being too volatile" so well as the older solvents. The volatility of carbon tetrachloride not only compels the use of totally enclosed systems, but entails the most meticulous care that the systems and pipe lines are maintained vapour-tight and free from leakage. The antithesis of the previous condition, that of "not being too non-volatile", is in complete agreement with the properties of carbon tetrachloride. The fixed boiling point of this solvent excludes the possibility of non-volatile portions, such as are met with in the case of the solvents of the petroleum type and aromatic hydrocarbons. The fixed boiling point gives the non-inflammable solvents a distinct advantage over the other solvents in that the steam consumption necessary for the drying-out process is considerably less.

The toxicity of the vapours of carbon tetrachloride brings this new solvent under suspicion, and is the cause of much hostile criticism. Its higher volatility is closely connected with increased physiological effect. This physiological effect is of great importance and must be fully recognised by all those interested in the solvent's application. Carbon tetrachloride was employed in the sixties of last century as a remedy for headache and neuralgia, and it was some time before its action was closely studied and its physiological action realised. In 1910 a young woman collapsed and died in a London hairdressing

saloon after her hair had been shampooed, and it was found that the shampooing fluid was carbon tetrachloride. This gave rise to a series of investigations, from which it was discovered that the solvent was definitely toxic when inhaled to any great extent.

The first indication of physiological action is anæsthesia of the victim, comparable with the insensibility caused by chloroform, but the time of exposure necessary to produce this is much longer than in the case of chloroform. Compared with chloroform, the insensibility is less profound, but, on the other hand, the effects are more lasting. Carbon tetrachloride has an injurious effect on certain organs, particularly the liver, causing necrosis of that organ. It acts as an irritant to the bronchial tubes and its inhalation may cause bronchitis and bronchial pneumonia, as well as nervous disorders, and may ultimately lead to death. Symptoms of carbon tetrachloride poisoning are sickness, loss of weight, irritation of the eyes, nose and throat, and inflammation of the skin. One authority has declared that "a proportion of 15 milligrammes per litre of air is sufficient to bring on sickness". With carbon tetrachloride, the amount necessary to cause insensibility is dangerously near the permanently harmful dose. It will be seen later, from the discussion on the physiological effect of trichlorethylene, that there is in this case a wide margin of safety between the anæsthetic. or narcotic dose, and the harmful or toxic dose, so that the collapse of the patient serves as a warning. This warning by narcosis is absent in the case of exposure to carbon tetrachloride. The odour of carbon tetrachloride should serve as a warning of its presence in the atmosphere, but "familiarity breeds contempt", and consequently this warning may pass unheeded. All users of the solvent should make a point of studying its physiological action and, as a result of this understanding, take every care that no vapours are allowed to be present in the atmosphere of workrooms.

The volatility of the solvent has compelled the makers of plant to design the machines to be vapour-tight and easily maintained; this type of machine reduces the toxic danger to a minimum. The real danger can only occur during the cleaning out of pipe lines, filters, and storage tanks. It cannot be too much emphasised that prolonged exposure is usually necessary before any appreciable physiological effect is observed, so that with any reasonably efficient installation such accidents as chronic injury should be a remote possibility. It is

interesting to note that workers who have been acclimatised to the petroleum solvents are not more resistant to the effect of carbon tetrachloride than new-comers. Yet another physiological effect of carbon tetrachloride is due to its high solvent action—it readily absorbs the fat of the skin, and so removes the fatty protective layer. The unprotected skin offers no resistance to the entry of poisons, and consequently grave risk may be incurred in this way.

The American Medical Association has issued a report of the investigations of Dr. L. D. McGuire, of Boston, who has been observing the physiological reactions of workers in dry cleaning establishments using carbon tetrachloride. The report, as quoted in *The Scientific American*, declares that carbon tetrachloride, extensively used in dry cleaning, is particularly poisonous to persons who drink alcoholic beverages, or who do not get enough calcium in their food. Dr. McGuire cites the case of seven workers in a felt manufacturing plant, all wine drinkers, who were poisoned by this substance when they used it in large quantities for cleaning felt. When used in small quantities to remove individual spots, it had no ill effect, but when the felt was passed through a large warm bath of fluid all the men fell ill, and one nearly died. Dr. McGuire states that calcium lactate or calcium chloride are successful remedies in the treatment of carbon tetrachloride poisoning.

This report indicates that, in the case under review, the solvent was being used in complete disregard or ignorance of its toxic nature. There is a point of interest about the use of calcium lactate as a remedy, for it supports the practice of allowing operatives to drink milk as an antidote to possible toxic effect.

VAPOUR.	Kills in short ti		Dangerous in 30-60 mins.		Maximum amount for 60 mins. without injury.		Maximum amount for prolonged exposure.	
	% Volume	Rela- tive Order	% Volume	Rela- tive Order	% Volume	Rela- tive Order	% Volume	Rela- tive Order
Ammonia	·5 to 1	9	· <b>25-</b> ·45	10	.03	8	.01	6
Benzene	1.9	10			·31-·47	11	.1231	13
Benzine	2.4	ΙI	1.1-5.5	II	·43-·71	14	?	
Chloroform	6.8-8.2	13	1.4	12	·5-·6	13	•2	8
Carbontetra-								
chloride	4.8-6.3	14	2.4-3.5	14	·4-·6	12	∙16	11

The United States Public Health Service, in Bulletin No. 185 of 1929, published the table on the previous page showing, the physiological response to various concentrations of some common gases and vapours. From this it will be noted that carbon tetrachloride is not as poisonous as ammonia or benzene.

Carbon tetrachloride may be bought in bulk at an average price of 4s. 6d. per gall. It is noteworthy that the non-inflammable solvents are usually quoted at a price "per ton", and that owing to the various specific gravities these quotations may be misleading. Obviously, the only rational method of comparison is the rate per gallon. At 4s. 6d. per gall., carbon tetrachloride is more than three times the price of the petroleum solvents. This high cost may be classed as an initial disadvantage, but offset against this handicap, some compensation is obtained by the fact that the solvent readily lends itself to a very fine degree of recovery. During the operation of an efficient installation, the recovery of the carbon tetrachloride is almost complete. Even the small amount of solvent that is driven off during the deodourising process can be reclaimed by use of chemical absorbers of the activated carbon type. The author is of the opinion that the price of carbon tetrachloride will tend to become lower as manufacturers take up its production on a large scale.

Item 9 in the ideal specification requires the solvent to be stable to storage and repeated distillation. The behaviour of carbon tetrachloride in this respect calls for special comment, since it is greatly influenced by certain circumstances. This solvent is only stable when pure and free from moisture. In the presence of moisture and in contact with certain metals, especially iron, it tends to decompose, with the liberation of hydrochloric acid and a highly poisonous gas, phosgene. Hydrochloric acid, liberated within a closed cleaning circuit, would produce disastrous results, not only on the goods being cleaned, but also on the metallic parts of machinery. This possibility has compelled engineers to construct the machines designed for carbon tetrachloride cleaning from special metals.

It has been found that monel metal is the most suitable for this type of plant, and, in consequence, it is customary to use monel metal wherever possible. Other metals, such as heavy-gauge copper, phosphor bronze and nickel, have been used. It will be seen that the machines for this solvent are necessarily of solid construction, and this

causes the cost of carbon tetrachloride installations to be comparatively high. Many interesting experiments are being conducted in Germany and America, as well as in this country, with the object of testing the action of carbon tetrachloride on various alloys, particularly those containing a relatively high percentage of silicon. It is, however, too early to give any indication of what the results may show.

An article in *The Dyer* (3 March 1933) on "Metal Corrosion due to Carbon Tetrachloride", states: "Expert opinion favours the employment of nickel alloys for the construction of linings to vessels, or special fittings. Now that nickel steels containing 10% nickel are obtainable at attractive figures, the substitution of this metal for those easily attacked is recommended. Monel metal, nickel copper alloy, is specially suitable for resisting attack, and on account of its high tensile strength it is very popular."

Some results of corrosion tests with carbon tetrachloride are given in the following table, expressed as change in weight in milligrammes per square centimetre per 24 hours:

Metal.	Dry	Solvent.	Wet Solvent.		
wetar.	15°C.	5°C. Boiling Point.		Boiling Point.	
Mild Steel		-0.03	+0.03	-6.7	
Copper	Nil	+0.05	+0.02	-8.7	
Brass	Nil	+0.01	+0.03	-o·3	
Tin	Nil	-0.09	+0.07	-1.1	
Zinc	Nil	-0.03	+0.1		
Aluminium	Nil	Totally	+0.03	+0.05	
		Disintegrated			
Lead	Nil	-0.03	+0.03	-o·4	
Monel	Nil	+0.01	+0.01	0.13	

The stimulating effect of moisture on the corrosion of metal by carbon tetrachloride is important and entails additional attention to the avoidance of moisture within the cleaning system. This calls for care in two directions: first, in order to prevent the introduction of moisture into the cleaning drum, and, secondly, to avoid the collection of water in the cleaning circuit. It may be said that moisture is never

absent from dry cleaning machines, and this is particularly true in the case of treatment of woollen garments. Most articles hold a remarkably high content of water without feeling damp, so that the goods themselves would be the means of introducing water into the machine. It is, therefore, essential that the moisture content in the goods should be removed. Cleaning by carbon tetrachloride must include the initial process of pre-drying, so that a drying tumbler is a very necessary part of the installation. This process is in marked contrast to the normal practice in dry cleaning with petroleum products. It will be remembered that in the latter case pre-drying is deprecated on the ground that it increases the danger from static electricity, a potent cause of fire and explosion.

With the precaution of pre-drying, the risk of injury to the metal of the cleaning machine and to the material of goods being cleaned is almost entirely obviated. The author has made repeated experiments in an endeavour to detect the presence of acid within the cleaning drum. The method used was to include in the ordinary cleaning load some pieces of silk and cotton material which had been dyed with Congo red. In every case no change in the colour of the Congo red was observed, which would lead to the inference that with pre-drying the cleaning drum could be kept free from acid.

Even with pre-drying, there is still the possibility of minute quantities of water collecting in certain parts of the installation until their cumulative effects are appreciable. The majority of carbon tetrachloride plants are designed to give continuous filtration of the soiled spirit. The dirty solvent is passed through cotton filters, either in the form of long tubes, or squares of material stretched over metallic gauze. These filters act as collectors of moisture and the resultant decomposition of the solvent and the liberation of hydrochloric acid is made evident by rotting of the cotton fabric of the filter. In order to avoid such accidents as the bursting of a rotted filter, it is necessary that the filter bags or sheets should be removed and washed at regular and frequent intervals, immediate replacement being made of any showing signs of "pitting".

Yet another characteristic of carbon tetrachloride installations is a series of traps for moisture within the circuit of the solvent. These traps are based on the same principle as the separator described under "Benzine", but in this case the water, being lighter than the solvent,

is drawn off at a point higher than the surface of the solvent. Experience has shown that the corrosion due to the decomposition of the solvent invariably occurs in the accessories to the cleaning drum and not in the cage itself.

In common with all other dry cleaning solvents, carbon tetrachloride is not miscible with water, so that methods of recovery involving washing with solutions of soda lye are applicable. This property also enables the solvent absorbed by activated carbon to be recovered by live steam. It is hardly necessary to add that carbon tetrachloride is colourless, as demanded by item 11 in the specification of the ideal solvent.

Carbon tetrachloride has no flash point. It cannot be ignited, is absolutely non-inflammable and non-explosive. Its peculiarity in this respect is indicated by the fact that it finds employment as a fire extinguisher in certain motor-car appliances, for example, the Pyrene extinguisher. The non-inflammability of the chlorhydrocarbons gives them a big advantage over the older solvents. This is certainly an enormous advance over such a highly inflammable substance as benzine. It must be admitted that the immunity from danger of fire and the entire absence of Home Office regulations are greatly attractive, and have been the fundamental reason why these new solvents have experienced such an increasing adoption, especially among laundries.

Yet another sharp contrast to the previous solvents is shown by carbon tetrachloride in that it has no distillation range. Being a single compound instead of a mixture of compounds, it distils entirely at one temperature, 76.8°C. This boiling point is much lower than the initial boiling point temperature of the majority of the inflammable solvents, so that the consumption of steam during distillation is very slight, and provides some degree of recompense to counter-balance the high initial cost of the solvent.

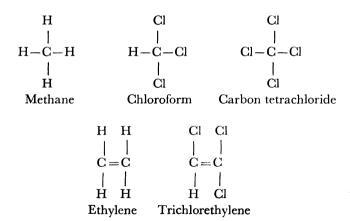
#### TRICHLORETHYLENE

The second product in the series of non-inflammable dry cleaning solvents is trichlorethylene. This, together with carbon tetrachloride, is one of the principal non-inflammable solvents in use at the present time. It has been quite largely adopted on the Continent, particularly in Germany, where its production has been the subject of many patents

since 1908. It has been used in numerous important syntheses, including that of indigo.

In common with all the non-inflammable solvents and in contrast to the inflammable solvents, such as the petroleum liquids and aromatic hydrocarbons, trichlorethylene is a manufactured product. One method of preparation, largely used in Germany, is an electro-chemical one in which carbon, lime, and salt are the raw materials. Trichlorethylene is composed of carbon, hydrogen, and chlorine, which explains the term "chlorhydrocarbon". Carbon tetrachloride may be described as methane with all its four hydrogen atoms replaced by chlorine, while trichlorethylene is ethylene with three hydrogen atoms substituted by chlorine atoms. The relationship of these two solvents to chloroform is demonstrated by the fact that chloroform is methane with three hydrogen atoms replaced by chlorine atoms.

These three compounds and their parent substances may be shown graphically as follows:



Trichlorethylene is known industrially as "Westrosol", "Tri", "Triklone", and "Trielene". The word "tri" for trichlorethylene is accepted throughout Germany, and the term is being widely adopted in this country. The commercial product is practically pure trichlorethylene, the freedom from admixture with other substances forming another point of agreement with the characteristics of carbon tetrachloride and one of contrast to the inflammable solvents.

Trichlorethylene is a clear spirit of specific gravity 1·47, which compares with sp. gr. 1·6 for carbon tetrachloride, and 0·7 to 0·8 for the petroleum solvents. As previously indicated, these specific gravities are important when the relative consumptions of solvents are being compared by determining the loss of solvent as a percentage of the weight of goods cleaned. Thus, the non-inflammable solvents would show percentages by weight double those of the inflammable solvents for equal losses expressed in gallons.

Trichlorethylene is probably the best solvent for grease and oil of all the fluids in use for dry cleaning at the present time. It has a thorough and rapid solvent action on all kinds of fats, grease, and oil, and will also dissolve many resins, tarry and bituminous substances, grass stains, paints, and lacquers. The great variety of substances which are dissolved by trichlorethylene is the cause of the remarkable efficiency of this solvent for dry cleaning. For example, it is the only solvent that has an appreciable effect on stains due to paint, grass, and tar, so that these stains rarely remain after processing with trichlorethylene, and thus the need for "spotting" is reduced considerably.

Because of its solvent action on grease, trichlorethylene is often employed, in conjunction with certain forms of soap, in the manufacture of preparations specially marketed for the purpose of removing grease in ordinary washing. There are many of these so-called grease-solvent-soaps now being used in the laundry industry and by engineers and motorists. The solvent action of trichlorethylene is more powerful and, therefore, quicker than that of the other solvents, so that the time of cleaning is shorter. Trichlorethylene is extensively used in metal de-greasing, and the greater portion of the output of "tri" is used in the metal industry.

Trichlorethylene is extremely volatile, giving off a heavy vapour at all temperatures. As is the case with all the chlorhydrocarbon solvents, evaporation losses can only be kept within reasonable limits by the employment of totally enclosed cleaning systems. Plants for the application of trichlorethylene are specially designed to avoid evaporation losses, in that they must be perfectly vapour-tight and easily maintained so. It is interesting to note that the firm holding the monopoly for the production of trichlorethylene in Germany has designed an installation essentially for the application of this solvent in dry cleaning. This plan seems to indicate the existence of that

happy co-operation between the chemist and the engineer, which is necessary for the successful application of the non-inflammable solvents to dry cleaning on a commercial scale. The high volatility of trichlorethylene enables the solvent to pass readily and easily through the varying phases of the cleaning operation.

The third item in the specification of the ideal dry cleaning solvent was the requirement of chemical inertness. It cannot be said that trichlorethylene conforms to this under all conditions. It is therefore imperative that the conditions under which trichlorethylene is not inert be fully understood and consequently avoided. The introduction of artificial silks, particularly those of the cellulose acetate type, has caused many modifications in the various methods of treating garments, both in the laundry industry and in the dry cleaning and dyeing industry. Many chemicals which had previously been used as spotting agents were found to be injurious to articles of cellulose acetate. It is well known that acetate silk is unique in many of its characteristics, and is subject to very definite limitations in comparison with other textile fibres. The launderer and the dyer and cleaner have been compelled to adjust their processes so that this material can be treated successfully.

It is particularly necessary to understand the limitations of cellulose acetate when processing it with trichlorethylene. Trichlorethylene is quite harmless to cellulose acetate at the ordinary temperature. This can be proved in many ways, but the most obvious evidence is the success of the Burtol system of dry cleaning which is being carried out in so many shop windows at the present time. The fluid used in the Burtol system is trichlorethylene and the temperature of the process is the normal temperature throughout. This system encounters no trouble in the treatment of cellulose acetate.

In a booklet issued by the British Celanese, Ltd., entitled *The Laundering and Cleaning of Celanese*, the reagents in common use for cleaning or spotting are classified as being safe or not safe for "Celanese". The non-inflammable solvents, carbon tetrachloride and trichlorethylene, are included among the safe reagents. It has been found that "Celanese" is unimpaired even after twelve hours' immersion in trichlorethylene.

Many experiments have been conducted to determine the exact conditions for the processing of cellulose acetate with trichlorethylene.

In the issue of Rayon for June 1932, there is an interesting account

of an experiment by a German investigator, Brandenburger, and the action of various solvents on cellulose acetate is expressed in figures, the figure 100 indicating the limit of safety, so that a solvent having an index figure below 100 would be without harm on this class of artificial silk. Brandenburger's results are tabulated as follows:

Carbon tetrachloride	14.7	Nitrobenzene	143.0	
Toluene	27.3	Acetyl chloride	215.0	Lincafe
Chlorobenzene	29.9   Safa	Dichloroacetate	293.0	
Benzylchloride	29.9 37.9 ≻Safe	Ethylenedichloride	615.0	
Benzene	47.2			
Trichlorethylene	81.3			

Imperial Chemical Industries, Ltd., have made a series of experiments, the results being published in their book, *Dry Cleaning with Triklone*. Hanks of various textile fabrics were soaked in trichlorethylene for 36 hours and then dried, all operations being conducted at room temperature. When the hanks were dry, the tensile strength of each material was measured by means of the breaking test on the Ballistic balance. For the sake of comparison, similar tests were carried out with carbon tetrachloride instead of trichlorethylene.

TENSILE STRENGTH OF MATERIALS WORK DONE IN INCH-POUNDS IN BREAKING THE MATERIAL

	Number	Count	Mean Reading on Ballistic Balance		
Material	of Threads	or Denier	Untreated	Treated with "Triklone" (as delivered)	
			inlb.	inlb.	
Cotton (bleached)	30	Count=	41.7	41.0	
		230			
Natural silk (spun)	20	Denier	75.6	75.8	
(boiled off)		120			
Viscose	25	120	66.4	70.2	
Cellulose acetate					
silk (soaped)	22	120	45.6	45·o	

Yet another series of investigations has been carried out by the American Government Research Institute, with which is incorporated the American Bureau of Standards. These investigations were carried out over a period of four months, and have resulted in the issue of numerous and very exhaustive reports on the effect of various solvents used in dry cleaning. Only one of these reports has bearing upon the present subject, and that is the one which deals with the tests on

	No. of days On which the Carbon Tetrachloride.					
Fabric	Washing Processes	goods were	Special Quality	Ord. Trade Goods	Trichlor- ethylene	Benzine
Wool	0	О	100	100	100	100
	3	20	103	103	109	101
	7	75	102	108	109	109
	10	126	101	100	102	104
Natural Silk	o	o	100	100	100	100
	3	20	94	96	102	92
	7	75	93	88	107	98
	10	126	86	90	92	96
Cotton	О	o	100	100	100	100
	3	20	99	94	102	99
	7	75	96	87	96	98
	10	126	83	84	102	93
Viscose Silk	o	o	100	100	100	100
	3	20	101	101	100	100
	7	75	95	96	96	98
	10	126	83	92	82	99
Acetate Silk	o	o	100	100	100	100
	3	20	104	87	110	94
	7	75	98	84	112	78
	10	126	102	80	98	83

the different capacities for resistance of fabrics before and after treatment with the various solvents. The report of the Institute issued in January 1932 explains that the tests comprised the repeated cleaning of various materials in a drum, centrifuging, drying, and then exposing the test sample to light in a moist condition, so that the condition of the fabrics between each cleaning operation would be similar to that of the fabric in wear. The effect of mechanical action was determined by submitting the fabrics to the same movement as previously, but without the use of solvent. The results are expressed in values showing the resistance capacities, where 100 represents the resistance capacities after the mechanical treatment without solvents.

The American report recommends that the fabrics should not be left in trichlorethylene for more than 30 minutes, as, after this period, the resistance capacity of this material may be reduced, but the fabrics regain their original resistance capacity on drying. In actual practice, garments being cleaned in trichlorethylene do not require a longer processing time than 30 minutes. It should be noted that, with the machines now employed, the fabrics are stirred in the solvent for 10 minutes only, and the whole cleaning process, dust-removing, and drying hardly occupy three-quarters of an hour.

So far the effect of trichlorethylene on the cellulose acetate structure itself has been discussed, and it can be clearly stated that for practical purposes trichlorethylene is harmless to this kind of artificial silk, but there is still another aspect of the relationship between acetate silk and trichlorethylene. There is a definite suspicion that trichlorethylene may exert a solvent action upon the colours used on acetate silks. Without doubt certain special dyes used on cellulose acetate are liable to bleed in trichlorethylene if the time of processing is prolonged unduly, especially in the case of very full shades. Dyestuffs of the basic class are the most readily affected, but this is the case with all the other solvents, more or less. Red and violet shades may discolour the solvent, but the amount of bleeding is rarely perceptible when the goods are dried. Once again reference must be made to the Burtol system of cleaning. The Burtol plants have no difficulty in handling coloured cellulose acetate.

The use of trichlorethylene calls for very careful control in the case of garments containing cellulose acetate. Cleaning of cellulose acetate

by means of trichlorethylene can be carried out with safety provided strict control of moisture, temperature, and time is maintained.

The solvent must be kept perfectly dry, and this is best assured by pre-drying the goods to be treated. A dry cleaner's tumbler would have the double effect of removing loose dust and drying out the moisture. Although live steam should not be necessary for the distillation of trichlorethylene, it is sometimes employed for the distillation of solvent from still sludge. In this case, care must be taken that the separation from the condensed steam is as complete as possible. Not only must the separator be accurately set, but the time for settling out of the solvent must be sufficient to give a clear solvent free from cloudiness.

Temperature is the most important factor controlling the solvent action of trichlorethylene on dyes used on cellulose acetate. It has already been emphasised that these dyes are soluble in trichlorethylene at high temperatures. This property is recognised by those dry cleaners who have installed refrigerating plants in order to cool the trichlorethylene below normal. The drying-out stage of the dry cleaning process is the real danger point. This danger point is in the region of 65°C. In the case of garments, which are classified as silks for dry cleaning, the "safety first" method is to extract the goods and dry them at ordinary temperatures.

In the case of totally enclosed installations, this entails opening the machine, when it is full of solvent vapour and removing the "wet" garments. This obviously means loss of solvent and risk to the health of the operator by inhaling the fumes. The loss of solvent could, however, be avoided by the installation of activated carbon absorbers to treat the "air" from machines and drying-rooms.

The time that trichlorethylene is in contact with cellulose acetate must be the absolute minimum. In view of the high solvent power of trichlorethylene, the cleaning time need only be about 10 minutes. It is very important that, if "silks" are removed after extraction, they should not be allowed to lie about (such as over a meal-time), but hung up immediately.

A drying apparatus heated by exhaust steam, and using a large fan to give a rapid movement of air through the cleaning drum, is the best alternative to drying out at normal temperature.

There are many systems of non-inflammable cleaning being sold to-day without any stipulation as to the solvent to be used. The

makers of these installations leave the choice of solvent to the purchaser. It should, however, be fully realised that the conditions under which carbon tetrachloride can be used with safety are entirely different from those which apply to trichlorethylene. It is not to be expected that an installation can be used with either solvent without some alteration in the control of temperature. With an appliance expressly designed for the employment of trichlorethylene, there is practically no danger in the cleaning of cellulose acetate, so that the results are better than those of any other process, especially on silk goods. In the hands of an experienced dry cleaner, trichlorethylene produces excellent results.

Trichlorethylene conforms to the third requirement in the specification of the ideal solvent only under certain very definite conditions, which must be fully understood in order that cellulose acetate articles may be successfully treated.

The fourth requirement of the ideal solvent is that of being free from offensive odour. In common with all the other chlorhydrocarbons, trichlorethylene has a peculiarly sweet smell resembling that of chloroform. The odour is distinctly pleasant to certain workers, who are attracted to it, and in whom the desire to smell the fumes amounts almost to a craving. It has been found in metal de-greasing works in Germany, where large amounts of "tri" are used, that workers are loathe to be transferred to other departments, and, if transferred, frequently ask to be returned to the de-greasing machines. There is a recorded instance of a female employee from another section of a metal works being found by the de-greasing machines, and, when rebuked, stating that she could not live without the smell of the "tri". The fascination may increase until the victim contracts what has been termed "tri mania", in which state there may be a desire actually to drink the solvent, with disastrous and often fatal results.

In dry cleaning practice there is no risk of any residual odour, even a pleasant one, remaining in garments after treatment with "tri". The high volatility of the solvent ensures that every trace is removed, even at ordinary temperature. Trichlorethylene most definitely conforms to the fourth demand in the ideal specification, in that it is free from offensive odour and cleaning by this solvent is absolutely odourless.

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The ideal solvent is required to be "not too volatile". With a boiling point of 87°C. and a rapid rate of evaporation, even at ordinary temperatures, trichlorethylene does not fulfil this condition so well as the older solvents of benzine or white spirit type. This high volatility delayed the introduction of the non-inflammable solvents for many years until chemical engineers overcame the difficulty of preventing loss by evaporation. The problem was solved by the adoption of totally enclosed cleaning systems, in which every phase of the dry cleaning process is carried through, under conditions of vapour tightness. It has already been emphasised that the commercial application of the non-inflammable solvents not only compels the use of totally enclosed systems, but also enforces strict attention to the maintenance of the systems, joints, and pipe lines in a vapour-tight condition. Only by these means can trichlorethylene be adjusted to give a compromise in the matter of "not being too volatile".

Trichlorethylene is an ideal solvent in the respect that it contains no high boiling fractions. The commercial product is completely evaporated at 87°C., so that the last trace of solvent is easily removed. Apart from the comparatively low boiling point, "tri" will rapidly evaporate at ordinary temperatures, so that it is completely volatilised in a very short space of time. It conforms to the sixth item in the ideal specification, in that it is "not too non-volatile".

The seventh condition in the ideal specification refers to the question of toxicity. The perfect dry cleaning solvent would be without physiological effect and therefore quite harmless if inhaled by operatives. As in the case of carbon tetrachloride, trichlorethylene is open to criticism as regards its physiological action. The advent of the chlorhydrocarbons has set on foot certain hostile investigations with the express purpose of emphasising the limitations of the new solvents, and the point of toxicity is seized upon as one of "strategic importance". The outcome of these searchings has been to lay great stress on the question of physiological effect.

It has already been shown that even the petroleum solvents, although considered harmless, can cause violent headache, and in certain cases a state of intoxication, and that with carbon tetrachloride, a very definite and dangerous effect is experienced, if the vapours from the solvent are inhaled for any length of time. The high volatility of trichlorethylene and its physiological action are closely related, but

its effects are in sharp contrast to those of carbon tetrachloride. This contrast may be summed up in the statement: Carbon tetrachloride is toxic and trichlorethylene narcotic.

Trichlorethylene is very similar to chloroform in its anæsthetic action, in that insensibility is produced by inhaling a certain quantity of the vapour; but recovery from the narcosis is sure and fairly rapid if no further inhalation takes place. Because of this temporary effect and quick recovery, trichlorethylene is considerably safer than most of the other chlorhydrocarbons used as dry cleaning solvents. As a matter of fact, the degree of the narcotic effect only plays a subordinate part in connexion with the valuation of the industrial hygienic effect of a solvent. A more or less narcotic action must be reckoned with in the case of all organic solvents, but considerably more importance must be placed upon the presence of specific toxic effects on different organs (liver, kidneys, and optic nerves) than on a stupifying effect.

The industrial Supervisory Board of Chemnitz, Germany, has carried out an exhaustive survey of the question of physiological effect of trichlorethylene, and the Medical Officer to the Board, Dr. Katharina Stuber, issued a very interesting paper in June 1932, under the title, Injuries to Health due to the Industrial Use of Trichlorethylene and the Possibilities of their Prevention. Dealing with the narcotic properties of trichlorethylene, the report states: "Such a narcosis, so long as it constitutes a reversible poisoning, is a painful event for the person affected, but in so far as it is without consequences, one may in the end be inclined, from an industrial hygienic point of view, not to take it too seriously as regards harmfulness. The narcotic dose and the fatal dose with trichlorethylene appear to lie fairly far apart. Changes in the kidneys have not been observed. Injuries to the mucous membranes from trichlorethylene are comparatively rare. The local caustic effect appears to be slight. Hitherto injuries to the liver caused by trichlorethylene have never been reported, very much in contrast to the case of chloroform, carbon tetrachloride, and tetrachlorethane."

The relative safety of trichlorethylene lies in the fact that the state of insensibility induced by inhaling comparatively large quantities of its vapour constitutes a warning before the danger point is reached. On the other hand, users of trichlorethylene must understand that this solvent can produce fatal results. So far no published report

indicates fatal or even chronic results arising from the use of trichlorethylene in dry cleaning. The German publication referred to reports 26 fatal accidents, which could be traced to, or be connected with, the industrial use of "tri", but in every case they have been in the metal de-greasing industry.

The essential precaution to be observed when cleaning with trichlorethylene is that of maintaining efficient ventilation. Totally enclosed, vapour-tight systems are desirable, but probably not so essential, on account of physiological effect, as with carbon tetrachloride, although the consideration of prevention of evaporation still remains. It will be seen that need for the economies which involve the prevention of evaporation also provide the solution to the problem of avoiding toxic or narcotic action.

The employment of trichlorethylene in the enclosed type of machine which is being widely adopted in dry cleaning is almost foolproof. There are, however, two sources of danger, one of which is common to all dry cleaning systems, no matter what solvent is being used. This occurs during the cleaning out of storage tanks, filters, stills, and pipe lines. All solvent should be drained away and allowed to evaporate before cleaning covers are removed. The 26 fatal cases investigated in the German report occurred under extreme conditions, such as descent into a trichlorethylene container, or similar foolish practice. No solvent would be free from injurious effect under such conditions. The second danger is connected with the odour of trichlorethylene. It has already been noted that the smell of this solvent, although distinctive, is not unpleasant, and that, in fact, the odour is considered by some persons to be quite sweet and attractive. The vapours of trichlorethylene do not irritate the mucous membranes of the nose. The odour of the solvent is readily discerned, even when the amount in the atmosphere is very small and therefore harmless. The perception of the solvent in the atmosphere by the odour does not constitute a warning especially to those accustomed to it, so that in the absence of irritation in the nasal passages, the limit of toxic concentration in the atmosphere may be passed unheeded. Apart from this, the pleasantness of the odour may cause operatives to inhale more than the unavoidable quantity.

There have been many attempts to estimate the relative toxicity value of trichlorethylene, but the figures given by various investigators

are so widely divergent that no conclusion can be drawn from them. For example, one research worker gives the toxicity value as one-third, while another gives the value as 13·1 times that of chloroform for fatal results. So far the experiments have been conducted with a view to determining the effect of inhaling solvent vapours in considerable quantity, and as such have little commercial value, for the reason that no commercial concern would allow such a quantity to be promiscuously evaporated into the atmosphere of work-rooms, if only on account of cost.

The results of experiments do, however, bring out one point of agreement, and that is the relative order of the toxicity values of the chlorhydrocarbons. A German investigator (Zangger) has arranged these in the following order, beginning with the least harmful:

- 1. Perchlorethylene
- 5. Chloroform
- 2. Trichlorethylene
- 6. Dichlorethylene
- 3. Chlorbenzol
- 7. Carbon tetrachloride
- 4. Dichlorbenzol

The position of trichlorethylene is in interesting contrast to that of carbon tetrachloride.

The cost of trichlorethylene must be classified as a disagreement with the ideal specification, being more than three times the price of the petroleum solvents. Once again, it must be pointed out that trichlorethylene (as with all the non-inflammable solvents) is always quoted "per ton", which, in view of the differences in specific gravities, may be misleading. The specific gravity of "tri", being 1.47, would show approximately double weight for equal gallonage of the inflammable solvents, specific gravity o.8. The present-day quotation for trichlorethylene is £35 per ton, which works out at about 4s. 6d. per gallon. Another result of high specific gravity is that the weight of solvent supported by the cleaning machine is double that in the case of petroleum solvents. Thus with a machine requiring a charge of 50 gallons of solvent, with trichlorethylene the cost would be £11 5s. and the weight 735 lb., compared with £3 6s. 8d. and 400 lb. in the case of white spirit. Some compensation to offset these figures is obtained from the exceptionally fine degree of recovery possible with trichlorethylene. As with carbon tetrachloride, the recovery of

solvent in an efficient installation is almost complete. Trichlorethylene lends itself ideally to recovery by chemical absorbers and, once more, reference must be made to recovery by means of activated carbon. Absorbers of activated carbon are almost theoretically efficient when working on trichlorethylene. The increasing demand for trichlorethylene will undoubtedly cause chemical manufacturers to go in for production on a large scale, with consequent reduction of cost of manufacture, some proportion of which may reasonably be expected to be passed on to the consumer.

The ideal solvent is required by the specification to be chemically stable to storage and repeated distillation. Carbon tetrachloride is not stable in presence of moisture in contact with metals, especially iron. In the case of trichlorethylene, no such decomposition takes place even when large amounts of moisture are present. Trichlorethylene has a special advantage over carbon tetrachloride in this respect, as it does not attack metals. It is definitely stable to moisture.

The Table below indicates the almost negligible action of trichlorethylene upon metals in both wet and dry condition.

ACTION OF TRI.:

Results Expressed as Change in Weight in Milligrammes per
Sq. Cm. per Day.

	Dry Sc	olvent.	Wet Solvent.		
Metal	At 15°C.	At Boiling Point	At 15°C.	At Boiling Point	
Mild Steel		-0.11	+0.01	-o·8	
Copper	-0.012	-0.03	+0.01	-0.03	
Brass	-0.05	+0.06	+0.01	-o·o7	
Tin	+0.001	+0.07	+0.04	-0.03	
Zinc	+0.023	+0.3	+0.1		
Aluminium	+0.03	-0.5	+0.01	+0.03	
Lead	+0.008	_	+0.01	Nil	
Monel	+0.02		+0.01	<b>−0.06</b>	

This stability in contact with metals obviates the use of special metals in the construction of machines and apparatus for trichlorethylene, and as a result there is no difficulty in constructing plant for its use at a reasonable cost. Hence the capital outlay for an installation designed for trichlorethylene only is considerably less than that for apparatus for carbon tetrachloride. It is generally the custom to construct "tri" plants from galvanised iron, in contrast to carbon tetrachloride plants, which require special materials, such as monel metal and chemical lead.

Trichlorethylene is not stable to the action of sunlight, which may cause decomposition into hydrochloric acid gas and phosgene. This decomposition is easily detected by the Congo red test, as described under carbon tetrachloride. A sample of cotton dyed in Congo red and immersed in trichlorethylene, contained in an ordinary glass jar, shows a change from red to blue after less than an hour's exposure to sunlight. In many instances it is not necessary that the exposure should be to direct sunlight, ordinary daylight being sufficient. The decomposition of trichlorethylene on exposure to daylight has a parallel in the decomposition of chloroform under similar circumstances.

Two expedients are used to retard or prevent the decomposition of chloroform. The one is the screening of the light by means of brown glass for winchesters and bottles. A similar device is employed in certain exhibition plants using trichlorethylene. One of these plants is equipped with a glass panel in front so that the motion of the cleaning cage can be seen, and this glass is coloured brown to retard the decomposition of the solvent by sunlight. The second expedient in the case of chloroform is the addition of compounds which retard the decomposition.

It is well known that certain alcohols are quite efficacious in this respect, and consequently the specification of chloroform in the British Pharmacopæia allows the presence of 2% ethyl alcohol. Similar compounds are employed to retard the decomposition of trichlorethylene. The decomposition of trichlorethylene is prevented by the addition of minute quantities of compounds known as alkylamines. Diphenylamine, diethylamine, dibutylamine and triethanolamine may be used. These compounds bear a resemblance to ammonia and thus not only tend to retard

decomposition but also neutralise any acid, which would be formed during decomposition.

It is of particular interest to note that trichlorethylene, which has undergone partial decomposition and is therefore in an acid condition, exerts an increased solvent action upon cellulose acetate and is distinctly unsafe for this material.

Another aspect of the effect of trichlorethylene upon metals concerns the action on iron. Trichlorethylene is such a powerful solvent for grease and oil that it is impossible to retain a protective film of oil on iron surfaces in contact with "tri," for the iron would be kept clean and constantly exposed to oxidation by the atmosphere. It is therefore necessary that iron surfaces which come into contact with trichlorethylene should be extra heavily galvanised.

While trichlorethylene is itself non-inflammable, some warning is necessary against its use in presence of naked lights. In the region of naked lights, trichlorethylene tends to decompose and produce the highly poisonous gas phosgene. The lighting of work-rooms must therefore be by electricity or outside lights.

Yet another danger may arise if trichlorethylene is used in contact with aluminium. It has been noted that during the cleaning of aluminium components on whose surface filings or dust exist, aluminium chloride is formed and settles as a thin layer on the trichlorethylene. Being extremely hygroscopic the aluminium chloride attracts water, and the heat of reaction may cause ignition of neighbouring materials. If larger amounts of aluminium filings are heated in contact with trichlorethylene, there is a sudden extensive development of heat and decomposition of the "tri" occurs, large quantities of hydrochloric acid being liberated.

It may be suggested that some of these circumstances are not likely to arise during dry cleaning, but even so it is well to note the conditions under which trichlorethylene may be more or less easily decomposed.

Every solvent in use as a dry cleaning agent is non-miscible with water, for this property is of importance during the processes of recovery. The ultimate and only certain method of recovery is that of distillation. Trichlorethylene can be distilled readily in an apparatus fitted with closed steam coils only, and does not require distillation in steam for the vaporisation of final residues. In spite of

the ease and economy of distillation trichlorethylene is often treated by other recovery processes. The property of non-miscibility enables the solvent to be recovered by chemical settling and washing, or by the application of chemical absorbers. The common method of settling involves the use of strong soda lye, which is agitated with the solvent, after which the mixed liquids are allowed to settle. The "tri" forms the lower layer and is drawn off by means of a separator. In the case of recovery by chemical absorbers the solvent vapours are absorbed by activated carbon until saturation point is reached. The solvent is removed from the carbon by a current of steam and the mixed steam and solvent vapours are passed through a condenser. The distillate obtained is a mixture of water and trichlorethylene which is then passed into a separator.

Trichlorethylene is without colour, and therefore conforms to item 11 in the ideal specification.

Of all the advantages claimed for the chlorhydrocarbons over the older solvents of white spirit and benzol types, there is no doubt that the property of non-inflammability is the greatest. The petroleum and aromatic solvents have always been handicapped by the risk of fire which is attendant upon their use in dry cleaning, and because of this they are far from being considered as ideal solvents.

The twelfth item in the specification of the ideal solvent is the requirement that the flash point should be as high as possible. To this item trichlorethylene conforms exactly, for it has no flash point at all, being absolutely non-inflammable. It cannot be ignited, and it is also non-explosive. It will be remembered that the inflammable solvents can only be used under licence, and that their employment is subject to very stringent regulations. The premiums demanded by insurance companies are proportional to the additional risk. Obviously, the adoption of non-inflammable solvents would bring insurance risk, and therefore the premiums, down to normal.

The comparative safety from toxic effect and the non-inflammability are the main reasons for the suitability of trichlorethylene for shop-window installations; while for works practice, the absence of Home Office restrictions and the freedom from licensing conditions are obvious advantages. Trichlorethylene must be classed as a solvent in complete agreement with item 12 of the ideal specification.

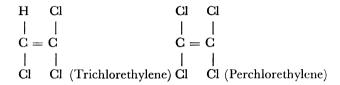
The specification of the ideal solvent indicates that the distillation range should be as small as possible. A close distillation range is very desirable, so that economy in time and steam necessary for drying out during the cleaning process and for redistillation during the recovery process may be realised. The commercial samples of trichlorethylene are pure substances, with a boiling point range within 1°C. The boiling point of trichlorethylene is 87°C., at which temperature the whole of the solvent is distilled so that for practical purposes, this solvent exhibits no boiling point range. Trichlorethylene requires a much lower consumption of steam heating during the drying out and redistillation than the petroleum solvents. In many cases exhaust steam will supply the necessary heat. The times of these operations are also much quicker than with the older solvents.

There is an interesting factor, which must be taken into account during the consideration of heating costs—one which is very often overlooked. The differences between the latent heats of vaporisation of the various dry cleaning solvents have a marked effect on the expense of drying and recovery. In the actual distillation of carbon tetrachloride and trichlorethylene, the temperature of the operation is obviously lower than that required with white spirit, which is the commonly used inflammable solvent. White spirit boils over a range 130°C. to 200°C., carbon tetrachloride boils at 76.8°C. and trichlorethylene at 87°C., but apart from these differences in boiling points, there are the variations in the latent heat.

It is not possible to give an accurate figure for the latent heat of white spirit, owing to the complex character of its many constituents. The figure for carbon tetrachloride is 47 calories, and that for trichlorethylene 57 calories. Thus, in comparison with carbon tetrachloride, trichlorethylene is at a disadvantage, first with a boiling point, 10°C. higher, and secondly, with a latent heat figure 10 calories greater. It has been found in works practice that all other conditions being equal, the times of drying out goods cleaned in carbon tetrachloride and trichlorethylene are proportional to the latent heats.

It has been estimated that the conversion of I gallon of white spirit from liquid at 70°F. to vapour at its boiling point would require 1,620 B.Th.U's., carbon tetrachloride in similar circumstances, 1,370 B.Th.U's, with trichlorethylene occupying a midway position.

Perchlorethylene.—While carbon tetrachloride and trichlorethylene have had a fairly wide adoption for commercial dry cleaning within recent years, there is yet another chlorhydrocarbon which is capable of application on an industrial scale. Perchlorethylene has not yet been employed to anything like the extent of the other two non-inflammable solvents, yet it possesses many advantages over each of them, in different ways. As the name implies, perchlorethylene is closely related to trichlorethylene, which it resembles in many of its properties. It may be said that, in general, the differences between the two are only differences in degree. The chemical relationship between them is indicated in the following diagrams:



Perchlorethylene is known as "Per", "Perawin", and tetrachlorethylene. It is a heavy liquid of specific gravity 1.64 (cf. white spirit o.8, trichlorethylene 1.47, carbon tetrachloride 1.63). Like all the chlorhydrocarbons, it is a synthetic product, the method of preparation being the treatment of pentachlorethane with mild alkalies.

As is demanded of any solvent for dry cleaning, perchlorethylene is remarkably efficient as a solvent for grease, oils and fats. It is considered to be equally as powerful and rapid in its action as trichlorethylene. It is claimed that this solvent has an excellent cleaning effect on all kinds of fabrics and tissues, woven felt, leather and similar materials, and especially on white goods, silks, artificial silks, and very delicate materials. The claim in respect of white goods will be of interest to every dry cleaner, for this class of goods presents a very difficult problem in practical dry cleaning. The powerful solvent action of perchlorethylene is the reason for the employment of this compound in certain proprietary spirit soaps.

Perchlorethylene agrees with the first item in the specification of the ideal solvent.

It is essential that a dry cleaning solvent should be a volatile liquid. Like all the chlorhydrocarbons, perchlorethylene is readily volatile,

giving off a heavy vapour at all temperatures. The rate of evaporation is considerably slower than that of trichlorethylene and carbon tetrachloride, and yet is quicker than that of the petroleum solvents. As in the case of the other non-inflammable solvents, losses by evaporation can only be reduced to economic limits by the use of specially designed, totally enclosed cleaning systems. In this connexion, it may be pointed out that the German firm which specialises in the manufacture of "per" has also designed and produced an installation expressly for its employment in dry cleaning. In spite of a relatively lower volatility, as compared with the other chlorhydrocarbons, perchlorethylene passes without difficulty through the various phases that occur during cleaning operations.

The requirement of chemical inertness is the third item in the list of the ideal solvent's properties, but it will be obvious that chemical inertness towards cellulose acetate is fast becoming (if it is not already) the crucial test for a dry cleaning solvent, and, as such, must be considered of equal importance as the property of being an efficient grease solvent. In marked contrast to trichlorethylene, perchlorethylene is above suspicion in its action on cellulose acetate. The precautions so necessary when processing cellulose acetate with trichlorethylene are superfluous when using perchlorethylene. Whereas the time and temperature during cleaning trichlorethylene must be under close control, it is found that dyed acetate silks show no discoloration or staining even after lengthy treatment with perchlorethylene.

The odour of perchlorethylene is a characteristic chloroform-like smell. In this the solvent again resembles trichlorethylene, and is certainly not offensive. Even this more or less pleasant odour is not imparted to treated goods, owing to the volatility of the solvent. While perchlorethylene would be completely evaporated at comparatively low temperatures, the time necessary for complete removal of odour at ordinary temperature would be much longer than that in the case of trichlorethylene. For practical purposes perchlorethylene is ideal in that it is free from offensive odour.

The volatility of the chlorhydrocarbons has been classified as a disadvantage, in that they do not agree with the ideal solvent which is required to be "not too volatile". The boiling point of perchlorethylene at 119°C., and the much slower rate of evaporation at temperatures below this, effects a compromise which gives the solvent a distinct

advantage over carbon tetrachloride and trichlorethylene. This lower volatility makes for appreciable reduction in the unavoidable loss of solvent during cleaning. The makers of "per" claim that the solvent consumption is only two-thirds that of the other non-inflammable solvents. It is, however, essential that this solvent be used in well constructed, totally enclosed plants.

The sixth item in the specification of the ideal solvent refers to the property of "not being too non-volatile". As with the other chlorhydrocarbons, perchlorethylene is free from admixture with other similar chemicals, and therefore contains no high-boiling fractions. Its boiling point being 41°C. higher than that of carbon tetrachloride and 32°C. higher than that of trichlorethylene, entails a greater consumption of steam and extended time for the drying-out process. The expense of recovery by distillation is relatively increased. The machines designed for the use of perchlorethylene are usually fitted with drying apparatus, giving greater volume and improved circulation of heated air. Perchlorethylene is the only non-inflammable solvent that can be said to be "not too volatile" and at the same time "not too non-volatile".

Yet another difference between perchlorethylene and the previously mentioned non-inflammable solvents is revealed by the consideration of its effect on operatives. Like all volatile solvents, perchlorethylene has a distinct physiological effect if its vapours are inhaled in sufficiently concentrated amount for a sufficient length of time. In these circumstances, the reactions produced are similar to those of chloroform and trichlorethylene, both in respect of the resultant narcosis and of the subsequent slow but sure recovery. The solvent has been classified as a toxic anæsthetic and volatile drug. Whereas the foregoing reactions are the results of deliberately volatilising the solvent in order to obtain high concentrations of solvent vapour in the atmosphere, and are only cited by the author to indicate that perchlorethylene is narcotic under specific conditions, the small evaporative capacity of this solvent in comparison with the other chlorhydrocarbons renders the approach to lethal concentration of its vapour in the air as almost impossible. In actual practice, it is found that perchlorethylene is the most innocuous of the non-inflammable solvents. It will be noted that the German investigator, Zangger, classified "per" as the most harmless of the chlorhydrocarbons.

The question of cost puts perchlorethylene at a disadvantage. Its present quotation is about 6s. 6d. per gallon. It does not, therefore, conform to the requirement that the ideal solvent should be cheap. It is more than six times the price of petroleum benzine. Some compensation may be obtained in that consumption of solvent during cleaning is approximately two-thirds of that of trichlorethylene or carbon tetrachloride. This high cost is the main reason for the delay in the wider adoption of perchlorethylene, and the handicap appears sufficiently large to outweigh the obvious advantages which this solvent possesses over the other non-inflammable solvents.

It has been suggested in some quarters that a mixture of trichlorethylene and perchlorethylene would satisfy a demand for a solvent which would be cheaper than the latter and less volatile than the former. Such a mixture would not be suitable for dry cleaning, as it would not have a constant boiling point, and its composition would be continually changing owing to the more rapid evaporation of the trichlorethylene.

The ninth item in the specification of the ideal solvent refers to the question of chemical stability. Here, again, perchlorethylene is shown to advantage, in comparison with the other chlorhydrocarbons. It is unaffected by any metal, including iron, even in presence of moisture. The construction of perchlorethylene plants involves no restrictions in the choice of materials, so that, as in the coase f "tri", there is no difficulty in constructing installations for its use at a reasonable cost. The systems are usually of heavily galvanised iron, in contrast to the monel and chemical lead required by carbon tetrachloride. The heavy galvanising of the iron is due to the fact that the powerful solvent action of perchlorethylene quickly removes protective grease films from metal surfaces, so that the iron is thoroughly cleaned and exposed to the rusting action of the air.

Like trichlorethylene, perchlorethylene is liable to decomposition by the action of sunlight. It is therefore essential to screen off the light, and consequently glass containers for this solvent are coloured brown. It is not certain whether the makers of "per" add some chemical to retard this effect, as in the case of "tri".

Perchlorethylene is an ideal solvent, in that it is not miscible with water, is colourless, and has no flash point. The advantage of being

non-inflammable has already been stressed and is claimed to the full for perchlorethylene.

As regards the final item, perchlorethylene is in complete agreement with the ideal specification, in that it has no boiling point range. With a boiling point of 119°C., the heat required for drying out and distillation is much greater than that required for the other chlorhydrocarbons, but the solvent distils completely at one temperature and shows the same constant composition. Its latent heat of vaporisation at 52 calories is midway between those of carbon tetrachloride (47) and trichlorethylene (57).

# TOXICITY OF DRY CLEANING SOLVENTS

While the petroleum solvents cannot be absolved entirely from the accusation of toxicity, it is in their favour that in almost every case the effects are transient and without lasting injury. The accidents that have happened, without exception, have been due to some foolish action which would not have been undertaken had the intoxicating action of the solvents been realised.

Of the petroleum solvents, white spirit is the least harmful. Its high boiling point range makes it almost impossible for the concentration of vapour in the atmosphere to reach even the lowest toxic limit. This makes white spirit the safest of all the dry cleaning solvents in use to-day, from a health point of view. Any danger arising from its use can only occur during the cleaning out of enclosed spaces, such as storage tanks or stills. White spirit, however, can have an injurious action on the skin. Rambousek states: "In addition to poisoning, the injurious effect of petroleum and its constituents on the skin must be borne in mind. Opinion is unanimous that this injurious action of mineral oil is limited to the petroleum fractions with high boiling point." Because of this action on the skin due to white spirit, certain workers are distinctly susceptible to a form of eczema, known as petroleum eczema. While this disease is more than probably due to personal idiosyncrasy, one factory inspector has pointed out that the cause may be insufficient care in washing.

In comparison with the petroleum solvents, the aromatic solvents are distinctly poisonous, particularly those of low boiling points. The presence of a large proportion of benzene makes the vapours of 90s. benzol extremely dangerous. Benzene acts as a blood poison, especially

# MODERN AND DYEING CLEANING PRACTICE

in the case of young women. Small and frequent doses may lead to chronic anæmia, while larger doses are often fatal. The vapours of 90s. benzols have an injurious action on the brain centres.

Solvent naphtha (90% at 160°C.) is much less noxious than benzol, probably due to the presence of a smaller proportion of benzene, with a correspondingly increased proportion of toluene and xylene. The higher boiling point of solvent naphtha tends to a slow rate of evaporation, and in consequence keeps the amount of vapour in the atmosphere within the limits of safety. The report of the Medical Inspectors of Factories for 1925 mentions one fatal case and two non-fatal ones connected with the use of solvent naphtha. "The fatal case was due to getting inside a tank containing naphtha to recover an iron rod, which had dropped in, and the two non-fatal, one to fumes from a rubber-spreading machine, and the other to cleaning out a tank, which had contained petrol and 25% benzene.

In the report for 1926, three cases (one fatal) were due to benzol vapour from stills, and one to cleaning out a storage tank. The fatal case was due to the workman accidentally omitting to turn the water on to the condenser with the result that the still-house became charged with a high concentration of benzol vapour. The report for 1926 also contains details of an interesting investigation undertaken by one of H.M. Medical Inspectors of Factories in a rubber works. Ten men, who were exposed to the vapour of 90s. benzol when mixing or spreading the rubber, were examined. "There was a definite history of slight gastric irritation with a distaste for food in three, and vertigo in one. Temporary conjunctival irritation was also complained of. The benzol solvent was replaced by a spirit solvent, and eleven months later a re-examination of the blood was made. Very marked improvement was shown, and there was an absence of any signs or symptoms of benzol poisoning."

Another fatal case due to benzol is reported during 1928, in which the victim was cleaning out a tank, which had contained benzol and petrol. Five other cases were also reported during this particular year, due to the fumes of naphtha, used either as a cleaning agent or as a paint solvent.

The non-inflammable solvents have come in for much criticism on account of their physiological action, and the authorities are watching closely for possible developments. There can be no doubt that, judged

from the consideration of what constitutes a fatal dose, the chlorhydrocarbons are the most potent and rapid of all the dry cleaning solvents. Because of this it is imperative that the toxic effects of these solvents should be fully understood.

The report for 1932 refers to the increasing use of the chlorhydrocarbons for many industrial purposes, and it is particularly interesting to note that the accidents referred to are all connected with cleaning out of tanks or apparatus. As far as dry cleaning is concerned, no case of poisoning from carbon tetrachloride was reported during 1932. This is a real triumph for the installations for carbon tetrachloride, and indicates the efficiency of the totally enclosed systems in preventing evaporation of the solvent into the atmosphere of work-rooms. On the other hand, it must never be forgotten that carbon tetrachloride is poisonous, and that its inhalation may cause functional disarrangement of the process of metabolism.

The action of trichlorethylene is much less harmful than that of carbon tetrachloride, in that its vapours are more narcotic than toxic. The narcotic dose produces temporary unconsciousness, from which recovery is fairly rapid and certain. It is patent that in studying the problem of toxicity the lasting toxic effect is the most important. With trichlorethylene the victim rarely experiences any permanent effect, the effect being merely transient. Trichlorethylene can cause death if inhalation continues, but the narcotic dose and the fatal dose are fairly far apart.

The reports of the Medical Inspector of Factories over the past few years indicate that unwise exposure to the vapours are usually to blame for mishaps. The report for 1924 states: "One fatal case from trichlorethylene and two non-fatal in rescuers were reported. The deceased man entered a kier after trichlorethylene had been poured in. No case of illness has been brought to notice caused by solvents such as acetone, amyl acetate, and trichlorethylene, though their use has considerably increased."

In 1925, five cases were due to "tri", "two (one of which was fatal) being due to painting the inside of a brewery vat with an enamel containing it". The report for 1927 refers to two cases of unconsciousness from trichlorethylene, one in the process of coating the inside of a tank with a preparation containing this solvent. During 1929, a severe case of poisoning by trichlorethylene arose in cleaning out the

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residue of a small tank, in which it had been used. The operation had been performed several times previously without ill effect, so that the operatives and the firm in question were unaware of the narcotic properties of trichlorethylene, and no steps were taken to prevent inhalation of the vapours. In cleaning out such tanks, unless they are previously adequately ventilated, the worker should be protected with a helmet connected with a fresh-air pipe line.

All available evidence indicates that the danger with the non-inflammable solvents can only arise during the cleaning out of enclosed parts of the cleaning systems, and even this danger can be reduced to workable limits by thorough ventilation of workrooms. As the vapours of all these solvents are extremely heavy compared with air, the "take-away" of any ventilation arrangement must be at floor level.

The introduction of perchlorethylene may go far to still the outcry of toxicity. It has been claimed that the vapours of this solvent are almost harmless, and that its low evaporative capacity precludes the possibility of any large concentration in the atmosphere.

# THE WACKER MACHINE

Since the conditions of working differ according to the solvent being used, the successful application of the non-inflammable solvents to commercial dry cleaning is only assured in machines which are designed expressly for the use of the particular solvent. A special design is all the more essential when the solvent is trichlorethylene.

The possibilities of trichlorethylene as a dry cleaning agent were fully recognised by the firm of Dr. Alexander Wacker, of Munich, who determined to produce a machine specially designed for cleaning by this solvent. The Wacker machine can rightly claim to be based on a complete understanding of the properties of trichlorethylene, for the makers hold the monopoly of the manufacture of trichlorethylene in Germany, and thus are the producers of both the machine and the solvent. The Wacker installations are marketed solely for the use of trichlorethylene or perchlorethylene, and, since neither of these solvents exercises a corrosive action on metal, even in presence of moisture, the use of special metals for their construction is not necessary.

The "tri" system has had a wide adoption in Germany and France, and is now being marketed in England. The Wacker machine for

"tri" cleaning is a totally enclosed one, in which the article to be treated are cleaned, extracted, dried, and deodorised. The washer is divided into two (sometimes more) horizontal compartments, and is loaded from the end, as with a Smith machine. The division is necessary in order to ensure an even distribution of the load about the main spindle during centrifugal extraction.

The loading door is clamped so as to be vapour-tight. During the cleaning process the liquor is in constant circulation, loose impurities being removed by passing the solvent continually through a centrifugal clarifier. After cleaning, the liquor is run off from the washer, which is then geared up to a high speed and becomes an extractor, working on a horizontal axis. This is a great advantage, as the greater part of the solvent held by the load is recovered without the use of heat. After extraction, the machine is slowed down to normal speed, and the goods dried by blowing in hot air, which is passed from the machine through a condenser in order to recover the evaporated solvent. To reduce the time required, and also the temperature of the drying operation, most of the Wacker installations are equipped with a steam jacket, which heats the casing of the machine, and so prevents the re-condensation of the solvent on the walls of the system The hot air for the drying-out process is obtained from an air jacket surrounding the steam jacket, so that a separate air heater is unnecessary.

When the flow of solvent from the condenser ceases, cool air is introduced into the machine and exhausted to atmosphere for a few minutes. This completely deodorises and cools the load. The Wacker machines have certain distinctive features, such as the introduction of the solvent and drying air at the centre of the cleaning drum. The makers claim that by this method a specially rapid and thorough cleaning of the articles is obtained, while in the case of the hot air all the air is forced through the load, effectively giving a rapid, complete, and uniform drying.

The lay-out of pipe lines for the "Tri" solvent enables the process to be run as an intermittent system of washing and rinsing, or as a continuous system where the solvent is passed through a centrifugal clarifier or a filter. The purification of the "Tri" is effected by distillation in a separate still, while the installations usually have four storage tanks, for pure solvent, centrifuged solvent, dirty solvent, and

distilled solvent. After distillation the "Tri" is washed with strong soda lye to remove acidity.

Three methods of driving are employed on the Wacker machines, according to the type of installation. One installation combines a neatness of arrangement with remarkable strength of structure. A series of electric motors of differing horse-power give the various driving speeds necessary for cleaning and extracting. This equipment is known as the "M" type, where an exclusive double motor drive is used, one electric motor for the cleaning process and a second one for centrifuging, no belting or gearing being used. The smaller cleaning motor drives the drum shaft through a worm, while the larger centrifuging motor drives directly on the main shaft, each motor being brought into action at will by a reversible coupling. The centrifuging motor can be brought to speed slowly, so that the washer gradually reaches the speed necessary for extraction. This particular drive is very compact and is extremely neat in the absence of belts. Equipment "R" is fitted for belt driving, with the usual arrangement of belts and pulleys for reversing and speed variation. Equipment "K" embodies a completely enclosed system of gearing, running in oil, and driven either by a single transmission belt or motor.

The Wacker machines are available in sizes ranging from the smallest of approximately 25 lbs. capacity to the largest of 240 lbs. capacity dry weight of work. The solvent loss is less than 10% of the dry weight of goods being cleaned, or less than  $1\frac{1}{2}d$ . per suit. The economical working of the Wacker installations is shown by the fact that the plant can be run at steam pressure of less than 10 lbs. per sq. in., so that the drying temperature does not exceed 65°C.

In addition to being the producers of "Tri", the firm of Dr. Alexander Wacker also manufacture perchlorethylene, which they market under the name "Perawin". Installations for the use of "Tri" or "Perawin" are now available. The machines for use with "Per" differ slightly in certain constructional details from those designed for "Tri" only. These machines are classed as "E" models; the largest size, E vi, is of sufficient capacity to treat carpets. The most apparent differences in the E type, as compared with the "Tri" machines, are that the loading door is in the front and that the inner washing drum is undivided. Owing to the drum being undivided, the revs. during extraction are only 300 r.p.m., compared with 600 r.p.m. in the other

models. With the "Perawin" installations the steam jackets are omitted. The introduction of hot air for drying out takes place at the centre of each end of the drum. It is claimed that this gives a quicker and more effective drying.

Messrs. Wacker strongly recommend the use of "Perawin" as an additional solvent rather than as a substitute for "Tri". The great advantages of "Per" in being above suspicion in its action on cellulose acetate, and in being the least harmful of the chlorhydrocarbons, have already been stressed. Unfortunately, the price of "Per" is about 33% more than that of "Tri", although some compensation is obtained in that the loss of solvent with the Wacker installation is only 7%, compared with "Tri" 10%.

The drives of the Wacker E model machines are similar to those employed on the "Tri" installation; thus, equipment "R" is belt driven, equipment "K" wheel gearing running in oil, and equipment "M" double electric motors (direct coupled). Machine E I is made only in equipment "R", driven by a belt from a motor or equipment "K"; E III and E vi are made only in equipment "K" or "M".

The Wacker installations are built as a complete unit, including electric motor drive, pumps, tanks, clarification device, and distillation apparatus. Articles cleaned by the Wacker process are found to be very clean and particularly bright and odourless. The users of these installations have no trouble arising from the presence of moisture in the system, while corrosion of the metal parts is unknown. The processing of cellulose acetate is completely successful and foolproof by means of "Per".

# THE BURTOL CLEANING SYSTEM

There is no doubt that one of the leaders of the revolution in dry cleaning methods which is going on at the present time is the Burtol unit. This unit is made up of three separate appliances—the Burtol machine itself, an extractor, and a drying-out drum. The system is an example of the semi-open type, in which the goods are removed from the washer, while still "wet" with solvent. The recovery of the solvent retained in the goods is carried out entirely by the extractor.

The Burtol unit occupies an extremely small floor space, and so

can be easily accommodated in any shop window of reasonable size. It has been expressly designed for shop-window display.

# THE ZORIC MACHINE

#### THE ALMCO GARMENT CLEANING UNIT

As there is apt to be a certain amount of incorrectness of name in connexion with the installation, which is marketed by the British Laundry Machinery Co., Ltd., the exact and full title is given. The plant under review can only be rightly termed "The Zoric" in the United States of America, but in Great Britain, owing to a different code of trade-mark laws, the machine is marketed under the name of "The Almco Garment Cleaning Unit", the solvent being "Zoric" fluid.

The Almco Garment Cleaning Unit is a self-contained unit. The machine is shipped from the factory as a unit on a rigid base of structural steel, and therefore requires no assembling on site. The crated machine of the No. 4 Almco Unit is 8 ft. 5 ins. high, 7 ft. 9 ins. wide, and 14 ft. 9 ins. long. No special foundation is required; any good solid floor can be used.

The Almco machine is specially designed and constructed to be used with the solvent known as "Zoric" fluid. Zoric fluid is a specially prepared solvent, which has carbon tetrachloride as its essential and basic constituent. From this, it is obvious that all the special materials necessary with installations using carbon tetrachloride are to be found in the Almco plant.

The whole installation is made of very heavy materials, is particularly compact and solid, giving the impression of being built to endure. The complete unit consists of a washer, distillation plant, drying out and deodorising equipment, and recovery appliance.

The washer is of ordinary pattern and is constructed of monel metal. It has a cleaning drum with a single compartment, perforated and fitted with "beaters" to ensure even movement of the load during cleaning. The door of the cleaning drum is firmly fixed by heavy latches, while the outer door is clamped so as to be vapour-tight. The first Almco installations had a capacity up to 70 lbs. dry weight of garments.

The method of cleaning employed is an intermittent one of a wash, followed by a number of rinses according to the nature of the load being treated. After the drum has been loaded and the doors clamped as tight as possible, the operation of a single lever releases the brake, and power is applied to the machine. The Zoric solvent is introduced into the machine by pulling a second lever over to "fill" position. This lever is held in the "fill" position until the solvent reaches a pre-determined height, as shown in the gauge glass at the right end of the machine, when the lever is released and allowed to return slowly to a neutral position.

The time of running for the first wash depends upon the class of goods being cleaned, but the average time is five minutes. A remarkably complete set of instructions for operating and maintaining the installation accompanies each machine, and the recommended times can be found in this. After the first cleaning, the machine is drained, fresh Zoric fluid being run in for the rinsing process. The rinsing times also vary according to the nature of the load, but the longest run is seven minutes for the first rinse, followed by one or more short rinses of three minutes' duration. The "run away" line is fitted with a sight glass, so that the extent and condition of the fluid leaving the machine can be observed. When the fluid in this glass still shows signs of being soiled, further rinsing is necessary, until the load is cleared. During the draining period, the cylinder is kept rotating.

When the fluid flowing from the cleaning drum shows no sign of being discoloured the load is prepared for drying.

The Almco plant is an example of an installation in which the solvent in the load is removed entirely by evaporation. During the time of the cleaning operation, the "heater valve" is slightly open, so that the heating elements are being gradually warmed up in readiness for the drying of the load. To begin the drying out, the valve marked "condensers" is opened, and this causes water to flow into the condenser while, at the same time, the switch marked "fan" is depressed. The fan blows air through the heater, into the load and exhausts by way of the condensers. In this way the Zoric fluid is removed from the goods by evaporation, and recovered by being condensed in the condensers. The flow of the condensing Zoric can be observed in a sight glass on the delivery line from the condenser. The drying out

is continued until this flow ceases, after which the heater valve is closed and another valve marked "cold air" is opened. Air at ordinary temperature is admitted to the machine and exhausted to an outside atmosphere. This operation removes the final traces of solvent and brings about deodorisation of the garments in about five minutes.

The Zoric fluid used in the washing and rinsing processes runs from the machine through a button trap into a sump tank. The dirty liquor is pumped directly into the main still (there are two stills with the Almco installation). It must be noted that there is no connexion between the sump tank and the machine, except by way of the still. The distillation of the solvent is carried on so long as the liquor evaporates fairly rapidly. When the distillation becomes sluggish, the residue in the still is run into a secondary smaller still, after which the main still is re-charged. A very fine degree of recovery is obtained by allowing the Zoric in the secondary still to evaporate very slowly. Each still delivers the evaporated fluid to condensers, where the Zoric is recovered and run into a clean storage tank. The Almco Unit requires about 300 gals. of Zoric fluid to maintain the solvent circuit efficiently.

All controls of the Almco installation are mounted on a neat panel on the right of the cleaning drum at such a height as to be level with the eye. Each control is clearly marked to show its particular function.

The Almco Garment Cleaning Unit is now marketed in two sizes, in which the dimensions of the cleaning cylinders are 25 ins. by 24 ins. and 32 ins. by 54 ins. The makers of the unit state that the solvent loss is  $\frac{3}{4}d$ . per lb. dry weight of work cleaned. This would give a solvent cost per suit of about 4d. The Zoric fluid is sold by makers of the machine at  $4\frac{1}{2}d$ . per lb., which corresponds to 6s. per gall.

The American Laundry Machinery Co. have made exhaustive tests on the reactions of the various textiles when cleaned with Zoric fluid. In each case the selected pattern is thoroughly examined before and after ten normal treatments in the solvent. The published reports of the United States Testing Co. show "the Zoric process of dry cleaning to have no appreciable deteriorating effect on any materials used in the experiments". The materials tested comprise a very wide range. All-wool overcoating, cellulose acetate taffeta, chintz (cotton), parachute silk, printed silk chiffon, pure silk (printed), Bemberg artificial

silk, Glanzstoff, weighted silk crepe, worsted jersey, worsted serge, worsted suiting, artificial silk velvet on silk back. The object of the examination was to obtain evidence of change in strength, shrinkage, loss of colour, chafing, harshness, attack on the finish, or any other alteration from the original condition of the fabric.

Yet another interesting investigation had been that to determine the amount of hydrochloric acid produced in the liquor during the treatment of certain wool materials. The lowest pH value recorded during many tests was 3.8, which indicates the presence of 0.000429% acid, an amount too small to have an effect on any textile material.

# THE "BAND-BOX" MACHINE

The "Band Box" dry cleaning unit was originally imported from America, where it has been sold in very large numbers. It is now manufactured in this country by the well-known engineering firm of Manlove, Alliot & Co., Ltd., Nottingham, by arrangement with Messrs. Courtney & Hill, Ltd., Nottingham, who are the sole European licensees. The "Band Box" unit is of unique design, in that it embodies a system containing twin washers. This makes for a continual and remarkably high output. The installation departs from the usual style of totally-enclosed arrangement, but an ingenious method of hydroextraction enables the solvent consumption to be kept within economic limits.

The two cleaning machines have a capacity of 10 lbs. to 12 lbs. of dry weight of work each. They are each mounted on a stand, the feet of which are hemispherical, and rest in metal cups, so that any vibration is taken up in much the same way as in the suspended type of centrifugal extractors and no vibration is transmitted to the floor. The outer casings of the washers are constructed of nickelled copper.

The washer is similar to the basket of an ordinary hydro-extractor, and is of hard aluminium. In the cleaning process the basket is tilted at an angle of 45°, and dips into a quantity of dry cleaning solvent contained in a reservoir in the bottom of the outer casing. Each cleaner is fitted with an aluminium lid, which is clamped down by four thumb-screws, and is easily and rapidly removed or mounted in position.

The method of cleaning is continuous the solvent being in constant circulation throughout the process. A vertical partition in the bottom of the casing acts as a weir, so that the solvent is kept at constant level. When in the tilted position the cleaning basket is clutched on to a low-speed shaft giving 34 revolutions per minute, without any reversing motion.

The provision of two cleaning machines assists in the orderly and rapid working of the unit. It can readily be seen that by spreading the work over the two cleaners, a rhythm of production is obtained as a matter of course, one machine being used as a washer, while the other is being used as an extractor. Loading, cleaning, extraction, and unloading in the two machines follow in perfect rotation. Each basket of the unit has a capacity of four loads per hour, so that the installation can give the remarkable output of 80 lbs. dry weight of work per hour.

The "Band Box" system of cleaning makes use of continuous filtration in which the solvent is withdrawn from the cleaning machine by a motor-driven rotary pump, passed through a special type of filter, and thence sent back to the machine. As the two machines alternately function as washers and extractors, the one filter serves both. The filter is of the vertical type, in a galvanised steel casing, fitted with lid, bolted down by swinging screws. This lid is readily removed when access to the filter is necessary. The filter consists of a series of leaves of metallic mesh supported in frames and covered with cloth bags. The mesh may be of galvanised steel wire or monel wire of fine mesh, the latter, of course, being a little more expensive.

The process of filtration entails the use of filter aid. This is a form of silica which is added to the solvent in the division behind the weir in the cleaning casing. This filter aid is carried with the solvent into the filter, where it adheres to the wire mesh, forming a porous layer. In passing through this layer, all loose particles of dirt and any moisture in the solvent are trapped, while the solvent is maintained in the best condition for cleaning. The filtration is correctly described as continuous high-speed cyclic filtration.

The arrangement of the filter allows the solvent to complete more than 90 cycles per hour. This system of filtration is similar to that used by the largest cleaners in this country and the United States.

It is claimed that installations using this system are not open to the charge of interfering with the natural grease in garments and so causing a harshness of feel.

The cleaning time with the "Band Box" unit is only seven minutes. In order to convert the cleaning basket into an extractor, the aluminium cover is removed and the basket lifted by the operator from the angle of 45°, and clutched on to a vertical spindle. It will be observed that the cleaning machine is open to the atmosphere during the conversion phase, but solvent losses are obviated by taking advantage of the heavy gravity of the spirit. The solvent vapour is very heavy and, since the machines are open only at the top, it tends to lie within them and not escape. This tendency is assisted and strengthened by the installation of a cooling system on the outside of the cleaning drum. Both cleaning machines are cooled by permitting cold water to circulate through a series of coils round the outside casing.

Since the basket is of aluminium, no great effort is necessary to lift it from cleaning to extracting position. It is necessary, however, for the operator to balance the load before re-starting the machine. The extraction speed reaches 600 r.p.m., and, being on a vertical spindle, is remarkably efficient.

An electric timing switch is supplied with each machine, and this automatically stops the driving motor at the end of the cleaning or extraction operation. The switch time is usually seven minutes. The various switches are mounted on a control panel, which is normally fitted in front of the filter.

The "Band Box" system does not attempt the recovery of the solvent remaining in the load after extraction. The treated garments are removed from the machine and hung on hangers on a rail, where the solvent is dried out in a current of cool air. The current of air is provided by an extracting fan. In order that this fan may act efficiently and give thorough ventilation, the machines and drying space are enclosed in a booth or small room, with adequate air inlets and outlets. It is found that where care has been taken to ensure efficient ventilation of the booth and drying space, the plant is exceptionally free from fumes of solvent.

The purification of the solvent is by distillation. The distilling apparatus is included in the installation, but is usually mounted some short distance from the cleaning machines. Owing to the efficiency

of the filtration system, distillation of solvent is spasmodic. The recovery is carried out in the usual arrangement of still with steamheated coils, over which is mounted a condenser with water-cooling coils. Both still and condenser are of nickelled copper. The capacity of the still is about 25 gallons of solvent.

The "Band Box" dry cleaning unit is quite suitable for display purposes, and can be seen operating in many shop windows. In most cases the plants working in this country use trichlorethylene as solvent. The special cooling coils, by keeping the temperature low during the operation, makes this solvent absolutely safe, even with the cleaning of coloured cellulose acetate. It is of interest to note that in America certain "Band Box" plants are being successfully operated with carbon tetrachloride. This is considered to be due to the fact that the absence of a recovery system avoids the intimate mixing of the solvent and moisture, which would be evaporated if deodorising took place within an enclosed system, and consequently risk of corrosion is very much reduced.

# THE PROSPERITY CLEANING UNIT

The Prosperity Dry Cleaning Unit is the unique example of automatic control applied to dry cleaning. The 3-B unit consists of a cleaning cylinder, 30 ins. diameter by 40 ins. length, in welded steel, divided into Y-pocket compartments, and with a dry-weight capacity of 45 lbs. It is manufactured by Messrs. Baker Perkins, of Peterborough. The plant is remarkably light and compact, weighing only 5,000 lbs., and occupying an overall floor space of 8 ft. by 6 ft. The cleaning operation is on the wash-and-rinse principle. The used solvent, in all stages, is pumped automatically through a water-cooled condenser into storage tank and thence to the still. The solvent condensed during the drying stage is also pumped into the still. In this way the makers claim that, since the solvent undergoes continuous distillation within an enclosed unit, solvent losses are maintained at an irreducible minimum. It is claimed that the solvent loss with the Prosperity plant is under  $\frac{1}{2}d$ . per lb. dry weight.

The outstanding feature of the Prosperity unit is the incorporation of an automatic control system, known as the formatrol. The valves of the installation are operated by compressed air and are controlled

by the formatrol. The formatrol is a disc with a series of slots which cause electrical contacts to be made at intervals determined by the length of the slot. The automatic control of the cleaning operation has one great attraction, in that uniform processing is obtained, since every load is cleaned according to a predetermined formula. The unit includes thermostatic control of temperatures of drying and deodorising. This temperature control has an important bearing on the cleaning of silks and of articles in which the colours are likely to bleed, for in each case the formatrol controls the temperature and time of drying, according to a formula free from risk. The control disc is changed to suit the nature of the articles being treated. Since the cleaning operation is fully automatic, the only attention required is the manual labour involved in loading and unloading the machine, which, the makers state, amounts to approximately six minutes per hour.

# THE KWICK PLANT

The Kwick plant is manufactured by Messrs. Atmer & Booth, Ltd., of London It was first imported from Germany, where it was known as the Jungnickel installation, but is now made in this country. The name "Kwick" was given to the installation suitable for shopwindow cleaning, but in recent times this name has been applied to all the installations of this firm, whether of the shop or factory type.

The Kwick plant is produced in eight different sizes up to a capacity of 135 lbs. dry weight of work per load. The cleaning cage is of monel metal, and the whole plant is designed for trichlorethylene or perchlorethylene. The cleaning drum is without partitions, and has the loading door in the front of the machine. The complete installation consists of the cleaning machine, clarifier, and still. The recovery plant is much larger than usual, so that the time of drying is correspondingly shorter.

During the operation of the plant the cleaning cage first acts as the normal rotary drum, and in the second stage is converted into a hydro working at 600 r.p.m. The Kwick plant is fitted with a special gear-box to cope with the various drum speeds required. This special gear-box has helical gears which are almost noiseless. The clarification of the used solvent is by filter or centrifugal clarifier, at the purchaser's

option, and is continuous during the cleaning operation. Although the plant may be operated on perchlorethylene, the makers always recommend trichlorethylene. The enlarged recovery plant on the Kwick installation enables the full process of cleaning, hydroing, drying, recovery, and deodorising to be carried out in 45 to 50 minutes. The medium size of plant to take 45 lbs. dry weight of work, has three tanks for clean, medium, and dirty solvent respectively.

# THE GEM TRIKOHL MACHINE

The Gem Trikohl, as the name implies, is essentially designed for use with trichlorethylene, but the makers emphasise that any part of the machine can be supplied in monel metal in the event of carbon tetrachloride being preferred as the cleaning solvent. The Trikohl installation is made in three sizes, in which the capacities of the cleaning drum are 15 lbs., 30 lbs., and 80 lbs. of dry weight of work.

The system is yet another example of a totally-enclosed installation, in which the whole range of the processes of dry cleaning is carried out while the plant is kept vapour-tight. The installation is entirely self-contained, and consists of a cleaning cylinder, which is geared to run at a slow speed for washing and at a high speed for extraction of solvent, a filter appliance, three storage tanks, and distillation equipment, all mounted as a complete unit.

The smallest size in the Trikohl range is especially suitable for exhibition purposes. The plant can be erected in the remarkably small floor space of 6 ft. by 4 ft. 9 ins. The cleaning drum is an individual cylinder 20 ins. in diameter and 16 ins. long. It is loaded through a circular opening in the end. The door of the cylinder can be removed completely and is held in position by two spring catches. The outer door of the machine is kept vapour-tight by means of a powerful screw clamp.

After the machine has been loaded with the goods to be cleaned, an electrically-driven pump is started, and this causes solvent to be introduced into the machine. A handle on the side of the machine is moved to the position marked "washing", and the drum begins to rotate, reversing after every third complete revolution. The method of cleaning is one of continuous circulation, the solvent pump keeping

the solvent in continuous flow from the machine through a fluff and button catcher, through a filter and back to the machine. The rate of circulation is such that the solvent circuit is complete every thirty seconds. The time of cleaning varies from five minutes for silks and delicate materials to ten minutes for suits and heavy articles.

During the drying process the temperature is regulated so as not to exceed 60°C.

After the cleaning process, the circulating pump is stopped, the solvent is drained from the machine and returned to a storage tank for medium spirit by way of the filter. When the flow from the machine has ceased, the cleaning cylinder is geared up to a high speed and becomes an extractor. The gears are operated by moving the handle on the side of the machine to the position marked "hydroing". The running of the machine is particularly smooth, as the cylinder runs in SKF ball-bearings. The extractor is continued as long as the solvent is observed flowing in the sight glass. This process usually requires about twenty minutes.

When as much as possible of the solvent has been recovered by extraction, the machine is returned to normal speed and the drying out phase commences. Air is blown over a large steam heater by means of an electrically-driven exhauster fan. The evaporated vapour is recovered by passing it through a condenser, while the air is maintained in circulation through a fluff catcher, back over the heater to the cylinder. The drying process continues until no more solvent is recovered from the condenser. The deodorising process consists of blowing a current of cool air through the machine and exhausting it to the outer atmosphere. The whole load can be cleaned in forty-five minutes in the case of heavy goods, and fifteen to twenty minutes for light materials.

The solvent tanks in the Gem Trikohl installation are neatly mounted one over the other. The three storage tanks are cylindrical and are fitted with gauge glasses, which are graduated to the number of gallons in the tank. The lowest tank is for dirty solvent, the next or middle tank is for medium solvent, while the third is for clean solvent storage. The condenser forms a fourth and top tank, and serves for the recovery of solvent from both the distillation process and the drying out process.

The solvent consumption of the Gem Trikohl is remarkably small. One installation is stated to have been running at a solvent loss of  $1\frac{1}{2}\%$  of the dry weight of goods cleaned during the actual working conditions over a period of three months. Yet another unit has returned figures showing that 200 garments were cleaned for a solvent consumption of 5 gallons, which gives a solvent cost of 1.35d. per garment.

The Gem Trikohl installations were originally imported from Copenhagen, but Messrs. Henius, Ltd., the British agents, point out that more than 75% of the plant is now manufactured in this country. The whole of the tanks, motors, and pipe lines are of British manufacture and materials.

The drives of the Gem Trikohl plant are entirely by very small h.p. electric motors, thus the main motor drives the cleaning cylinder, other smaller motors drive the solvent pump and the exhauster fan. The smallest installation requires a total h.p. of 2; the medium size 5 h.p.; and the largest size 7 h.p.

#### THE BARBE INSTALLATION

Dry cleaning in totally-enclosed machines is by no means a recent innovation, and is not peculiar to the non-inflammable solvents. There are two installations on the market designed for the use of benzine, and they have been used for some years now. These machines embody the principle of the closed circuit for the cleaning spirit, but differ from each other in certain important features.

The machines are the subjects of various patents. The Barbe process is covered by patents held by the firm of Robatel, Buffaud & Cie., of Lyons. The installation is of particularly solid construction. The whole of the cleaning processes are carried out while the machine is maintained in a vapour-tight condition; that is, the cleaning drum acts as a washer, extractor, and dryer, in rotation. The distinguishing feature of the Barbe process is that the operations are conducted in the atmosphere of an inert gas, carbon dioxide, instead of the ordinary atmosphere.

It is obvious that the risk of fire and explosion would be increased if such a highly inflammable solvent as benzine were used in closed

circuit without any precaution. The presence of carbon dioxide eliminates this danger almost entirely.

The complete installation for the Barbe process consists of a cleaning drum of capacity up to 200 lbs. weight of work, a number of spirit tanks, usually five (one for clean spirit, three for used or medium spirit, and the other for dirty spirit), a condenser, a steam heater, a gas holder for the carbon dioxide, a compression chamber for carbon dioxide, an air circulating pump, and a still.

It is usual to treat the goods in order to remove loose or surface dust and moisture by shaking them in a heated dust wheel as a preliminary to the cleaning process proper, and also to sort the load according to the nature of the materials and colours, as in the case of cleaning with benzine in an ordinary washer.

The cage is charged with the weighed amount of work, and then the machine is closed so as to be spirit-tight. The air in the installation is withdrawn by means of a vacuum pump until the pressure is reduced to approximately a quarter of the normal atmospheric pressure, after which carbon dioxide is introduced into the machine, being pumped from the gas-holder into a pressure chamber at three to four atmospheres' pressure, and thence to the cleaning drum. The cleaning spirit is then run in to the required level.

The machine is rotated at a slow speed for about fifteen minutes, the cleaning time varying with the class of goods under treatment. After cleaning, the spirit retained by the load is partially removed by centrifugal extraction. The machine is fitted with a series of driving pulleys, so that the speed may be varied from a slow rate for cleaning to a comparatively high one for extraction. It is interesting to note how closely this arrangement has been followed in many of the installations now being marketed for the application of the non-inflammable solvents.

The cleaning process in the Barbe machine is an intermittent one of washing, followed by one or more rinses, with a quick extraction process in between each rinse. The extraction usually takes some four or five minutes, after which benzine for the rinsing phase is run into the drum and the rotation reduced to slow speed. The *modus operandi* is subject to many variations, according to the nature of the loads; for example, the extraction between the rinses can often be omitted, and, of course, the number of rinses given varied at will.

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When as much as possible of the spirit has been recovered by centrifugal extraction, the machine is geared back to normal running. The solvent still retained by the load is evaporated in a current of hot carbon dioxide. The carbon dioxide is kept in constant circulation during the drying process, passing in circuit through a fluff catcher into a condenser, where the spirit is recovered.

From the receiver of the condenser the carbon dioxide is pumped through a heater and back into the machine. In order to assist the drying out, the cleaning machine is fitted with a steam jacket, into which steam is introduced and the machine gradually heated up to 100°C., if necessary. The drying out is carried on as long as the condensing solvent can be observed flowing through the sight glass attached to the condenser receiver.

Before the load can be removed, the carbon dioxide must be returned to the gas-holder, and then the pressure inside the drum must be equalised to that of the atmosphere. The gas is removed by means of a suction pump, which reduces the pressure to about 8 lbs. per sq. in. The pump is then stopped and all valves closed for a few seconds. The door of the machine has a small cock fitted to it, and this is opened to admit air to the inside of the drum until normal pressure is obtained. The load can now be removed. The output of the Barbe machine is up to ten loads per working day, each load taking on an average 1½ to 2 hours.

The advantages claimed by the makers of this installation include the freedom from fire risk and an enormous reduction of solvent losses. The complete elimination of fire risk depends entirely upon the machine being maintained vapour-tight, as any leaks at the joints may cause air to be sucked into the vapour within the drum and the possibility of formation of a highly inflammable mixture in the enclosed space of the plant, a circumstance which is made more dangerous with the use of heat in the drying out process.

The Barbe is said to show a reduction of 80% in the losses of solvent in comparison with the open type cleaning machine. It is obvious that the labour cost of operating the Barbe process is less than with the older method, and that the need for drying-rooms and appliances is avoided.

The solvent used in the Barbe must conform to well-defined specification; for example, the spirit must be readily volatilised, so that the

time of drying is not prolonged unduly, and consequently high boiling fractions are to be avoided. Because of this, the more volatile spirits are employed, and the makers claim that these low-boiling solvents give better cleaning results.

In these days when the non-inflammable solvents come in for criticism on the grounds of toxicity, it is interesting to note that the absence of benzine vapour from work-rooms using the Barbe plant is cited as another advantage.

The Barbe machine is particularly heavy, and, as may be imagined, entails a relatively high initial outlay; but the main difference from the ordinary cleaning plant is in its much smaller output.

Yet another point of topical interest is the risk that the hot solvent used may exercise a solvent action on certain colours, while the heat of the drying out is suspected of imparting a harsh feel to some goods and of causing dark colours to show white on their surfaces. Such criticism seems familiar. Evidently it is not confined to the chlorhydrocarbons.

The other totally-enclosed plant designed for the use of benzine solvents is the Smith installation. It resembles the Barbe plant in that it embodies the principle of the closed circuit of the cleaning spirit, but differs from it in many important features.

### THE SMITH MACHINE

The Smith installation was designed and employed by its patentees in their own cleaning works for a considerable time before it appeared on the market. It would seem that they built the machine for their own private use rather than as something for the open market.

It will be remembered that in the Barbe process the whole of the cleaning phases are carried through while the installation is kept vapour-tight and in an atmosphere of an inert gas, such as carbon dioxide. The Smith process differs from the Barbe in that the drying out phase does not take place within the machine.

The load is removed and drying is done in a second external appliance, usually a drying tumbler. Thus, in the Smith process, only the washing, rinsing, and extraction takes place in the machine.

Yet another difference is that the Smith machine is loaded from the end, a type of loading which has been copied in many of the

newer non-flam. plants. The cleaning drum is an extremely large cylinder, divided into four compartments, with a capacity of about 300 lbs. weight of garments. The division of the cleaning drum ensures an equal distribution of the load, and consequently smooth running during the extraction. It is also claimed that this division prevents the garments under treatment from becoming roped or entangled during the running of the machine. Another feature of this arrangement is that the goods are being dipped into the solvent and lifted out again, alternately, as the machine rotates.

Instead of using carbon dioxide to displace the air within the installation and so reduce the risk of fire, as in the Barbe machine, the Smith system is carried out in partial vacuum. After the machine has been loaded, the air within is removed by means of a vacuum pump, which is also employed for the circulation of the solvent. The amount of spirit required for the Smith process is about 200 gallons. The Smith machine is fitted with steam pipes, so that the solvent can be heated during the cleaning process. It must be observed that the temperature employed must be varied according to the nature of the load being treated. While the cleaning in warm spirit certainly results in an improvement in the result of the cleaning, many users of the Smith machine prefer to clean without heat. The rotation of the various phases in the cleaning process is that of washing and extracting alternately. In this way a number of quick washes or rinses with extraction between each can be made to produce excellent cleaning results.

It is claimed that the extraction under vacuum makes for a remarkable increase in the efficiency of the recovery of the solvent. The makers point out that "the average weight of spirit retained by the goods after extracting in the ordinary way is about 20%, whereas the spirit retained by the goods after extraction in a partial vacuum is only 7% to 10%, and that under no system would it be possible to recover this small amount to pay for the cost of recovery."

The Smith process may be modified so that the load is completely dried out in the machine, but it is interesting to note that the makers claim that drying out within the installation has several disadvantages, and may even be injurious to the goods being treated. It is explained that the heat necessary to dry out the last traces of the petroleum solvents, being generated in a limited space, would tend to dull white

or cream goods causing them to take on a grey or yellow cast. A second disadvantage is that certain colours are affected by heat and would tend to be altered in tone. This is particularly interesting in view of the criticisms made that the non-inflammable solvents may affect certain colours, particularly the colours of cellulose acetate. Another disadvantage of drying in the machine is that most of the textile fibres are injured by great heat. It is pointed out by the makers that "the natural moisture is all driven off, and the fibre of the material is attacked, the goods losing the soft handle and suppleness which is the chief effort of the finishing process to produce. Woollen goods once exposed to an undue heat always retain a harsh feeling." It is the author's opinion that much of the criticism made as to the noninflammable solvents causing harshness in woollen goods is not so much connected with the disturbance of what is called the natural grease content as with this loss of moisture due to the heat of the drying out process within the machine.

It would appear that the length of time required for the recovery of the last drop of the expensive solvent, causes a loss of condition in the case of certain goods, which is not regained afterwards.

The Smith plant has two outstanding advantages, these being a really effective cleaning, resulting from the use of vacuum combined with heat and a remarkably low solvent consumption. It is claimed that the solvent loss is about one gallon of spirit for every 100 lbs. weight of goods treated, or about 8% by weight.

### RECOVERY OF SOLVENTS

No system of dry cleaning could be made commercially successful unless the greater proportion of the spirit used were recovered over and over again. The treatment of soiled spirit is, therefore, an essential part of dry cleaning practice.

# THE RECOVERY OF SOILED SOLVENT

#### REDISTILLATION

There can be no doubt that the only absolutely certain way of repurifying soiled spirit is that of distillation. While the results of distillation are the best of all the methods of recovery as regards the

quality of the product, there are several points which have disadvantages. One disadvantage is that the process of distillation is expensive, because of its consumption of steam. The steam consumption is the main expense of distillation, and naturally varies with the solvent being used. In addition, the process is relatively long, and entails the carrying of large quantities of solvent in stock and circulation. These two factors of steam consumption and the time required for the distillation must, of course, vary with the type of apparatus.

Easy volatilisation is the essential property of any dry cleaning solvent, and during distillation the spirit is heated in order to convert it into a gas which is conducted through pipes into a cooling condenser. The drop in temperature in the condenser causes the gas to be reconverted into liquid. This condensed liquid is almost entirely pure solvent, all the solid soiling matters being left behind in the still.

The process of distillation may be carried out in an ordinary boiler or still, usually of copper, at normal atmospheric pressure. In this case it is frequently difficult to evaporate the last traces of the solvent by means of steam-heated coils alone, and consequently live steam has to be blown into the spirit. This distillation in steam causes the solvent vapours to be carried over at relatively lower temperatures than those required by the normal boiling points of the solvents. One disadvantage of introducing steam into the vapours is, naturally, that the condensate is a mixture of spirit and water, the latter having, of course, to be removed before the spirit is ready for use.

Distillation under normal pressures is also a lengthy process, and does not result in the solvent being in the best possible condition for re-use, and, moreover, the amount of "tailings", or irrecoverable residue remaining in the still, is quite considerable, especially in the case of some of the petroleum solvents.

Distillation under reduced pressures is undoubtedly the most efficient method of recovering volatile solvents. In this system, the air within the still and condenser is withdrawn by means of a vacuum pump. This exhaustion of the atmosphere within the still reduces the vapour pressures of the solvents, and, in consequence, reduces the temperature at which they boil.

# SET TLIN G

While distillation reproduces the solvent as nearly as practicable, in pure condition it is open to the criticism of causing the loss of the

soaps used in dry cleaning. These being non-volatile, are retained in the sludge in the bottom of the still. Because of this, and the length of time required for the distillation process, several other methods for the recovery of the spirit have been employed, particularly during the period immediately following the War.

One method depended upon the soiling matters in the solvent being coagulated or agglomerated into large particles, which were not kept in suspension by the solvent, and, therefore, sunk to the bottom of any container in which the solvent was kept undisturbed for some time. The settling of the soiling matters was effected by the use of certain reagents, whose essential function was to dehydrate the spirit. The reagents used were all dehydrating agents; that is, they had the power of attracting water to themselves. It would appear that the soiling matters are held in suspension by the quantity of moisture in the spirit, which forms a film round each small particle of dirt, and so prevents the particles from joining together to form larger aggregates which are then too heavy to be hold in suspension. The removal of the moisture film allows the particles to agglomerate, and thus settling takes place.

The solvent is usually treated with the dehydrating agent, and the mixture then agitated or stirred vigorously for a short time. The liquor is then allowed to stand, upon which the soiling matters settle more or less rapidly, according to the reagent used. Eventually all the dirt settles to the bottom of the container, leaving a layer of clear solvent which can be drawn off by means of a pipe, the intake of which is above the level of the sludge formed by the soiling matters.

Two dehydrating agents have had considerable application in settling processes. One of these is an acid, and the other an alkali. It will be readily understood that one effect of the acid method will be to decompose the soaps contained in the soiled solvent, but the main constituent of the soaps, the oleic acid will still be retained in solution in the spirit. The dissolved oleic acid can be reconverted into soap by the addition of a small quantity of ammonia to the settled liquor.

The dehydrating properties of sulphuric acid are well known, but this acid, in its concentrated form, has a specific gravity nearly twice that of water, and more than twice that of most of the solvents used in dry cleaning. This high specific gravity would cause the acid to

sink immediately to the bottom of the container if used alone. The difficulty, when using sulphuric acid for removing moisture from dry cleaning solvents, is to disperse the acid evenly through the solvent so that dehydration may take place before the acid sinks to form a layer of its own at the bottom of the tank. It has been found that the acid can be mixed with certain oils and fats to give homogeneous liquids. These specially prepared liquids, when mixed with soiled spirit, cause an even distribution of the acid as the oils or fats are dissolved away by the solvent itself. The use of sulphuric acid incorporated into oleic acid is the subject of a patent, and the compound is marketed under the protected name of "Lorcol".

The proprietary reagent, "Lorcol", for the settling of soiled spirit in dry cleaning can only be used under licence from the Lubricating Oil Recovery Co., Ltd. This concern permits licence-holders to make their own reagent from a secret formula supplied by the company, and also gives instructions as to the best type of settling apparatus to be used with the reagent. Should the licence-holder not wish to make the reagent himself, he can purchase it from the company.

The essential feature of this settling process is the thorough mixing of the reagent with the soiled spirit. This is usually brought about by mechanical means. One way of ensuring agitation is to arrange that the reagent and the solvent flow in two separate streams on to the buckets of a kind of "water wheel", which is rotated in a deep. narrow tank and causes complete mixing and agitation of the liquids. From the mixing tank, the dirty solvent is run into the settling tank proper, and allowed to remain for some time, probably a little more than an hour. The dehydration of the spirit takes place fairly rapidly as the sulphuric acid becomes dispersed through the spirit by the solution of the oleic acid; the particles of soiling matter then coagulate and fall to the bottom of the tank, so that eventually a layer of clear solvent is formed over a layer of sludge. By means of a pipe fitted to the tank in such a way as to leave the sludge in the bottom, the clear liquid may be drawn off and can be used for the cleaning of all but whites and very light-coloured goods. The settled spirit obtained from this process is usually a very pale straw colour, although the actual colour depends on the class of goods previously treated in the solvent.

The settling of soiled spirit by means of alkaline reagents depends upon the dehydration of the spirit with the aid of saturated solutions of sodium carbonate. It has been pointed out in connexion with the use of acid reagents that the essential condition is the complete mixing and agitation of the solvent with the reagent, followed by a period of quiescence. The same condition must be observed with the alkaline reagents.

In the acid settling, the specific gravity of the reagent is considerably greater than that of the solvent, so that there is an immediate tendency for the reagent to settle and carry down the soiling matters with it. In the case of alkaline treatment, however, the sodium carbonate has to be added in the form of its saturated solution, which does not show a greatly increased specific gravity compared with the spirit, and consequently the process of settling is much more protracted than in the case of acid settling. The time of settling can be shortened by use of a substance such as French chalk, which is kept in suspension in the solvent only during agitation and afterwards settles very quickly, carrying down with it the lighter impurities in the spirit.

An actual specification of a settling process, using the alkaline method, is as follows: To clarify 300 gallons of dirty spirit, 3 lbs. of 58° alkali and 1½ lbs. of French chalk are mixed with about ¾ gallon of water. This gives a fairly saturated solution of sodium carbonate with the French chalk in suspension. The soiled spirit is thoroughly stirred and agitated, while the solution is added. The agitation is continued for some time to ensure the complete mixing of the solution into the solvent. The saturated solution of alkali acts as a dehydrating agent and removes the moisture from the spirit; this causes the coagulation of the larger particles of soiling matter, so that they settle by their own weight. The lighter and more flocculent particles are carried down by the settling of the wet French chalk. Settling by this method takes about three hours, but the process would probably have to be slightly altered according to the condition of the dirty solvent and its specific gravity. Furthermore, after settling approximately three hours, the clarified spirit is run through an asbestos filter, thus ensuring the elimination of any impurities not carried down by the French chalk. The asbestos filter is arranged so that it can easily be removed at frequent intervals and the impurities destroyed by burning. The asbestos can be used over and over again.

The alkaline method does not cause the decomposition of the soaps which may have been used in the previous dry cleaning processes, and consequently the recovered solvent requires no addition before being used again. This method may also be said to eliminate the possibility of any injurious chemical finding its way into the cleaning solvent and coming into contact with goods being cleaned, as might be the case when insufficient time for settling was allowed with the acid method. Further, the alkaline solution would not be corrosive in its action on the metal of storage or processing tanks, as is liable to be the case when acid compounds are used.

It may be pointed out that the clarification of soiled non-inflammable solvents by means of the alkaline method is adopted with some of the new systems, especially those using trichlorethylene. There is no likelihood of acid being used in this connexion, owing to the undesirability of the new solvents in acid condition. Probably the chief reason for washing the non-inflammable solvents with a dehydrating agent before re-use is to ensure that they are free of moisture, which is an important factor in causing decomposition in certain circumstances.

Yet another example of settling the soiling matters from used dry cleaning spirit by means of alkaline agents is the employment of the caustic alkalies as dehydrating agents.

In one method, used on the continent, caustic potash is the chemical which attracts the moisture from the spirit. The caustic potash, in the form of a highly concentrated solution, is thoroughly mixed with the dirty solvent in specially designed apparatus for some time, after which the mixture is allowed to stand. In time, the potash solution settles to the bottom of the container, carrying down with it the soiling matter previously held in suspension and leaving the clarified solvent ready for decanting from the top.

One firm who make agitators and settling tanks as complete units, stipulates the use of caustic potash in stick form, but it will be found that the commercial form, in lumps will answer just as well in practice. Compared with the settling by means of sodium carbonate, the process using caustic potash would appear somewhat expensive, and in addition, the sludge, being a caustic solution, would be much more difficult to handle.

Caustic soda may also be used to accelerate the coagulation and settling of the soiling matters from the dirty spirit.

It is held by some authorities that caustic alkalies exert a secondary function of neutralizing the free fatty acid, which is always present in benzine soap, with the formation of a neutral soap. This neutral soap, being only slightly soluble in the cleaning solvent, is precipitated out and settles, carrying the suspended matters with it.

It must be pointed out that when using the caustic alkalies great care must be taken to ensure that only the exact quantity necessary be used, as too little would mean incomplete settling, while too much would cause a certain amount of the caustic alkali to be carried forward into the reclaimed solvent with the subsequent risk of damage to goods being treated.

Owing to the possibility of damage arising from the use of caustic alkalies, many experiments have been carried out in an endeavour to replace them with some other less dangerous chemical. Some measure of success attended the attempt to use trisodium phosphate in place of caustic soda. The trisodium phosphate was able to function by neutralising the acid benzine soap, and when used in conjunction with activated carbon, a clear spirit quite free from colouring matter could be obtained.

#### CENTRIFUGAL CLARIFICATION

The methods of recovering used solvent after dry cleaning seem to have remained stationary for many years after dry cleaning had been adopted as a commercial process. It has already been pointed out that distillation was the only means for the reclamation of the spirit until quite recent times, when chemical settling was introduced. Following this introduction of gravity clarification, there appeared on the market a number of machines designed to effect separation of the soiling matters by means of centrifugal action. The essence of each machine was to introduce the soiled spirit into a vessel which was rotated at a very high speed, so that the centrifugal force exerted on the particles in suspension would cause them to be thrown to the sides of the vessel while the clarified spirit was retained in the centre.

It is an established fact that the weight of a body is a determining factor in the amount of force resulting from centrifugal action. In the case of soiled solvent, all the factors are the same with the exception of the relative weights of solvent and soil. The difference

between these weights is further dependent upon the difference between the relative specific gravities of the liquor and the solids held in it. In the case of the petroleum solvents, there is a considerable difference between the specific gravities of the spirit and the soiling matters and consequently the separation by means of centrifugal machines is comparatively easy.

It will be noted that every recovery process dealt with up to the present has been a separate and secondary one to the actual cleaning process, and has been carried out intermittently as sufficient spirit became soiled. But the introduction of the centrifuge for the clarification of soiled dry cleaning solvents, opened up the possibility of the reclamation being part of the cleaning process, cleaning and recovery following each other in continuous flow.

For continuous flow operation, the centrifuge is connected directly to the cleaning machine. A circulating pump draws the solvent from the cleaning drum and delivers it to the centrifugal clarifier. By continuous passage through the clarifier the soiling matters are removed from the spirit almost as soon as they are removed from the garments being cleaned, while the clear spirit is returned to the machine. This method is certainly a great advance, for the goods need no longer be revolving for certain periods in solvent containing a large amount of soiling matter in suspension, but can all the time be treated in solvent free from soil. Further, no rinsing is necessary as in the older method when fresh solvent had to be run into the cleaning drum. The continuous flow method produces better results with considerable saving in the time of operation, while it is obvious that the process requires much less attention.

Centrifugal separators may be used to assist the separation of the soiling matters after the solvent has been treated with certain reagents as in the case of chemical settling. In this case the time of settling can be almost entirely saved by passing the treated solvent through the centrifuge after thorough agitation with the coagulating and dehydrating agent. The use of sodium carbonate as settling agent is the most suitable in this instance, as the resultant sludge will not exert an injurious action on any part of the centrifuge as would be the case if acid or caustic alkalies were used.

There are three makes of centrifugal clarifier which have had a fairly wide adoption in this country, the Sharples, the De Laval and

the Lang, while the Krupp and the Pratique are better known on the Continent than here.

# THE DE LAVAL CLARIFIER

The De Laval clarifier is an example of one type of machine now in widespread use among dry cleaners for the continuous separation of the soiling matters removed from the goods during the dry cleaning process.

In general, the layout of plant for continuous clarification is the same whatever the type of centrifuge employed, in that the dirty spirit is drawn from the bottom of the washer and delivered by means of a pump to the centrifuge. The passage of the solvent through the clarifier removes the soiling matters and the spirit is discharged into a suitable tank, from which it is drawn by a second pump and returned to the washer. Thus the solvent is in continuous circulation during the cleaning process, and the soiling matters are removed from the spirit as soon as they are removed from the goods being cleaned.

Yet another advantage of centrifugal clarification is that the method is entirely a physical one; no chemical process is involved and there can be no criticism arising out of the use of chemicals, as in the case of some of the processes of settlement by dehydration.

The De Laval clarifier consists, in the main, of a bowl-shaped chamber in which the clarification takes place. This bowl rotates at a speed of 6,000 to 8,000 revolutions per minute. The dirty solvent is pumped through a pipe so that the liquor enters down the centre of the bowl, and travel upwards through a series of vanes or cones.

The distance between the vanes is such that the solvent is split up into a very large number of thin films. The spinning vanes cause the dirt, under the action of centrifugal force, to travel to the underside of the cone and then downwards to the edge of the cone. At the edge of the vane the solids thus separated are thrown into the space of the bowl and become packed round the sides of the vessel in the form of a solid cake. The treated solvent passes upwards between the vanes, overflows at the top of the bowl and is collected in a small tank for return to the cleaning machine by means of a pump, or, in certain cases, the clarifier is mounted on a plinth at a height sufficient to allow the return flow to the washer to be a matter of gravity.

A sight glass fitted to the outlet of the clarifier permits the progress of clarification to be observed. Fluff, buttons, pins, etc., are prevented from getting into the pump by fixing a special strainer box as close as possible to the outlet of the washer. The De Laval strainers are either of the circular basket type or of the rectangular plate type. It must be noted that the latter type of strainer can be cleaned while the clarifier is working, but the circular type can only be cleaned after interrupting the circulation of the solvent. The pumps used in the De Laval circuits are usually of the centrifugal type, as these have been found to be extremely efficient when used for the circulation of spirit between the De Laval and the washer. In cases where the spirit has to be lifted to any great height, or to be forced through a long or complex system of pipework, the duty imposed on the pump is such that a centrifugal machine cannot be used and a special rotary gear pump has to be employed.

The De Laval machine maintains its efficiency as a clarifier so long as the bowl has space enough for the separated dirt, and there is no doubt that the cleaning process is considerably reduced, thus giving greater capacity to the washers.

It has been found that a De Laval clarifier working in conjunction with a large Barbe machine reduced the time of cleaning from 35 minutes to 10 minutes, and eliminated the rinsing phases. Another advantage claimed for the De Laval is that it obviates the necessity for dusting goods, either before or after cleaning, the reason being that all loose dust is trapped at once within the clarifier, and no soiling matter is left in the solvent which would otherwise resettle on the garments being treated and cause the dusty appearance.

Generally, several loads of work can be treated before the machine has to be cleaned. The removal of the dirt is quite simple, as it can be removed as a solid cake from the bowl. As no chemical action on the solvent or the soaps dissolved in the solvent takes place, a saving in the cost of soaps results. It is also claimed that as the centrifuge causes the separation of water from the spirit the cleaning system is freed from moisture

The De Laval can be conveniently worked in circuit with two, or even more, washers. Fixed between two washers, it may be operated simultaneously on both, or worked intermittently. With intermittent working, the centrifuge could clarify the spirit from the one machine

while the other was being unloaded and reloaded, so by the time the second washer was ready, the cleaning of the load in the first would be complete. In this case, the pumps of the De Laval would serve to withdraw the solvent from the one machine and deliver it to the second.

It will be obvious that by passing all used spirit through the De Laval before return to storage, no collection of dirt can take place in the storage tanks, the cleaning out of tanks is almost completely eliminated and, since the same quantity of solvent is in continuous circulation all the time, the amount of stocks of solvent required to be carried is very much lower than in the case of cleaning by the ordinary methods. The makers of the De Laval claim that storage is reduced by at least 75%. Since the spirit can be used over and over again, simply by passing it through the De Laval, no accumulation of soiled spirit, awaiting reclamation by distillation, occurs and a further saving is effected, not only by the economy in solvent, due to less distillation, but also by the reduced consumption of steam and water otherwise used during distillation.

The speed of the De Laval is only one-third that of some of the other types of centrifuge, and consequently the working parts of the machine are less liable to wear, with increased expectation of life and a reduction in maintenance costs. The De Laval is marketed in a range of five standard sizes for use with the petroleum solvents.

De Laval Size No.	Nominal Capacity in g.p.h.	Size of washer for which suit- able. Gallons of spirit used	Approx. H.P. required to Drive
200	150	10	1
300	300	20	$\frac{1}{2}$
500	700	40	I
700	1,200	8o	2
900	2,200	160–300	4

Special types of De Laval are supplied for use with hot spirit and with the non-inflammable solvents.

#### MODERN AND DYEING CLEANING PRACTICE

The cleaning out of the De Laval centrifuge does not entail any long interruption of the clarification process. When it is required to clean the bowl, the hinged cast-aluminium cover is opened by unscrewing the three clamping screws, which are easily worked by hand, and throwing back the cover. This reveals the top or hood of the bowl.

This hood is held down tightly on the bowl itself by a ring which screws on to a special thread on the outside of the bowl.

The removal of the ring enables the hood to be lifted out of the machine, revealing the bowl with the distributor.

The distributor must be taken out in order that any solid matter between the discs may be cleared away.

This can be done by immersing the discs, without dismantling, in clean solvent. It is essential that the discs be perfectly clean before re-assembling, otherwise there is risk that the balance of the bowl may be out of true.

The largest accumulation of dirt will be found round the walls of the bowl, and usually the deposit has been packed so tightly by the centrifugal action that it forms a solid cake, which may easily be removed in one piece. It is surprising how much solid matter can be removed from a centrifuge in a comparatively short time of working.

It is claimed that the De Laval can be cleaned out in less time than any other type or shape of bowl. It is said that, assuming an average type of labour and average conditions, the total time during which a De Laval clarifier is out of commission for cleaning need not exceed the following:

Size	Total Time for Stopping, Cleaning, and Running up to speed		
300 500	7 minutes		
500	9 "		
700 900	12 ,,		
900	15 ,,		

# THE SHARPLES CENTRIFUGAL CLARIFIER

The Sharples centrifuge differs from all others in that the revolving component is of small diameter, but the speed of the revolution is much greater than in other types of centrifugal machines. The machine is designed on the principle: "Make the rotor small in diameter and turn it fast". The bowl of the Sharples is, therefore, in the form of a long narrow cylinder, which rotates in certain types at the enormous speed of 17,000 revolutions per minute.

The weight of the bowl is only some 30 to 35 lbs. and the diameter only  $4\frac{1}{2}$  ins. The outer casing of the machine is long and tapering, almost like a fire extinguisher, and is of cast-iron. The centrifuge is belt-driven from above, the speed being obtained by direct drive with the necessary combination of pulley ratios.

As in all systems of solvent clarification, the dirty spirit is drawn from the washer by means of a pump, which delivers it to the centrifuge. In the case of the Sharples machine, the dirty solvent enters at the bottom of the clarifier and rises through the whole length of the clarifying cylinder. It is claimed that this arrangement makes for a perfect streamlined flow of solvent through the machine, and ensures a steady flow of spirit through the whole system. The centrifugal force exerted on the solvent throws the solid matters against the inside of the cylinder and allows the clear liquid to flow upwards in the centre, to overflow at the top. The clarified fluid is returned in circuit to the washing machines, by means of a second pump, or it may be returned by gravity feed if the centrifuge is mounted sufficiently high.

It will be noted that the Sharples clarifier has two outlets. This arrangement allows the machine to be used as a separator as well as a clarifier. Any water which may be in the dirty spirit is separated during centrifuging and is discharged from the machine through the second and lower outlet. In the normal benzine clarification the discharge of moisture rarely becomes more than a trickle.

The distribution of the dirty solvent in the Sharples machine is effected by the simple device of inserting a small baffle plate in the inlet to the bowl, which merely breaks up the flow of the solvent as it enters the machine. As there may be some possibility of the column of solvent moving upwards without being rotated by the bowl, the clarifying department is divided into three by means of a Y-shaped

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partition, which rotates with the bowl. This prevents the liquid slipping within the bowl.

The cleaning of the Sharples bowl is comparatively simple. The drive on the top of the machine is easily uncoupled and raised out of the way of the hood or covers.

After removal of the covers, the long cylindrical bowl can be readily lifted out and, since its weight is only 35 lbs., it can be easily handled by one man. In addition, the straight cylinder is very quickly cleaned.

It is claimed by the makers that the Sharples clarifier is quick starting, requiring only three minutes to reach clarifying speed, and is correspondingly quick stopping, being braked from top speed to stop in only two minutes.

As the Sharples can be cleaned out in ten minutes, the entire time required for stopping, cleaning and re-starting is only fifteen minutes.

# THE LANG CLARIFIER

The Lang clarifier has had a wide adoption in the dry cleaning industry. It is particularly well built and gives the impression of solidarity. The machine stands on four legs, which are all part of the main casting for the body of the machine. The legs are fixed by bolts to the floor, thus forming a firm foundation secure from vibration.

The clarification chamber in the Lang plant is bowl-shaped, similar to that of the De Laval. The solvent being treated is divided into a series of very small streams by means of a distributor, consisting of a number of cones mounted on a spindle so that the space between them is extremely small. The discs or vanes revolve with the machine, and, in consequence, the spirit is resolved into a number of films and submitted to centrifugal action.

The centrifuge is connected up in circuit with the washer, strainer box, and two gear pumps. As in all the cases of centrifugal treatment, the solvent is drawn from the cleaning machine by means of one pump and delivered to the clarifier. From the clarifier the recovered spirit passes into a reservoir or tank from which it is drawn by the second pump and returned into circulation through the washer again.

The soiled spirit flows down the centre of the distributing discs to the bottom of the bowl and then rises up between the vanes so that the clarified liquor overflows at the top of the bowl.

The Lang machine is fitted with particularly large sight glasses, giving clear views of the progress of the operation. Another usful accessory is a speed indicator which is fitted on the front of the pedestal of the centrifuge. The Lang clarifier is made in four sizes, with capacities for clarifying benzine at the rate of 880, 1,760, 2,250 and 4,400 gallons of solvent per hour respectively.

The cleaning of the Lang machine is carried out by first throwing the driving belt on to its loose pulley, and the bowl is braked by means of two double-acting brakes, hand operated. The outer cover is removed by releasing the three clamping screws and throwing the cover back on its hinges. This discloses the hood of the centrifuging chamber. The hood is removed by being unscrewed and lifted off. The distributing vanes are then lifted out of the bowl. The bowl itself is lifted out by means of a small crane which is fitted on to the body of the machine. In certain cases, the Lang clarifier is equipped with an automatic cleaning device, which enables the solid dirt to be removed from the bowl easily and quickly.

# CENTRIFUGES FOR THE NON-INFLAMMABLE SOLVENTS

It will have been noticed that, so far, the centrifugal machines described have been intended for the treatment of the older petroleum solvents, in other words, solvents which have specific gravities smaller than that of water, which is 1. As all the newer non-inflammable solvents are considerably heavier than water, treatment by centrifugal machines is much more difficult, and, in fact, is impossible in the machines which have proved successful with the benzine solvents. With the non-inflammable solvents, the difference between the specific gravity of the solvent and the specific gravity of the soiling matter is extremely small, and this fact accounts for the difficulty encountered when treating these solvents.

The problem has been solved by making a modification in the direction of travel of the solvent through the clarifier. Instead of permitting the spirit to travel along the lines of direction of the centrifugal force, the flow is deliberately baffled across the direction of this force.

Thus the spirit has to flow alternately in upward and downward directions in its passage through the machine from the centre of the bowl to the outer edge.

One effect of the high specific gravity of the non-inflammable solvents is that the soiling matters held in suspension in the used spirit are of two kinds: those which are lighter and those which are heavier than the solvent. The modification of the clarifying bowl described causes the light dirt to collect on one side of the baffle while the heavy dirt collects on the other side.

Both the De Laval and the Lang companies produce special centrifuges for the treatment of the non-inflammable solvents. In the case of the Lang machine, special features claimed by the makers are the hinged metal hood which makes an air-tight joint with the frame of the machine and so ensures complete vapour tightness (an important consideration when dealing with expensive and, in certain cases, toxic solvents like the chlorhydrocarbons); simplicity of operation; and high efficiency.

In the De Laval "Non-flam" separator, one interesting feature is the continuous separation and discharge of water from the solvent. The De Laval Company claim that complete dehydration of the solvent is effected. Yet another feature worthy of mention is the self-draining bowl of the De Laval centrifuge for the new solvents; the liquid which would normally remain in the bowl is automatically discharged as the bowl decreases in speed and when the machine is opened, all parts are left free from liquid and at room temperature. The dirt extracted is left in practically the form of a dry cake.

The separators for the non-flam solvents may be operated by a direct electric drive or from line shaft drive.

Because of the difficulty of treating the heavy chlorhydrocarbons in centrifugal machines of the type employed with the petroleum solvents, the first installations designed for the newer solvents relied upon filtration for the continuous removal of the soiling matters. With the introduction of clarifiers designed to treat the heavier solvents, there have been many indications of a growing preference for such centrifuges to be included as part of a non-inflammable installation, and in many cases the centrifuge is now a standard accessory, especially on the Continent.

One advantage claimed for the clarifier is the simplicity of removing the dirt as compared with the corresponding operation with a filter. The cleaning of a filter is stated to be much more cumbersome and unpleasant. The cover has to be removed and a number of tubes

and channels have to be cleaned. The dirt at the bottom of the tank has to be removed, and this is a rather unpleasant procedure. All this work is claimed to be simpler with the centrifuge.

#### FILTRATION

The removal of the soiling matters from used dry cleaning solvents by means of mechanical filtration has attracted a great deal of attention from certain investigators during the last ten years. The ordinary filter press does not answer efficiently when operating on the usual dry cleaning solvents. A reason for this failure was given by Professor J. W. Hinchley, in a paper read at a joint meeting of the Chemical Engineering Group of the Glasgow Section of the Society of Chemical Industry. After pointing out the difficulty of disposing of the residues from the distillation and settling processes of purification, Professor Hinchley said that if the dirt could be removed by a simple mechanical operation, the solvent would be suitable for re-use, and since it would still contain a certain amount of grease in solution, a great improvement in the handle of the cleaned garments would result.

The following were his observations on the nature of the grease and dirt obtained from used solvents: "The grease obtained from dry cleaning spirit is interesting, in that it is common to find over 70% of mineral oil and unusual to find more soap than has been added for the dry cleaning process. The dirt is also an interesting material; its minute particles are so small that it seems impossible for any commercial filtering process to be adequate for their separation. In fact, the engineering difficulties presented by the material are greater than would be realized in a manufacturing process on account of the variability of the articles cleaned."

The outcome of the investigation of the problem of the removal of finely divided particles suspended in a liquid was the invention of the streamline filter by Dr. Hele-Shaw, and Professor Hinchley realised the possibility of applying this type of filter to dry cleaning processes. It was demonstrated that if a liquid carrying solid particles is forced between sheets of paper held together so as to form a pack, separation of the suspended matters takes place at the edge of the pack. The sheets of paper in the pack are placed so close together that only the roughness of their surfaces prevents close contact and leaves room

for the flow of the solvent through the pack. If the paper be perforated the number of edges may be correspondingly increased and with that the rate of separation.

Professor Hinchley experimented on the laboratory scale for several months, using the various kinds of solvents employed by dry cleaners. From these experiments, it was found that the roughness of the surface of the papers used in forming the filter pack determined the effective width of the channels through which the spirit must flow, and that as the fibres of the paper were elastic, some form of control or check on this width was desirable. Consequently, the pressure on the pack was controlled by means of springs, so that the papers must be brought closer together, so reducing the width of the channels.

The stream line filter pack (or edge filter) may take several forms. One type consist of sheets of paper of standard size  $(8\frac{1}{2} \text{ ins. by } 6\frac{3}{4} \text{ ins.})$  pierced with 32 large holes and 53 small ones, the dirty solvent entering the filter by the large holes and being discharged clarified through the small ones. A second type takes the form of sheets of paper in strips pierced with a line of holes. The solvent is introduced at the outer edges of the strips and leaves through the holes, round the edges of which the dirt and soiling matters are deposited.

Using many types of filter pack, it was found that there was no difficulty in controlling the pressure of the pack, so that effective filtration was obtained at a rate applicable to commercial dry cleaning, but the removal of the dirt from the filter when the packs had become too soiled or choked for successful clarification presented a problem which called for further investigation. The usual methods of cleaning commercial filters were of no avail.

The simple method of reversing the flow of the spirit through the filter was unsuccessful, but after several experiments it was found that the filter could be cleaned by producing the reversal of flow by means of compressed air. Professor Hinchley's own description of the experimental form of laboratory filter is as follows:

"An enclosed form of laboratory filter was used, with a vertical cylinder as a pressure vessel containing the liquor to be filtered. By means of a bicycle pump, the air in the upper part of this vessel is compressed to the pressure of filtration.

"When the rate of filtration had fallen to a point of inefficiency, the compressed air in the upper part of the cylinder was made to produce

the reversal of the flow, itself passing through the filter after the reverse filtrate. This "blow-back" method was thoroughly tested, and it was found that the original rate of filtration could be restored practically continuously, although on some occasions the original rate was not quite restored.

"The dirt is discharged as a sludge or thick liquor, and may be distilled at once. With a normal dirty liquor from 5% to 10% of the liquor is required for the "blow-back", so that over 90% of the spirit may be obtained in a filtered condition."

The streamlined filters used on a commercial scale follow closely the laboratory type used by Professor Hinchley in his experimental work. The commercial filter comprises a series of units of filter columns. The columns are built up of filter packs made from circular sheets of special paper mounted on metallic rods, the whole being assembled in the filtration vessel. Springs for the regulation of the pressure on the columns are fitted on the outside of the vessel or so arranged that they can be operated from the outside without opening the filter. The dirty solvent enters the filter at the bottom of the machine and flows from the edges of the paper rings, between the layers of paper in extremely thin films to the centre of the pack, where perforations allow it to pass upwards round the centre rod into a collecting chamber at the top of the apparatus. From the header, or collecting chamber, the filtered spirit leaves by means of a pipe at the top of the machine.

The dirt and soiling matters in suspension in the solvent are deposited on the edges of the paper rings or outside the packs. In practice the filtration is continued until a pre-determined pressure is shown in the filter, when the dirt has to be cleared from the packs. The period of filtration will obviously be dependent upon the rate of filtration and the character of the soiling matters present in the dirty solvent, but the normal time of operation of the filter should be more than an hour. When the time for removing the dirt comes, compressed air is admitted to the top of the filter and blows through the pack with a reversal of flow of a certain amount of filtered spirit. The removed dirt forms a sludge in the bottom of the filter, from which it is withdrawn periodically to be distilled.

The presence of moisture in the dirty spirit may cause some difficulty in the application of the streamlined filter on a commercial scale.

This moisture may arise from the garments which have been cleaned in the spirit or from certain kinds of soaps used in the cleaning process. In view of this, the soaps employed should be completely soluble in the cleaning spirit. There is a decided trend towards the introduction of a controlled amount of moisture into dry cleaning solvents. Whether the soaps used in dry cleaning should be completely soluble for the best results is being closely investigated at the present time, and the question cannot be answered with any degree of certainty in the present state of knowledge.

During filtration on a large scale, it has been found that the efficiency and rate of filtration is greatly improved by heating the solvent before introduction into the filter chamber. The commercial filter is therefore equipped with a small steam-heated coil, controlled by a thermostat.

The chief advantage of the filtration process for the clarification of dry cleaning solvents is that no reagent is added to the spirit, and consequently all danger to fabrics which are to be cleaned and which may be injured by certain chemicals used in the reclamation of spirit is eliminated. A second advantage is that the oils, soaps and fats are not removed from the solvent, so that the treated garments have a softer handle than when cleaned in spirit containing no dissolved fats. The comparative ease with which the dirt can be removed and disposed of after the filtration process is yet another advantage.

The inquiry into means of continuous clarification of dry cleaning solvents resulted in the adoption of what is now known as "filter aid". This was thoroughly investigated by A. E. Hatfield in the works of Messrs. Achille Serre, Ltd., and there was devised and patented a process of continuous clarification, in which the dirty solvent was reclaimed for further use by mechanical filtration, in conjunction with a particular form of silica.

The use of silica in a finely divided condition has been known for some time in connexion with the refining of cane sugar. The silica is added to the solutions of the sugar in order to assist in the filtration. The silica is in the form of Kiesulguhr, repeatedly ground to a fine powder.

There are two interesting patents referring to the improvement of Kiesulguhr to be used as filter aid. The first patent, dated 1923, deals with the treatment of Kiesulguhr to improve its quality and to render

it more effective as a filter aid. The process consists of mixing the Kiesulguhr with certain chemicals, such as common salt, and heating to a suitable temperature. The resulting product is a white powder of low density, much more fluffy and of softer consistency than the original, the degree of improvement being dependent upon the temperature and the time of treatment. For example, the heating or calcination of a finely divided mixture of the siliceous earth and a salt of an alkali-forming metal at 1,800°F. in a muffle furnace for an hour would give a product with a density of 10 lb. per cu. ft. from an original powder with a density of 17 lb. per cu. ft.

The second patent is really an amendment to the first. In this, the process of improving the Kiesulguhr without the admixture with alkaline salt is described. The patentee claims that the use of alkaline salts during the calcination is not necessary, provided the silica is in a sufficiently finely divided condition beforehand. Each of the two products was found suitable for the Hatfield-Serre process.

The Hatfield-Serre patent specification declares that in the various methods employed for the clarification of dry cleaning solvents (filtration, centrifugal separation, dissociation, precipitation), no cases of the effluent being perfectly free from moisture and foreign matter were found. They then state that by the use of "finely divided porous cellular silica", in conjunction with ordinary filtration, a completely clarified and dehydrated solvent is obtained.

The process entails the pumping of the solvent from the cleaning machine, the addition of a certain definite amount of filter aid (mixed with solvent to form a suspension), passage through an ordinary filter press and thence back to the machine, in continuous circulation.

In the Hatfield process the rate of flow of liquid is such that the solvent in the machine is changed every five minutes. The filter aid is specified as being sufficiently porous to maintain the emulsions and suspended dirt of the solvent in a porous state. Obviously, the film of filter aid, which is built up within the filter press, must be porous; otherwise, a pressure would be set up in the press which would retard the flow of solvent and eventually stop it.

The introduction of the filter aid is attained by way of a proportioning apparatus, in which the filter aid is mixed with dirty solvent to form a suspension, which is then passed into circuit of the solvent before passing to the filter press. This proportioning apparatus also

has the additional duty of agitating the mixture of filter aid and solvent.

The density of the filter aid is given as not substantially greater than 12 lbs. per cu. ft., "calculating on the wet process from the minimum bulk occupied by the filter aid when sieved into water and allowed to settle". The filter aid may also be used with certain alkaline reagents. These compounds, such as caustic potash, caustic soda, or ammonia, the carbonates of potash, soda or ammonia or other chemicals, such as aluminate of soda or silicate of soda, are introduced to assist in maintaining the film in the filter press in a porous condition. The reagents may be mixed with the cleaning spirit before or after the addition of the filter aid. When these substances have been used, neutralisation with inorganic acids is recommended.

The use of filter aid in conjunction with acid settling agents, as previously described is also covered by the patent.

The process may be carried out in any known type of pressure filter press, while several washing machines may be coupled with the same filter press, provided the press is sufficiently large to enable the pressure to be kept down to within practical working limits. Several loads of work may be cleaned without changing the solvent: it is changed when the accumulation of deposit within the press prevents an easy flow. At this point, a rise in pressure indicates that the press must be cleaned out. The solvent retained in the deposit is removed by blowing with air or steam. In certain large plants it is economical to use a large capacity main press in conjunction with the smaller press. The cake of deposit from the main press is washed down with solvent to form a suspension and then fed into the smaller press for recovery of the solvent. This mode of operation reduces the time during which the main press is out of action to a minimum.

The time of the Hatfield process is given as from 20 to 30 minutes, during which the goods are being treated with clean solvent, as the soiling matters are transferred to the filter press as soon as they are removed from the garments.

In this way, rinsing is not required in most cases. It will also be seen that the soaps used in the cleaning process are retained by the solvent and, in consequence, a much better handle is imparted to the treated goods.

The advantages of the process can be summed up as follows: (1) Economy of solvent; (2) more efficient cleaning; (3) improved handle; (4) reduction of time of cleaning process, hence increase in production; (5) removal of bacteria, which are held in the filter.

The continuous clarification of dry cleaning solvents by means of mechanical filtration combined with the use of "filter aid" calls for special attention to the character of the soap employed in the cleaning process. It is obvious that the soap must not be retained in the filter in any form. Retention would, of course, result in choking up of the filter and consequent reduction in the rate of flow of solvent through the filter press. In addition, the length of time between the cleaning-out operations would be very much reduced and the press be out of commission for a greater proportion of the working time than would be economical.

The patentees of the Hatfield-Serre dry cleaning process are also the patentees of a series of processes for the manufacture and production of soaps which are not only specially adapted for use in the dry cleaning but may also be employed in normal laundry work. In the preparation of these soaps great care is taken to ensure that they are completely soluble in the usual solvents. The specification of the patent describes the test for solubility applied to soaps by the patentees.

"A test for solubility is to dilute one portion of the soap in not less than 500 parts (preferably 1,000 parts or more) of the solvent or liquid used, according to the variety of soap and the purpose to which it is to be applied. With the soaps prepared according to this invention, dissociation does not take place in the sample after standing for twenty-four hours at a temperature of 32°F. It is found also that, after goods have been cleaned by soaps of this character, the materials are free from soap and of foreign matter and no trace of foreign matter can be extracted even when employing a higher solvent, such as ether, and the like. Such a test must, of course, be applied after thoroughly rinsing the goods with soap-free solvent or liquor."

Apart from the question of solubility of the soap for the purpose of preventing the filter presses from clogging up, it is observed that such highly soluble soaps give better cleaning result.

It has been proved that soaps of poor solubility and low emulsifying power in spirit are apt to retain the dirt which has been released from the articles being treated, and so redistribute it over the materials. This property is most noticeable in the dry cleaning of white and cream woollens, and results in what is known as "lead coloured whites". A somewhat similar effect is mentioned in the patent specification as being noticeable in certain instances in connexion with laundry washing, where soap with associated dirt may be re-deposited on the fibre. Furthermore, the retention of soap in the fibres of cleaned articles may cause difficulty in the subsequent process of spotting, in that, upon the applications of reagents containing water, as is usual for local treatment, the soap is dissolved with the formation of rings or sweals. The patentees of these special soaps claim that soluble soaps prepared by their processes do not produce these ill-effects, and that improved dry cleaning is obtained by the use of such soaps.

The production of soaps of such high solubility is found to depend upon two factors. The first of these concerns the fatty acid employed in the manufacture of the soap. The fatty acid should be such that no solidification or crystallisation of solid components takes place above 36°F., and it is preferable that the acid should still remain liquid at temperatures below freezing point, 32°F., except in certain circumstances, when the critical temperature may be as high as 50°F.

The second factor is the presence of an emulsifying or carrying agent. The function of this emulsifying agent is to maintain the soap in a perfectly soluble condition. These agents are sometimes referred to as auxiliary solvents, and comprise a wide range of chemicals. It is found that an excess of fatty acid in the soap will act as an emulsifying agent, so that when dry cleaning soap is made from oleic acid a large proportion of the acid may be in the free state.

The English Patent 289,582 (May 3, 1928) describes three types of soaps, which are examples of the use of free fatty acid, chlorinated hydrocarbons and cyclohexanol as the respective auxiliary reagents.

Example 1.—"100 lbs. of spirit-soluble soap in which 25% to 35% of the fatty acid is left unsaponified". 70.58 lbs. of pure oleic acid (known as special pale or special frozen oleic acid, 99.5% to 100% pure or any similar oleic acid or oleine having a melting point of less than 36°F., and preferably less than 31°F.) are placed in a jacketed mechanical mixer and raised to a temperature of at least 170°F. To this is added slowly with agitation an aqueous solution containing 12.5 lbs. of pure potassium carbonate (99.5% to 100% pure), or an equivalent amount of sodium carbonate, or of the hydroxides of

potassium or sodium, or of ammonia liquid of sp. gr. o.880. The solid alkalies should be previously dissolved in 8 lbs. of boiling water. The alkaline solution should be added slowly with agitation until all the fatty acids are saponified to the extent of 65% to 75%. Considerable ebullition takes place during saponification, but this may be controlled by adding a little alcohol or industrial methylated spirits during the process. Agitation is continued until the desired saponification is attained and the mass appears as a transparent homogeneous liquid. Nine pounds of alcohol or industrial methylated spirit are added and mixed thoroughly with the soap, which may then be removed from the vessel."

Example 2.—"100 lbs. of neutral spirit-soluble soap, with chlorinated hydrocarbons." 45.55 lbs. of pure oleic acid, or any similar oleic acid or oleine having a low setting point, are placed in a jacketed mechanical mixer and raised to a temperature of at least 170°F. To this is added an aqueous solution containing 11.38 lbs. of pure potassium carbonate or an equivalent amount of the hydroxides of potassium or sodium, or of liquid ammonia.

The alkaline solution should be added slowly until the fatty acids are completely saponified. When the mass, which should be homogeneous with a transparent appearance, has subsided, 27.51 lbs. of perchlorethylene, pentachlorethane or trichlorethylene, or the like are added. Agitation is continued until the mixture is in a liquid state, when it is removed from the vessel."

Example 3.—"100 lbs. of neutral spirit-soluble soap, with cyclohexanol". 45.55 lbs. of oleic acid are placed in a jacketed mechanical mixer and raised to a temperature of at least 170°F. Any reasonably pure oleic acid or oleine may be used, but the setting point should be preferably below 60°F., and to get the best results the more solid fatty components should set below 36°F. The fatty acids are saponified as in the previous cases until a homogeneous transparent mass is obtained, when 27.51 lbs. of cyclohexanol are added. Agitation is continued until the mixture is in a liquid state. Additional cyclohexanol may be added before, during or after saponification to increase the solvent action either in water or in the spirit."

During the past few years, C. L. Bird has conducted some important researches on the effects of soaps in the dry cleaning bath, and has arrived at certain definite conclusions. It has been demonstrated

that the potassium soaps are more soluble than those made from other alkalies and that the oleic soaps are more soluble than soaps from other fatty acids. Bird also showed that the soaps made by the combination of sodium alkalies with stearic and palmitic acids were unsuitable, while the presence of free oleic acid in potassium oleate soap produced a stable solution in white spirit. In the cases where the soap is to be used in a filtration system over a long period the amount of free oleic acid should not be less than 40% of the total fatty acid. In his experiments on the auxiliary solvents, Bird found methyl-cyclohexanol, tertiary amyl alcohol, and dibutyl tartrate to be the best when examined for complete solubility.

# MANLOVE, ALLIOTT CYCLIC FILTRATION

A filtration unit, especially designed for use in conjunction with "filter aid" in the continuous clarification of dry cleaning solvents, has been placed on the market by Manlove, Alliott & Co., Ltd. In describing their continuous cyclic process of dry cleaning they claim a remarkable list of advantages:

(1) Better work, alike on woollens, whites, and silks; (2) greater ease and speed of operation; (3) goods are not washed in baths of dirty solvent; (4) various classes can be cleaned together in the same load; (5) adequate compensation may be made for the loss of the original cloth oils, which results in a greatly improved "handle" of the cleaned work; (6) output is increased according to various reports, doubled—even where filters replace modern clarification installations; (7) rinsing is dispensed with, except in very isolated cases; (8) the storage of solvent is greatly reduced; (9) the quantity of soap needed is considerably decreased; (10) "spotting" is much reduced, because the process removes many water soluble stains not touched by the usual dry cleaning methods; (11) distillation costs are greatly reduced; (12) the solvent is maintained in a highly sterile condition; (13) operation and installation costs are low.

The filtration unit consist of a filter press, a proportioner for adding the filter aid, a strainer, and a circulating pump. The body of the filter is built of cast iron, which may be lined with one of a variety of materials according to the use to which the filter is to be put. Thus, the lining may be of rubber, vulcanite, or lead. The filters are constructed to withstand pressures up to 60 lbs. per sq. in. in the larger sizes and up to 80 lbs. per sq. in. in the smaller ones. The body of the filter may be described as being of two parts, one fixed, the other movable by being hinged to the first part, like the door of a cupboard. The moving clam, or half, is hinged vertically and carries a joint ring of such composition that when closed the filter is spirittight. A combined screw and toggle lever serves as the gear, ensuring the firm closing of the filter. The height of the filter box is set so that a small truck can be run underneath it to receive the dirt removed during the cleaning out operations.

The essential part of the filter consists of a battery of filter leaves, usually seven. The leaf is built up of two surfaces of fine wire mesh, held together at the edges, with a fixed distance between them. Two types of leaf are in use, the all-wire leaf which consists of woven monel metal filter cloth panels carried on a heavy, open mesh iron draining support, strongly secured in iron edgings, and the leaf arranged to take cloth filter covers, which consist of heavy open mesh iron wire draining supports and edgings as before, but fitted with studs to hold the filter cloths to the frames.

The leaves are fitted into a header, which occupies the upper part of the filter, and can be fixed at 2-in., 3-in., and 4-in. centres, as required. On each side of the header two wheels are fitted, so that they run along hinged arms attached to the inside of the filter body. When the filter is opened for cleaning out purposes, the wheels of the header run along the arms and carry the header and its attached filter leaves clear of the body of the press. This device gives easy access to each leaf. Besides an opening for the discharge of filtrate into the header, each leaf has a bottom outlet for draining when necessary. The inlet for soiled spirit to the press is at the back of the filter near the bottom, and it connects to a passage which runs the full length. This passage is covered with a plate for the purpose of distributing the solvent. The feed inlet may also serve as a drain, or may have compressed air or steam connected to it.

The proportioner is an appliance designed to feed the filter aid to the dirty solvent in a definitely regulated proportion, so that the suspended soiling matters are treated with a fixed ratio of the filter aid. The proportioner is a tank provided with an internal strainer, a stirrer, and a float-controlled outlet valve, giving constant level of solvent in the tank. The proportioner operates in the following manner: A flow of solvent is taken into the proportioner tank from the filtered or clean spirit line and operates the constant level device so that a corresponding overflow of spirit into the dirty solvent line is caused.

Thus, dirty spirit intimately mixed with filter aid is pumped into the filter press. The proportioner is re-charged at the beginning of each cleaning operation, and so relatively large amounts of filter aid are washed into the filter just at the time when the large amounts of dirt are being removed from the goods under treatment. As the cleaning proceeds the amount of dirt in the spirit rapidly decreases, and this is compensated for by the amount of filter aid in the proportioner having become very small also.

The unit is completed by means of a circulating pump. This is normally of the positive belt-driven rotary type. In certain cases where the Manlove, Alliott system of cyclic filtration is employed with vacuum cleaning machines, a triplex plunger type pump is recommended. With vacuum machines it is also necessary to adapt a diluting pump to flush the proportioner with spirit from the return line, as this would be impossible by gravity flow.

The operation of the cyclic filtration process of Messrs. Manlove, Alliott is as follows: At the beginning of the day the washers and the circulating system are charged with clean solvent. The pump and the proportioner are started, and a small quantity of filter aid is added and pumped into the filter to form a pre-coat. This pre-coat acts as a preventative against fouling of the filter leaves by the dirt and ensures that the solvent is properly clarified at the start of the operations. The pre-coat is only necessary at the beginning, and is not repeated after the first load has been treated.

After the washer has been charged with its load, a measured quantity of filter aid is put in the proportioner tank. The cleaning machine is then set in motion, soap being added as required. The solvent is then kept in constant circulation by means of the pump, and on its way to the filter receives a proportionate amount of filter aid. In this way, a porous film of filter aid and soiling matter is built up within the press, this film effectively retaining dirt and moisture. The load is usually finished in from 15 to 25 minutes. The washer is unloaded without draining the machine, the solvent being used for the next

load, the only addition required being that of the filter aid to the proportioner. This cycle of operations is continued until the pressure of the filter has risen to 60 lbs.

The size of the filter is such that the plant will run for the whole of a shift or to the end of the day. Once the pressure has risen, it is necessary to clean out the filter and build up the pre-coat of filter aid before commencing operations again.

The cleaning of the filter can be effected quite easily. As soon as the rise in pressure indicates that the filtration is being retarded by the thickness of the cake on the leaves, the circulation of solvent is stopped and the liquor in the press drained off. Steam is then admitted to the body of the press, in order to evaporate the spirit retained by the cake, the spirit being recovered by passing through a condenser. When the cake has been dried out, the press is opened by operating the large hand-wheel attached to the screw and toggle lever. This causes the movable clam or half of the body to swing open and disclose the whole of the filter leaves. The two arms in the side of the body are lowered and the leaves and header run forward on the arms, in which position the dirt can be readily scraped off the leaves.

#### ACTIVATED CARBON RECOVERY

During recent times a great deal of progress has been made in connexion with the recovery of volatile solvents by means of activated carbon. The use of charcoal for decolorising certain solutions has been customary in many industries for a long time, particularly in the sugar refining industry, bone charcoal, for instance, being an efficient adsorbent.

It is interesting to note that the need for protecting troops against, gas attacks during the Great War was the incentive which directed attention and research to the properties of certain types of charcoal of adsorbing large amounts of gases. As one result of this war-time research, activated carbon became one of the ingredients in the canisters of gas masks, and it proved of great value in the adsorption of poison gas. Since then there has been an unending series of researches and studies into the properties and applications of activated carbon in connexion with solvents, with the gradual elaboration of the methods of solvent recovery.

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The action of the activated carbon is such that air containing vapours of solvents is "stripped" of those vapours by the simple process of passing the air over the carbon. The carbon has the property of adsorbing the solvent and allowing the purified air to pass on.

This property would, of course, be useless if the retention of the solvent was in any way firm or stable, but the great advantage of activated carbon is that the process can be reversed quite easily. The adsorbed solvent can be driven off from the carbon by the simple process of heating, or by blowing steam through the mass. The adsorption of solvent can be carried on until a certain limit or saturation point is reached, after which the solvent is recovered and, at the same time, the activity of the carbon is restored. This alternation of adsorption and regeneration can be continued almost indefinitely.

The peculiar power of adsorption of activated carbon is due in part to its porous structure and the adsorbed solvent may be considered as being held or trapped in the myriads of pores of the carbon. As already pointed out, the adsorption properties of bone charcoal have been known for a considerable time, but it is only recently that methods of increasing the adsorption capacity have been devised. By certain processes of treatment the capacity of the charcoal can be greatly improved and carbon activated by these methods will adsorb solvent vapours from the airs completely, until saturation point is reached. The general theory regarding the enormous improvement in the adsorption capacity is that the porous structure of the carbon is greatly increased by the special treatment, and that a very large surface of carbon is presented to the vapour-laden air stream.

The processes of activating charcoals to be used in solvent recovery are numerous, and have been protected by many patents, but in general the methods divide themselves into two categories—chemical and physical. In the chemical process the charcoal is treated with certain chemicals and heated to high temperatures, while the physical process entails the heating of the charcoal in special furnaces, in the presence of a selective oxidising air stream.

Whatever the method of manufacture, adsorptive carbon for industrial purposes is required to be firm, hard, dense, and pure; able to withstand the various phases of alternating adsorption and regeneration without deterioration, over a considerable period.

Activated carbon is capable of application to air containing solvents such as acetone, benzine, alcohol, benzol, trichlorethylene, carbon tetrachloride, and solvent naphtha. It is remarkable that the adsorption of the vapour takes place in almost theoretical quantity, even when the vapour content of the air is comparatively small. The speed of adsorption is also another feature to be noted. It has been shown that, with a vapour concentration of 1 to 2 grams per cubic metre, a well-designed and well-operated activated carbon plant has an efficiency of 95% to 98%, even with the air passing at such a speed that the time of contact is only about one second.

It will be realised that such a high degree of recovery from extremely small concentrations of solvent in air will be an economical proposition in dry cleaning. Whatever process or solvent may be employed for the dry cleaning, there is always a certain loss of spirit, which appears to be inevitable. With the aid of activated carbon this loss can be reduced to an absolute minimum, beyond which recovery would not be economical. This is particularly so in the case of the non-inflammable solvents, as these are recovered in almost theoretical quantity. The relatively high initial cost of these newer solvents makes the reduction of solvent loss very attractive. Trichlorethylene and carbon tetrachloride lend themselves readily to a very high degree of adsorption by means of activated carbon.

The essential appliances for the recovery of solvent vapours are exhauster fans, adsorber chambers charged with activated carbon, condenser, a separator, and a tank for the collection of the recovered spirit. The exhauster fans serve to draw the vapour-laden air from work-rooms with brushing tables, spotting slabs, and extractors, and from machines and pump it through the activated carbon adsorbing chambers. The carbon retains the solvent, while allowing the stripped air to be exhausted to atmosphere. The adsorption is carried on until such time as the carbon shows signs of becoming exhausted. This stage can usually be detected by the smell of the spirit being observed in the exhausted air. At this juncture, the passage of the air is stopped (it is a very desirable arrangement to have two adsorbers, used alternately for adsorption and regeneration) and the solvent recovered from the carbon by passing hot air or, in the case of solvents which do not mix with water, steam through the adsorption chamber. The mixture of solvent vapour with hot air or steam is passed into a

condenser where the vapours are converted into liquids. When the solvent is removed from the carbon by means of hot air, the condensate is pure solvent, but when steam has been used a mixture of solvent and water is, of course, obtained. In the latter case, the liquid is passed through the separator to remove the water. The carbon is dried by passing hot air through the adsorbers and, after cooling, by blowing in cold air, is ready for re-use.

There are two standard processes of recovery by activated carbon, which are connected with two standard types of apparatus.

At a meeting of the Institute of Rubber Chemistry at Manchester in 1932, J. C. Liddle said:

"The successful application of active carbon to all solvent-using industries depends on its remarkable adsorptive capacity for organic solvent vapours at low concentration, and also on its selectivity for organic solvent in preference to water vapour.

"The term 'adsorption' is a word coined specially to describe the particular action of active carbon and similar substances, and to distinguish it from 'absorption', which is a somewhat vague term, used in a wide sense, covering chemical combination and solution formation as well as adsorption.

"Adsorption is believed to be the result of attraction by surface condensation, but the mechanism of its action is somewhat obscure.

"Active carbon contains countless numbers of microscopically fine capillaries, which have a vast area in proportion to the bulk of carbon.

"For example, it is estimated that one cubic centimetre of active carbon may contain pores having a total wall area of about 600 sq. metres. Organic vapours contained in the air in the close vicinity of the carbon permeate into these capillaries and are immediately condensed on the walls, thus reducing the vapour tension. Fresh vapour takes its place and adsorption continues until either all the vapour is extracted, or the carbon is so fully charged that it is in a state of equilibrium with the vapour. The quantity of solvent which carbon will take up in proportion to its weight varies with the concentration; the higher the concentration, the higher the load. It will be seen that adsorptive action is entirely physical and there is no chemical change in either the carbon or the solvent, both of which can be used over and over again without detriment."

This property of being able to undergo physical changes without alteration in chemical constitution is an essential characteristic of solvents used in dry cleaning, and it is interesting to note that this is also emphasised in the case of activated carbon.

The raw material for the manufacture of activated carbon can include such differing substances as cocoa-nut shells, fruit stones, wood, peat, lignite, and coal.

The carbonisation takes place at high temperatures in the absence of air, and the resulting carbons are activated by means of steam or certain chemicals, such as zinc chloride or phosphoric acid. The properties of the activated carbon are determined by the method of manufacture, so that the ultimate purpose for which the carbon is intended is the deciding factor in the selection of the manufacturing process.

- J. C. Liddle, in the paper already referred to, gives the following qualities of a good carbon for solvent recovery and gas treatment:
- (1) The carbon grains should be reasonably large and regular in shape with smooth surfaces. It has been found in practice that spherically or cylindrically shaped granules of about  $\frac{3}{16}$  in. to  $\frac{1}{4}$  in. diameter are the most suitable. Sharp edges, rough surfaces, and high points offer resistance to the passage of the gas. Projections rub down and cause fines, due to the slight but unavoidable movements of expansion and contraction caused by the changes in temperature. The uneven formation of fines and channels, which occur when unformed grains are used, causes unequal loading of the carbon, which is then not used to the best advantage, whereas smooth, regularly shaped grains keep the power consumption to a minimum and have a much longer working life.
- (2) A high adsorptive capacity is necessary, but an optimum point should be achieved where the effect of high adsorptive capacity is not counter-balanced by too high a retentivity. These two characteristics go together. A high residual charge may make regeneration difficult and cause losses to occur during the drying and cooling.
- (3) The reaction of the carbon should be neutral, so that there will not be a tendency to set up corrosion in the vessels.
- (4) Low friability is important, but physical hardness and the capacity of withstanding external shocks are not so important as the capacity to withstand constant rubbing, and also to resist the internal

strains which are set up whilst the solvent is being driven out. This calls for a certain degree of toughness or elasticity.

# THE CARBO-UNION PROCESS OF SOLVENT RECOVERY

It will be remembered that the need for defence against gas attacks during the Great War gave rise to extensive development in the application of activated carbon. After the War, the Continental manufacturers of activated carbon formed a combine under the name of the Carbo-Norit-Union, to pool information and experience. Some years afterwards, the British Carbo-Union was formed in England to acquire the rights of the Carbo-Norit-Union and to exploit its processes in this country. The new company thus had the advantage of the experience of the parent company on the Continent.

The standard type of plant designed for the recovery of solvent in dry cleaning consists essentially of twin vessels containing the activated carbon, a suction pump, and a condenser. Every point at which the dry cleaning solvent may escape is fitted with a large canopy or collecting hoods. By means of the suction pump the solvent-laden air is aspirated into the collecting hoods and pumped through the activated carbon in one of the adsorbers. It will be seen that hoods should be fitted to every appliance used in dry cleaning with the open type of plant; for example, the washing machines, hydro-extractors, dryingrooms or tumblers, and brushing slabs or spotting tables.

When totally-enclosed machines are employed, as is common with the large production non-flam plants, the recovery of the solvent by means of activated carbon can, nevertheless, be practised by treating the air from the deodorising process. The carbon retains the solvent vapours while the stripped gas or air is passed off to the atmosphere.

The solvent vapours are pumped through the adsorbers until the carbon is fully charged, as shown by the presence of vapour in the exhaust air. When the carbon in the one adsorber is exhausted the stream of vapour-laden air is diverted through the second adsorber. Thus, the two adsorbers are in use alternately.

After the switching over of the vapour stream the solvent charged adsorber is regenerated and the solvent contained within it is recovered. Steam is passed through the loaded carbon. The steam has a two-fold

action, that of producing a heating effect combined with a rinsing action, which easily sets free the solvent vapours. The released vapours are passed through a condenser as in distillation. From the condenser the distillate passes into a separator where the solvent is recovered and returned to the storage tanks ready for re-use.

When the flow of solvent from the condenser ceases, the next process in the regeneration of the carbon is that of drying. This is brought about by blowing hot air through the adsorber until the carbon is completely dry. The final process of regeneration is merely that of cooling the carbon by passing cold air through it. After the cooling down process, the carbon is again in an active condition and ready to adsorb a new charge. Thus, the activated carbon exhibits a parallel to the dry cleaning solvent itself, in that it passes through the phases of steaming out, drying, and cooling without deterioration.

The consumption of the carbon is slight, and it has been proved to be very stable over a number of years.

The British-Carbo Union make plants for the recovery of all the solvents used in dry cleaning, whether of the petroleum or the newer non-flam type.

#### ACTICARBONE RECOVERY

The advance in the technique of solvent recovery, which has been particularly rapid since the War, is well demonstrated by the Acticarbone recovery installation. The plant is an outcome of the attention and research which war conditions focused upon the properties and applications of activated carbon.

The Acticarbone installation consists essentially of an appliance to collect the vapours by aspirating the vapour-laden air into specially constructed ducts, twin absorbers, and a distillation and condensing plant. Additional parts of the system are a filtration battery for preventing dust in the atmosphere from being drawn forward into the absorber and special fans for aspiration and propulsion of the vapours, working on the turbine principle.

The working of the plant entails the sucking of the solvent vapours from cleaning machines, rooms, brushing slabs, and drying appliances into the ducts by means of the aspirating fan, and through the Acticarbone in one of the absorbers. This continues until the exhausted

air is shown to contain vapour. When the one absorber is saturated with vapour, the second one is brought into action. The vapour in the first absorber is then recovered by distillation by means of steam, and the mixture of steam and vapour is passed through a condenser where separation is brought about by decanting the liquid solvent from the surface of the condensed water. This operation, i.e. heating, also regenerates the carbon, which, after drying out, is ready to absorb more vapour, the process of adsorbtion and regeneration alternating almost ad infinitum.

The following is a specification of an Acticarbone recovery installation for a dry cleaning works:

#### OPERATION AND DESCRIPTION OF THE INSTALLATION

(1) Collection.—The vapours of the spirit given off from the different points in the work-rooms, as follows:

2 large washers 1 brushing table

1 small washer 1 drying-room with air circulation

2 hydro-extractors I drying tumbler

—will be collected by means of the fans of the Acticarbone plant, which will aspirate these vapours through a special filtration battery, placed in a brickwork chamber. This battery will operate in three stages to eliminate all dust drawn forward in the air. The pipe-work for collection, connecting the installation proper to the points of collection, should be in light sheet metal.

(2) Recovery.—The installation will comprise essentially:

Two absorbers of Acticarbone, special type, with horizontal grids and fans of aspiration and propulsion.

One system of condensation and decantation.

The operation of the installation is completely continuous, with the distillation of an absorber every two or three hours.

Dimensions.—The following is the approximate space required for the installation itself: Length, 20 ft.; width, 11 ft.; height, 13 ft.

In addition, provision must be made for the filtration battery which will have the approximate following dimensions: 6 ft. by 7 ft. 6 ins. in plan and 10 ft. high.

#### SPECIFICATION OF THE ACTICARBONE SUPPLY

- (1) One filtration battery in three stages, comprising separation by shock, by washing, and by S.C.A.M. filters. This battery will be placed in a brickwork chamber.
- (2) Two absorbers of Acticarbone patented type with horizontal grids with thermic elements for heating and cooling.
- (3) Two special fans for aspiration and propulsion, turbine in aluminium bronze driven by pulleys.
- (4) One condenser.
- (5) One decanter separator.
- (6) The apparatus for control and measurements.
- (7) The pipe-work, including valves, taps, purgers, etc., necessary for the operation of the installation and contained within its limits.
- (8) The quantity of Acticarbone A quality for the recovery of spirit, necessary for the charging of the absorbers.

The conditions of operation for the installation being as follows: Actual quantity of spirit lost in the department, 12,000 gall. per annum, collection being carried out according to Acticarbone plans, and the goods being dry on exit from the driers—Acticarbone guarantee with the proposed installation: (1) A total commercial recovery of 55% of the spirit consumed in the dry cleaning department; (2) consumption of activated carbon 0.8 kilos per ton of spirit recovered.

The plant to which this specification referred was a particularly difficult one, because of its layout, and the problem was really one of how to collect the vapour-laden air efficiently. The details given are interesting, especially the figure for the consumption of the activated carbon. The actual working costs of this plant are in the region of 3d. per gall. of solvent recovered. The Acticarbone plant finds application in all processes where volatile solvents are employed, and in certain cases has shown a recovery efficiency of 80% of the solvent.

### ALFA-LAVAL "WATERWHITE" RECOVERY

The use of activated carbon for the recovery by adsorption of solvent vapours has already been discussed, but activated carbon also has the property of being able to adsorb colouring matter from liquid solutions, a characteristic that has been turned to account in the refining of molasses, and has more recently been made use of in the dry cleaning industry in connexion with the purification of dirty solvent in its liquid state.

The process is a combination of the settling method (already described) and filtration or agitation with activated carbon. It will be remembered that the basis of recovery by settling depends on the dehydration of the spirit, and that such chemicals as caustic soda solutions, sulphuric acid, and sodium carbonate find employment as dehydrating agents. By combining the settling action of caustic soda with the decolorising action of activated carbon, an excellent recovered solvent can be obtained.

The Alfa-Laval Co. has incorporated this system of the combined treatment of solvents with caustic soda and activated carbon with its system of centrifugal recovery, and markets an apparatus under the name of the De Laval "Waterwhite" system. This process has the great advantage of being continuous, and the solvent can therefore be kept in circulation during the whole of the cleaning operation.

The "Waterwhite" system makes use of the De Laval centrifuge in order to remove the dirt, carbon, and the watery solution.

The "Waterwhite" plant consists of a specially constructed treatment tank joined up in circuit with a De Laval centrifuge and by circulating pumps with the cleaning machine—or machines, for the circuit may be extended to any number of machines. During the operation of the system the soiled spirit is drawn from the washers and discharged into the "Waterwhite" tank. Here the solvent is treated with a measured amount of caustic soda in solution and a fixed amount of activated carbon. The caustic soda causes the coagulation of the dirt and the saponification of the fatty and greasy compounds dissolved by the spirit, while the carbon removes the colouring matters. The spirit is then washed by a stream of water and the mixed liquids passed into the centrifuge for separation. In the "Waterwhite" system the decolorised solvent is obtained in a condition equal to that of new and unused solvent. By returning the clarified spirit to the machine a continual flow of absolutely clean solvent is maintained in contact with the goods being treated.

It is claimed that the "Waterwhite" system gives better cleaning, and that the solvent obtained is in better condition than when

redistilled. Obviously, any danger of the constitution of the spirit being altered by "cracking" during distillation is avoided.

The makers of the apparatus set out the following list of advantages: More economical to operate than any other system of dry cleaning; capital cost of installation is less than for any other equivalent system; no still needed; a continuous stream of "Waterwhite" deodorised spirit is produced, thus providing better and more hygienic cleaning; De Laval "Waterwhite" plant has a large output compared with others; it effects a saving in soap; it can be coupled up to one, two, or three washers; it shortens the washing period; it does not require a skilled operator; the plant is practically automatic in operation; it is not necessary to pre-dry the goods; it reduces the time taken in sorting the goods prior to cleaning; it reduces spirit storage; no steam or heat required; reduces spotting time.

The "Waterwhite" plant is manufactured in five sizes, ranging from a capacity of 100 gallons of spirit treated per hour to one of 1,250 gallons per hour.

#### SPOTTING

The immersion of garments and other articles in dry cleaning solvents only removes soiling matters which are soluble themselves, or which are stuck to the textile material by a compound, which is soluble in the particular solvent. The treatment with solvents is merely "de-greasing". Staining matters, which are soluble in water, are not usually removed in dry cleaning. Other stains, such as those caused by paint or tar, require more powerful solvents than the usual dry cleaning liquids.

The removal of stains that remain after dry cleaning is a highly skilled and costly process. "Spotting" has been rightly described as an art. It calls for long experience and involves a peculiar attitude and an infinity of patience.

The vital feature of the process is that no signs of the effort must be apparent whether the stain is removed entirely or not. In the case of silk articles, no alteration in lustre or finish must show.

Obviously, a white patch due to difference in the reflection of light from the surface of the silk would be worse than the original stain.

The successful handling of delicate fabrics can only be acquired after long training. Because of their finer sense of touch, girls usually make the best spotters.

As with many other processes in dyeing and cleaning, the operator in the spotting process has no knowledge of the cause of the stain, but must rely on experience in deducting the probable cause. When the stain cannot be identified, a routine procedure should be followed, so that by a process of elimination, the exact nature of the stain is discovered and the necessary treatment applied.

The following general rules for spotting are taken from the booklet, Dry Cleaning with "Triklone", issued by Imperial Chemical Industries, Ltd.:

#### GENERAL RULES FOR SPOTTING

- (1) Spotting should be done in a well-lighted room, preferably with north lighting.
- (2) The spotting table should be near a window so that vapours from any of the solvents used may be carried away from the worker. If necessary, a fan should be installed to assist the ventilation.
- (3) A clean cloth or absorbent pad should be placed on the table underneath the stained part of the fabric and, after most of the stain is removed, it should be readjusted to bring a fresh, clean part underneath the stain. Alternatively, the spotting bench should have a glass or tiled top. The articles to be spotted are spread out on this and the stain is padded with a chamois leather which has been moistened with the spotting reagent. The chamois leather is rinsed out with distilled water each time the spotting reagent is changed.
- (4) The mildest reagents should be used first. As a rule the order should be: (a) water, cold or hot; (b) organic solvents; (c) inorganic solvents, dilute solutions of acids or alkalies; (d) bleaching (oxidising or reducing) agents. Several applications of one mild reagent will often remove the stain with less damage than would be caused by one application of a more dangerous liquid.
- (5) All chemical reagents should be washed out thoroughly before the article is dried.
- (6) It should be remembered that alkaline solutions are dangerous to animal fibres such as wool and natural silk, but relatively harmless to vegetable fibres (cotton, linen, etc.) and rayons.

On the other hand, acid solutions are dangerous to the vegetable fibres and rayons, but may be used for wool and silk.

(7) Always test for acetate rayon in the article if there is any possibility that this fibre may be present. If it is present acetone must not be used.

#### MATERIALS REQUIRED

- (1) Glass rods, soft and stiff brushes, chamois leathers, reagent bottles, wash bottles, small pipettes of the fountain-pen-filler type, and small hand-bowls or beakers.
  - (2) Chemical reagents.
    - (a) Distilled water.
  - (b) Organic solvents: acetone, alcohol (rectified or methylated spirit), benzol, trichlorethylene, and others as required for special purposes.
    - (c) Acid solutions:

Acetic: 1 oz. of commercial acid in  $\frac{1}{2}$  pint of water. Use for 2 to 5 minutes cold.

Oleic: As purchased.

Oxalic: 1 oz. to 1 pint of water. Use warm (below 100°F.) for 5 to 15 minutes.

Note.—Oxalic acid is poisonous, and the fabrics and hands must be washed thoroughly after using it.

Hydrochloric: 1% solution (approximately 1 oz. of  $28^{\circ}$  Tw. acid to  $1\frac{1}{3}$  pints of water).

Potassium hydrogen fluoride: Use cold for 5 to 10 minutes as directed in chart.

(d) Alkalies:

Sodium carbonate: 5% solution (1 oz. to 1 pint of water). Use cold for 5 minutes.

Trisodium phosphate detergent: 1 oz. to 2½ galls. of water. Use cold or at 140°F. for 15 minutes.

Ammonia: 25% solution.

Note.—Special care should be taken in using ammonia with coloured goods, because many dyes are wholly or partly stripped by it. This applies chiefly to basic (wool) dyes of blue, violet, or green shades.

# (e) Oxidising agents:

Hypochlorite: 2 ozs. of 15% sodium hypochlorite liquor to 3 pints of water. Use cold (never warm) for 5 minutes.

Note.—Hypochlorite must never be used on wool or natural silk, because it has a pronounced tendering action on these fibres.

Sodium perborate: 1 oz. in 4 pints of warm water. Use only a fresh solution of 140°F. for 15 to 30 minutes.

Potassium permanganate: 1 oz. to 4 galls. of water. Use cold or luke-warm for 15 minutes.

Hydrogen peroxide: 10 volume. Use cold for a few minutes only.

# (f) Reducing agents:

Sodium hydrosulphite: I oz. of sodium hydrosulphite, "Hydros", "Oblit", or "Laundros" to I pint of water. Use immediately, either cold or warm.

Note.—Hydrosulphite is a very powerful stripping agent for many dyes, and should never be used on coloured articles unless the dyes have first been tested.

# (g) Soaps:

Use a good laundry quality.

As a solvent soap, use "Westropol" or "Westoran".

#### PROCEDURE

The type of stain should be identified as far as possible by examination, and the first reagent chosen accordingly. For example, it is useless to employ water on a grease or tar mark, whilst an organic solvent has no effect on iron-mould. Where identification is impossible, water should always be used first.

Knowledge of the correct procedure can be gained only by experience, and until the spotter is sufficiently trained, tests should always be made first on an inconspicuous part of the article to find the effect of the reagent on the colours or fabric.

# MODERN DYEING AND CLEANING PRACTICE SPOTTING CHART. White Goods

Stain	Cotton, Linen, Rayon	Wool and Natural Silk
ALBUMEN— EGG STAIN, DISCHARGE, BLOOD	hot) water and soap (hot liquids then, if necessary, blo	
BEER, WINE, FRUIT, TEA, COFFEE, COCOA, CHOCOLATE	Wash with warm water and soap. For more resistant stains use trisodium phosphate and, if necessary, bleach with hypochlorite  Wash as for Cotton but if bleaching is necessary use hydrogen pero ide, perborate, or perma ganate followed by oxaliacid	
CREAM, MILK	Wash with lukewarm water and soap. If grease remains use trichlorethylene, "Westropol" or "Westoran"	
Dyes	The removal of dyes generally demands special knowledge because different treatments are necessary according to the class to which the dye belongs. For the complete stripping of garments or dyed articles reference should be made to dyeing handbooks or textile reference books.  For small stains the following should be tried in order:  (1) Wash with warm water and soap  (2) Alcohol or methylated spirit  (3) Hypochlorite solution made slightly acid with acetic acid  (4) Hydrosulphite solution (warm or hot), preferably made slightly alkaline with soda ash or caustic soda solution  (a) Wash with warm water and soap; or  (b) Rub oleic acid into the stain, then treat with ammonia and again rub well. Rinse off thoroughly with water. Then treat with rectified spirit	

Note. (a), (b), (c), etc., represent alternative treatments

# MODERN DYEING AND CLEANING PRACTICE SPOTTING CHART, WHITE GOODS—continued

Stain	Cotton, Linen, Rayon	Wool and Natural Silk
GREASE, OIL AND WAX	(a) Spot with trichlorethylene or "Westoran" (b) Rub in "Westropol" and wash off with warm water (c) Rub in oleic acid, then ammonia, and wash off If metallic dust remains after removal of the grease, treat as under Iron Note.—In spotting with trichlorethylene or any other organic solvent a ring should first be made round the stain with clean solvent and the stain should then be rubbed or padded gently with a soft cloth moistened with the solvent. Rubbing should be done from the outside towards the centre to avoid spreading the stain. After the solvent treatment, dab the fabric with a dry cloth, especially round the edges of the wet patch, to prevent the formation of "sweal marks"	As for Cotton
Gravy and Meat Juice	Sponge with warm water and soap, then treat as for Grease	As for Cotton
GLUE AND GELATINE	Sponge with hot water	As for Cotton
Inks: Writing	(1) Pad with oleic acid and ammonia as for Grease, wash, and (2) Treat with oxalic acid to remove iron (3) Treat with hypochlorite solution, and then (4) Wash immediately with cold water. If a slight brown stain remains, treat with hydrogalable in	(1) Pad with oleic acid and ammonia  (2) Treat with oxalic acid (3) Treat with hydrosulphite. If necessary, repeat (2) and (3) and finally if necessary, bleach with peroxide or perborate.

Note. (a), (b), (c), etc., represent alternative treatments

ate

sulphite

# MODERN DYEING AND CLEANING PRACTICE SPOTTING CHART. WHITE GOODS—continued

Stain	Cotton, Linen, Rayon	Wool and Natural Silk
Marking	Silver marking inks. Treat with ordinary tincture of iodine, then with a solution of sodium thiosulphate (ordinary photographers' "hypo")  For other types of marking ink, treat with "Westropol" and then bleach with hypochlorite	As for Cotton
Copying	Treat with rectified or methy- lated spirit or with oleic acid and ammonia. If necessary, bleach with hypochlorite	Treat with rectified spirit and, if necessary, bleach with peroxide
Printers'	Treat as for Grease and bleach with hypochlorite	Treat as for Grease and bleach with peroxide
Iron	(a) Treat with oxalic acid or, better, with a solution containing:  (b) 1 oz. of oxalic acid and 1 oz. of acid potassium fluoride in 1 quart of water  Pad until the stain disappears, then rinse with water and finally with dilute ammonia  (c) Treat with hydrochloric acid  Do not use any oxidising agent	As for Cotton  As for Cotton
Iodine	Treat with sodium thiosul- phate (photographers' "hypo") or with alcohol and ammonia	As for Cotton
Lipstick	(a) First remove the grease with trichlorethylene, "Westropol" or "Westoran", then remove the basic dye with alcohol and, if necessary, bleach with hypochlorite or hydrosulphite  (b) Alternatively spot with butyl acetate and bleach if necessary	As for Cotton except that bleaching should be done with hydrosulphite or peroxide, not with hypochlorite

NOTE. (a), (b), (c), etc., represent alternative treatments [ 161 ]

# MODERN DYEING AND CLEANING PRACTICE SPOTTING CHART. WHITE GOODS—continued

Mustard	Wash with warm water and soap	As for Cotton
	30mp	
Mildew	Wash well with warm water and soap, then bleach with:  (a) Hypochlorite or (b) Permanganate and oxalic acid; (c) Peroxide Care should be taken in usin forms of mildew accelerate its addering	Wash, then bleach with (a) Permanganate, fol- lowed by oxalic acid, or (b) Peroxide g peroxide because some ction and may cause ten-
Paint	(a) Use trichlorethylene or a mixture of equal parts of acetone and amyl acetate. If necessary (b) Use trichlorethylene and acetone alternately (c) Rub in "Westropol" and then wash off with warm water (d) Equal parts of oleic acid and trichlorethylene  Note.—Do not use acetone or amyl acetate with fabrics composed of or containing acetate rayon	As for Cotton
Perspiration	Wash with warm water and soap and bleach with perborate or permanganate, followed by oxalic acid	As for Cotton
SHOE POLISH	Wash with warm water and soap. Spot with trichlorethylene or "Westropol" or with a mixture of amyl acetate and acetone, or with alcohol If necessary, bleach with hypochlorite	As for Cotton but, if necessary, bleach with perborate or peroxide

Note. (a), (b), (c), etc., represent alternative treatments
[ 162 ]

# MODERN DYEING AND CLEANING PRACTICE SPOTTING CHART. WHITE GOODS—continued

Stain	Cotton, Linen, Rayon	Wool and Natural Silk
SCENT	Use warm water and soap, and then if necessary ammonia and oxalic acid alternately Note.—Do not use organic solvents to remove scent stains from dyed acetate rayon fabrics. If this is done, some of the dye may be removed and a light spot will result	As for Cotton
Scorch Marks	Wash well with warm water and soap. Little further can be done without damage to the fabric. Bleaching with peroxide or hypochlorite is sometimes of advantage	As for Cotton. Bleach with peroxide, not hypo- chlorite
Tar or Pitch	Spot with trichlorethylene or rub in "Westropol" or "Wes- toran" and wash off with warm water	As for Cotton
VARNISH	Treat as for paint Shellac varnish is removed easily with alcohol or methy- lated spirit	As for Cotton
Unidentified Stains	Use reagents as follows: (1) Water and soap (2) Organic solvents, or "Westropol" (3) Ammonia (4) Acid, or oxalic acid- potassium fluoride mixture (5) Hydrosulphite (6) Ordinary hypochlorite or peroxide bleaches (7) Hypochlorite solution made just acid with acetic acid	As for Cotton, but avoiding the use of hypochlorite

Note. (a), (b), (c), etc., represent alternative treatments (See also notes on next page).

#### COLOURED GOODS

The treatments for coloured goods are the same as for all-white goods, but the reagents should first be tried separately to see if they affect the colours.

If they do there are two courses open:

(a) To leave the stain as it is;

(b) To give the treatment required and then either tint back the faded part with suitable coloured crayons or dye solutions, or to re-dye the whole garment.

Generally it is better to leave the stain alone, otherwise the final effect may be worse than the original stain itself.

#### RAYON AND MIXED GOODS

The only material requiring special care is cellulose acetate rayon. This rayon is easily identified because it is the only textile fibre that dissolves in acetone.

It is distinguished also from the other types of rayon, and from the vegetable fibres cotton or linen, by the fact that when a flame is held near to it, it melts and forms a hard black bead. Wool and silk also form black beads on burning, but these are soft and give off a peculiar smell similar to that of burnt feathers.

As in the actual dry-cleaning, so in spotting, any goods containing acetate rayon yarns should be treated as though they were composed wholly of acetate rayon. Similarly mixed goods containing wool or natural silk should be treated as though they were composed wholly of these fibres.

Thus it is important to ensure that only solvents without action on acetate rayon are used in spotting mixed goods containing this material. Hot alkaline solutions cannot be used, otherwise the acetate rayon will be seriously affected. In the same way, neither hypochlorite bleaching agents nor hot alkaline liquors may be used for mixed goods containing wool or natural silk.

In all cases, the treatments given should be those suitable for the more sensitive fibres or yarns present.

#### SANITONE PROCESS

Since spotting is both costly and relatively a slow process, many attempts have been made to remove water soluble stains by the controlled introduction of water into the dry cleaning solvents. A great deal of research has been undertaken with the object of discovering suitable reagents to act as carriers of water, so that no separation of water and solvent occurs. C. L. Bird, in his investigations into the function of soap in dry cleaning, reaches the conclusion that soap acts as a water carrier. The Sanitone process of cleaning is one in which water is carefully introduced into the solvent in the later stages. The water carrier is a substance sold under the trade-name of Sanitone.

Sanitone is a dark brown oily liquid. It absorbs a high proportion of water gradually becoming thicker until it becomes a thin jelly. The jelly is miscible in white spirit and forms a stable emulsion.

The moisture present in the cleaning bath when Sanitone is present does not behave like pure water and does not bring about the troubles, including shrinkage, wrinkling, colour running, and size removal, which are associated with a water treatment.

The quantities of moisture which give the best results with various types of fabrics have been accurately ascertained, the moisture is therefore always under definite control. Garments cleaned by the Sanitone process are free from odour and have a new appearance, since both the water soluble and greasy soiling matters have been removed, the colours are brightened, and the finishing materials smoothed and levelled.

The operations are based on the following principles:

- Amount of moisture added is calculated on weight of material cleaned.
- 2. One gallon of Sanitone will absorb nearly 18 pints of water (26.7 fl. oz.).
- 3. Sufficient Sanitone must be present when cleaning each load to absorb the amount of water added.
- 4. The Sanitone solution must be periodically renewed, or the oil and grease which accumulate in the Sanitone may cause odours and interfere with proper cleaning.

#### SILKS AND SANITONE PROCESS

These materials must be given a preliminary break in solvent alone. During the Sanitoning operation continuous filtration is essential and rapid circulation desirable. When water is being absorbed by the Sanitone the solution is circulated by the pump and the filter cut out by a by-pass. After the Sanitone treatment the silks must be rinsed. It is recommended to break and rinse in one washer and to "Sanitone" in another, transferring the loads. If this is not possible, a small tank must be provided, to hold the Sanitone solution during the break and rinsing operations.

# PROCEDURE (AS ISSUED BY MAKERS OF SANITONE PLANT).

- (1) Sort white and pale colours separate from blues and reds and blacks. This is because some blues and blacks topped with basic colours tend to bleed into the solvent and the colour may be taken up by some of the de-lustred art silks; certain reds also tend to bleed. This trouble can be avoided by the use of E170 powder and carbon (see below).
  - (2) Weigh and pre-dry.
- (3) Break 10 to 15 minutes; rinse from the previous load may be used.
- (4) Sanitone operation.—A table shows the amount of Sanitone which may be present in the washer, based on the maximum weight of material cleaned in one load.

### Examples are:

Load, Silk	Gallons, Sanitone
10 lbs.	I
40 —	$2\frac{1}{3}$
70	$3\frac{1}{3}$

The Sanitone solution is prepared in the washer, using such a quantity that it corresponds to the figure in the table, e.g., if for 40 lbs. loads 50 gallons are required in the washer, and 100 gallons in the entire system, the amount of Sanitone must be 4\frac{2}{3} gallons. Water is then added directly to the washer, the proportions being given in a further table:

10 lbs. silk require 21 fluid ozs. water (just over 1 pint).

40 lbs. silk require 57 fluid ozs. water.

After the water is added to the solvent, it is circulated for 3 to 5 minutes. In cold weather it should be warmed to 70°F. Absorption is complete when the solvent appears clear in the sight glass. The load is then introduced, together with filter aid and E170 powder, and solvent circulated for 1 or 2 minutes without the filter, and then 20 minutes with the filter in circuit. The load is then removed, extracted if possible, and rinsed 10 minutes in clean solvent.

(5) Drying.—In case tumblers are used, commence with cold blast for 5 minutes, follow with hot blast, not above 120°F., until dry, and finish with a short cold blast; this prevents wrinkling.

- (6) Maintaining the Solution.—Sanitone must be added with each load, the amount depending on whether the extracted solvent is returned to the washer or not. Calculations are given for both cases. Spent Sanitone solution may be used for woollens, as the effect of dirty Sanitone is not noticeable in wool.
- (7) General Notes.—A small addition of E170 powder is made when cleaning blues and blacks. The bleeding of red shades which stain cotton is overcome by the addition of a little activated carbon.

#### WOOLLENS AND SANITONE PROCESS

No break is required in the case of woollens. In order to avoid using the very strong Sanitone solution, which would be required to carry the necessary amount of water if the methods of working were the same as for silks, it has been found advisable to use a 1% Sanitone solution for all woollens, and add the moisture gradually to this solution as it is circulated through the filter. This is effected by means of a special humidifier, furnished with a water injector. The washer is first run with Sanitone solution for about 10 to 15 minutes; the water is then introduced during the next 15 minutes, and the run is continued for 5 to 10 minutes to filter out the dirt loosened by the Sanitone.

#### HATS AND SANITONE PROCESS

Hats are first immersed in clear solvent, then placed on a block and brushed vigorously with a solution prepared by mixing five parts of Sanitone and one part of hot water, and dissolving this mixture in six parts of solvent. They are afterwards rinsed in the washer in clear solvent and finished as usual. Details for cleaning of gloves and suede jackets may be obtained from the makers of the Sanitone plant.

# Chapter III

## WATER

THE quality of the water used for wet cleaning, laundering, scouring, bleaching and dyeing of textiles is of supreme importance. Faulty work and damaged goods in many cases can be traced to the use of unsuitable water. It is peculiar that water is rarely suspect because it may look bright and clear.

Absolutely pure water never occurs in nature, and although distilled water may be taken as pure for practical purposes, it is rarely entirely free from traces of impurities.

For technical purposes, the sources of water may be classified under three groups: (1) rain water; (2) spring and well water; (3) river and surface water. Rain water collected under suitable conditions is the purest form of natural water, but its character depends largely upon the district. That which is collected in the country is purer than that collected in the town. The rain as it falls removes many impurities from the atmosphere; thus, in the towns, rain water may contain relatively large quantities of sulphuric acid derived from the burning of sulphur in coal. A slate roof is the best for the collection of rain water. In hard water districts, when no water softening apparatus is available, the collection and storage of rain water for scouring operations is advantageous.

The remarkable solvent properties of rain water, particularly for such atmospheric gases as carbon dioxide and sulphur dioxide, accounts for much of the hard water occurring in springs and wells.

Considerably more than third of the rainfall soaks into the ground and percolates underground. During this process, the dissolved gases impart to the water the property of dissolving certain minerals. This is particularly the case with limestone (calcium) or magnesium rocks. The calcium and magnesium carbonates (the chemical constituents of these minerals) are hardly soluble in pure water, but are soluble to an appreciable extent in water containing carbon dioxide.

The deeper the rain water percolates into the earth the greater its solvent action becomes, owing to the increase in pressure. In certain cases, the water passes through a depth of permeable layers of the earth until an impervious stratum is reached. Then the water accumulates to form an underground lake. Such an accumulation of water forms a vast (apparently inexhaustible) lake below London. In other cases, the water passes down to great depth until a fissure is reached and pressure forces the water to the surface in the form of a spring. Huge quantities of water for industrial purposes are obtained by boring, sinking wells, and then pumping from the underground accumulations.

Spring and well waters contain considerable quantities of mineral matter, such as carbonates, sulphates, chlorides, nitrates and silicates of calcium, magnesium, sodium, potassium, aluminium and iron.

River water, being fed from springs, contains mineral matters of the spring's water in addition to organic matter from plants and trees growing on the banks or on the bed of the river. Some of the soluble matter in spring water is deposited owing to the loss of the carbon dioxide. River water usually contains considerable quantities of suspended matter. For technical purposes, the matter in solution is of much more importance than that in suspension, since the latter may be removed by filtration.

A few simple tests will give some indication of the constituents in any sample of water.

The presence of calcium salts (commonly called lime salts) is indicated by the formation of a fine white precipitate on the addition of ammonium oxalate with ammonium chloride to the sample.

Following the test for calcium, the sample is boiled and filtered, and then evaporated. On cooling a small quantity of ammonia is added, and then a solution of sodium phosphate. A white crystalline precipitate would indicate the presence of magnesium.

An extremely delicate test for magnesium compounds in water depends on the formation of a blue compound, that magnesium forms with certain yellow dyestuffs of the aminoazo-benzene series. This test will detect magnesium to  $\frac{1}{2}$  part per 100,000.

Sulphates, if present, will give a white precipitate after the addition of a solution of barium chloride and hydrochloric acid.

Bicarbonates and free carbon dioxide are indicated when a white precipitate is obtained by the addition of clear lime water.

Chlorides give a white curdy precipitate on the addition of nitric acid and silver nitrate. Iron is almost certain to be the cause of a reddish-brown (rusty) deposit appearing when the water is exposed to air for some time. In the case of water containing considerable amounts of iron, this reaction may be used as a preliminary treatment for its removal. Much of the iron may be removed by exposing the water in large shallow tanks. Extremely sensitive tests for iron depend on the formation of easily distinguishable colours with potassium ferrocyanide or with potassium thiocyanate. The sample of water is evaporated to small bulk, when a few drops of pure hydrochloric acid and pure nitric acid are added. The addition of potassium ferrocyanide will produce a blue coloration or precipitate, and potassium thiocyanate a blood-red coloration, if iron is present.

Alkaline carbonates may be detected by boiling the sample of water for 10 minutes, filtering and testing with red litmus or lackmoid, which would be turned blue.

Acid waters turn a neutral solution of lackmoid red, or blue litmus red. Organic matter in water usually causes a distinct brown coloration, which is intensified by the addition of alum.

The suitability of water for dyeing and cleaning may be considered from three angles:

- (1) As used in wet cleaning:
  - (a) after dry cleaning,
  - (b) previous to dyeing.
- (2) As used in dyeing.
- (3) As used for boilers and steam raising.

The presence of calcium, magnesium, and iron in water for wet cleaning is undesirable. The salts of these metals impart the property of "hardness" to the water. "Hardness" may be described as "soap-destroying power". Soap is a combination of sodium or potassium with a fatty acid such as oleic, palmitic or stearic acid. The combination of calcium, magnesium or iron (in fact, most metals), with these fatty acids produce a sticky, insoluble, curdy precipitate. When soap is added to hard water, an interchange between the calcium, etc., with the sodium of the soap takes place and the insoluble calcium soap is

precipitated as a scum. This precipitation proceeds until all the calcium has been removed, after which further addition of soap will produce the usual soapy lather. In this way, a considerable quantity of soap may be "killed" before a lather is produced.

The hardness of water is expressed in degrees, and although the meaning may vary according to the comparative scale employed, it is common in England to imply English or Clarke's scale. For practical purposes, this may be taken to express the degrees of hardness as representing the number of grains of calcium carbonate per gallon. In technical and scientific circles, it is customary to express hardness in "parts of calcium carbonate per 100,000". One part calcium carbonate per 100,000 is equivalent to 0.7 grains of calcium carbonate per gallon, that is, 0.7° hardness. Whereas the hardness of water is expressed in terms of calcium carbonate, it does not mean that calcium carbonate is actually present in the water. Calcium carbonate is almost insoluble in water, but the constituents causing hardness can conveniently be expressed in equivalent amounts of calcium carbonate.

The property of "killing" soap is employed in the determination of hardness in water. This can be carried out quickly and requires very little apparatus or manipulative skill. A standard soap solution is made up so that I c.c. of solution is equivalent to I° hardness. If such a solution is run from a Burette into 70 c.cs. of water in a small bottle, drop by drop, until a permanent lather is formed by shaking, the exact amount of soap "killed" by the water is obtained. Since a certain amount of soap must go to form the lather, it is usual to compensate for this by deducting I c.c. from the amount of soap used. Thus, if 5 c.cs. of soap solution are "killed", the hardness is recorded as 4°. It must not be claimed that this test is scientifically accurate, but rather the opposite, and in certain circumstances, may be erratic. It serves, however, as a guide for most practical purposes.

During the test, it may be observed that the lather appears and then on the addition of more soap solution, disappears. This is due to the fact that the formation of the calcium soap is quicker than that of the magnesium soap. This delayed action may serve as an indication not only of the presence of magnesium but also of its amount.

The loss of soap due to hard water is not the only disadvantage. The sticky, insoluble calcium and magnesium soaps, are liable to be

formed unevenly on textiles and thus be the cause of uneven dyeing, especially with mordant colours.

The presence of iron in water for mordanting, dyeing and rinsing after dyeing is very undesirable. Traces of iron would sadden or dull the shades of certain dyestuffs, particularly those of the Coomassie navy blues, and certain of the mordant dyestuffs. The carbonates of calcium and magnesium and even of soda may have similar effects, but to a much lesser degree.

Too much attention cannot be paid to the character of water as used by the dyer and cleaner. Many expedients are tried in order to overcome disadvantages due to hardness of water. When the hardness is due to presence of carbonates, correction for dyeing may be obtained by the addition of acetic acid, which produces harmless acetates. Excess of acetic acid must be avoided, particularly when dyeing mixture fabrics with direct cotton or Union dyestuffs. In this case, the wool would be fully dyed, while the cotton would be left undyed.

Steam is an essential in every dyeing and cleaning plant, and consequently the character of water for steam-raising is highly important. The use of hard water for steam-raising considerably effects the efficiency and therefore the economy of the boiler plant. The continual evaporation of hard water results in the deposition of the calcium and magnesium compounds in the form of a hard scale or "fur". This scale is a poor conductor of heat and consequently entails considerable loss in the heating efficiency of the fuel used. Not only this, but the difference between the rates of contraction and expansion of the scale and the boiler shell may cause distortion of the shell and shell seams. Further, excessive foaming within the boiler may result. Apart from the formation of scale due to hard water, it may be explained that the use of soft water also necessitates strict conditioning in successful boiler control in order to prevent priming and foaming, to prevent attack of fittings and avoid danger of embrittlement of the boiler metal.

The softening of water has the definite object of removing the calcium and magnesium compounds, which are the cause of temporary hardness and permanent hardness. Temporary hardness is caused by the presence of bicarbonates.

Water of temporary hardness may be softened merely by boiling. This causes the bicarbonates to be converted into carbonates, which are insoluble and are precipitated. Permanent hardness is caused

by the presence of soluble compounds of calcium and magnesium, mainly sulphates. These sulphates are to a certain extent soluble in water and are not altered by boiling.

Water is best purified by distillation, but obviously this is not a commercial proposition. Yet in many works, a considerable quantity of condensed water (i.e., distilled water), is available from steam pipes, engine exhausts and from many types of heating apparatus. The collection of this condensed water is carefully controlled in many plants, but it is surprising that the practice is not universal.

There are two commercial methods of chemically treating water in order to make it suitable for industrial purposes. The process known as the "lime-soda" process has been used in the softening of industrial water for a little over a century. At first, the use of lime to remove hardness due to lime compounds confuses the layman. It has been explained that calcium carbonates is only soluble in water containing dissolved carbon dioxide. The function of the lime is to neutralise this carbon dioxide, so that the whole of the calcium carbonate is precipitated and is removed by filtration. It is important to note that the amount of lime added to the water must be just the correct amount, any excess would of itself cause hardness. The lime of the "limesoda" process is to remove the temporary hardness, that is, the bicarbonates of calcium or magnesium. The soda is added to convert the sulphates and other soluble salts of calcium or magnesium into insoluble carbonates. These reactions are not the only ones that are considered to take place in the lime-soda process. It is held by many, that a series of complex chemical reactions occur also.

It is clear that in the mixing of lime and soda in preparation for water treatment, reaction takes place between the two ingredients whereby some of the soda is used in converting the lime into caustic soda with the precipitation of calcium carbonate. Until recently, lime in the form of hydrated lime and soda in the form of pure sodium carbonate (58° alkali) were used without any other additions. In these circumstances, it was not possible to reduce the hardness of water below 5°, owing to the difficulty of removing the whole of the calcium and magnesium (particularly magnesium). Nowadays, a third chemical has been introduced. This is sodium aluminate. The addition of sodium aluminate to the lime and soda in the proportion of  $\frac{1}{2}$  lb. to  $\frac{1}{2}$  lb. per 1,000 gallons of water treated, not only permits

the hardness to be reduced to  $1-2^{\circ}$ , but also causes the precipitate to be much more flocculent and therefore easily filtered. Using lime and soda, the time of treatment is important and should not be less than 4 hours, but the addition of aluminate enables the reaction time to be considerably curtailed. Since the lime-soda-aluminate treatment is so effective, it is found that the amount of reaction materials required is so much less, that in spite of the cost of the sodium aluminate, the final cost of the treatment on the basis of cost per 1,000 gallons is less than by the older method. The many types of plant employed for the softening of water by lime-soda-aluminate process conform to the three general features:

- (1) Tanks for mixing the reagents;
- (2) an appliance for measuring the reagent into the water;
- (3) a filter for the removal of the precipitated calcium and magnesium compounds.

The Lassen and Ihort softening plant is typical of lime-sodaaluminate softeners. The mixing tanks are circular, and fitted with mechanical stirrers. The correct quantity of lime and soda must be carefully calculated, as well as the correct amount of water for the mixture. The correct quantities depends upon the amount of hardness and the constituents causing hardness of the untreated water. It is also highly important that the mechanical stirrers be really efficient and kept in constant motion during the mixing. From the mixing tanks, the reagent liquor is delivered to a header tank mounted over a huge tank, in which the water is actually softened. The reagent tank is equipped with an agitator, which prevents accumulation on the bottom of the tank, and the mixture is accurately measured into the water by means of a dumb-bell valve. This valve is attached to a length of shaft and is operated by the untreated water filling and tipping two buckets fixed to the opposite sides of the same length of shaft. The amount of mixture introduced into the water may be altered by adjusting the distance between the shoulders of the dumb-bell. The valve works in a short length of pipe, so that, at the top of its movement, the top shoulder is clear of the top of the pipe, allowing the space between the shoulders to fill up with reagent, while at the lower end of the movement, the lower shoulder is clear of the bottom of the pipe, so that the measured reagent falls into the water.

The water and the measured reagent are thoroughly mixed by falling down a wide tube, which extends almost the full height inside the water softener tank. This mixing tube opens out into a large inverted cone at its lower end. After thorough mixing with the lime-soda-aluminate, the calcium and magnesium salts are precipitated, so that the water becomes "milky".

The removal of the precipitated compounds is obtained by allowing the water to percolate through a layer of wood-wool of about 3 ft. thickness. This filter is usually arranged near the top of the huge tank, which acts as the reaction tank, or the water softener itself. The flow of water gradually fills the tank and the softened, filtered water is drawn away from an overflow outlet. The filter requires very close attention and must be kept clean. It must be kept open, so that the flow of water is not impeded. The life of the filter is limited, so that the wood-wool is entirely renewed every few months. Eventually, an accumulation of precipitate forms a sludge in the bottom of the reaction tank. This sludge is removed regularly by flushing, an operation which takes time and uses large quantities of water.

The second process of water softening is dependent upon the power of certain complex compounds to exchange their sodium content for calcium and magnesium. The reaction of these zeolites as they are called is extremely simple. When hard water is allowed to percolate through a layer composed of zeolite the water is completely softened. Zeolites are insoluble in the water, and contain sodium, aluminium and silica. The zeolite is effective until all the sodium has been exchanged, when it is said to be exhausted. Treatment of the zeolite layer with a solution of ordinary salt causes the zeolite to be regenerated and made capable of softening water again. Subject to certain alternate processes of softening considerations, the and regenerating can be carried on indefinitely. There are Originally zeolite was of zeolite. types from natural sources, but more recently artificial zeolites have been prepared. The artificial zeolites have a greater and quicker reaction power than the natural variety. In some cases, the natural zeolite is employed as the nucleus for the precipitation Zeolites are known by many names, such of the artificial. as permutit and doucil.

This process of softening is known as the base exchange method. The reactions may be illustrated as follows:

(1) Softening. Sodium zeolite +calcium salt = calcium zeolite + sodium salt.

(It will be noted that after softening, the water contains sodium (soluble in water) compounds equivalent to the original calcium or magnesium salts.)

(2) Regeneration.

Calcium zeolite + sodium chloride = sodium zeolite + calcium chloride (soluble in water).

A base exchange softener usually consists of a steel cylinder containing a certain quantity of the zeolite. The softener is equipped with pipes and valves for the inlet and take-away of the water and for flushing of the plant. The different plants adopt varying forms of distributing the untreated water and of collecting the soft water. Attached to the softener are receptacles for the preparation and mixing of the salt solution.

The cycle of operation of base exchange consists of four stages. The process of softening is that of allowing the untreated water to flow through the layer of zeolite. As the cylinder is closed, the water may be pumped through the softener and delivered to the header tank under its original pressure. The second stage follows immediately the zeolite is exhausted. In practice, the complete exhaustion of the zeolite bed is avoided, and the plant allowed to treat only a definite quantity of water. For the second stage, the flow of water through the cylinder is reversed and the water run to a drain. This washes and opens up the bed, so that the water flows freely through the plant. This back flushing should be carried out regularly, if the plant is to be maintained at its full efficiency.

The third stage is that of salting or regeneration. In this, a definite quantity of salt is introduced into the cylinder until the whole of the zeolite bed is saturated with brine. It is desirable that the regeneration should take as long as possible, in order that the exchange process should be complete. Usually, this takes about half an hour.

The final process is with the object of washing the plant free from salt and the soluble calcium chloride. A sufficient quantity of water is allowed to drain from the cylinder, until no taste of salt is detected,

or until a sample of the water shows a good lather with a few drops of soap solution. The plant is then ready for the softening stage again.

The great advantages of base-exchange softening are its simplicity of operation and the fact that complete removal of hardness is obtained.

Whatever system of water softening is employed, it must be emphasised that the softened water has an alkaline reaction, due to the presence of sodium salts.

This effect is very strong with water of temporary hardness when softened by the base-exchange method. The alkalinity of softened water is advantageous in the scouring or laundering processes, but may cause some difficulty during dyeing processes, especially those using the direct dyestuffs. Some correction of this alkalinity may be desirable either by means of acetic acid or by mixing with untreated water so that a water of a few degrees hardness is obtained.

Since the War period, there have been many interesting chemicals introduced with the object of overcoming the disadvantages of hard water during wet cleaning and dyeing. These chemicals are somewhat complex derivatives of the fatty acids. They have been misnamed the sulphonated fatty alcohols. It is more correct to describe them as the sulphonic esters of the fatty acids. These compounds have the property of forming soluble compounds with calcium and magnesium, so that the calcium and magnesium do not "kill" soap. In addition, these compounds are efficient "wetting-out" agents, and so are powerful assistants in penetration even of thick material. Gardinol, sulphonated lorol, igepon, are typical examples of "sulphonated fatty alcohols". Certain of these are not only soluble in acid solution, but exercise detergent action. This property enables articles dyed with fugitive colours to be wet cleaned in an acid solution without risk of the colour running. Thus igepon T. may be employed in an acid bath.

Within the last two or three years, a new water-softening agent has been introduced under the name of Calgon. Calgon is chemically sodium hexa-meta-phosphate. The distinguishing property of calgon is the power of softening water without forming a precipitate. The mechanism of its action is that any calcium or magnesium in the water is exchanged for the sodium in the calgon to form stable soluble compounds. In the case of goods impregnated with lime-soaps due to washing in hard water, calgon will remove the deposit, by dissolving

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the lime and liberating soap. Although calgon is of particular interest to launderers, it may also be used to considerable advantage in the wet cleaning of garments after dry cleaning or as a preparation for dyeing. This is particularly applicable during the treatment of silk goods.

# Chapter IV

# WET CLEANING AND PROCESSES PREPARATORY TO DYEING

Among the great variety of articles sent to cleaning establishments to be treated, there are many which are not suitable for dry cleaning. Also in certain cases, many articles which cannot be successfully dry cleaned followed by spotting must be submitted to wet cleaning. Obviously, as wet cleaning involves another process, the cleaner only resorts to this when other methods are inapplicable or unsuccessful.

Wet cleaning includes many separate processes involving the treatment of the whole article or garment with water. It includes treating the article with water only after dry cleaning, known as water brushing. Sometimes the article is brushed with a weak solution of ammonia. At other times, the treatment involves a thorough cleaning with soap and water, without a previous dry cleaning.

Dry cleaning is unsuitable for such articles, as lace curtains, window blinds, cretonnes, chintz, blankets, white flannels and rubber-proofed raincoats or mackintoshes, and these are passed direct to the wet cleaning section.

The selection of soap for wet cleaning is dependent upon the type of article to be treated, in so far as the type of article determines the temperature at which the process may be satisfactorily undertaken. Thus, white flannels and coloured goods must be treated at low temperature in order to avoid shrinkage and loss of colour, while white cotton articles, such as lace curtains, may be treated in hot liquors. The suitability of soap for high or low temperature wet cleaning depends upon the melting point of the fatty acid, from which it is made. This melting point is usually referred to under the term "titre of soap". High titre soap would be made from fatty acids with melting point about 100°F., while low titre soap would have a titre about 75°F. Tallow soaps such as curd or mottled soap belong to the high titre class. These soaps gelatinise in solution at ordinary temperature

and are extremely difficult to rinse out unless high temperatures are used in rinsing. Because of this, high titre soaps are not generally employed in wet cleaning. On the other hand, the low titre soaps are particularly suitable for wet cleaning. These soaps are usually neutral oil soaps made from many varieties of oleine, and remain in solution at all temperatures.

Of recent years the introduction of many "assistants" to the functions of soap has greatly improved the processes of wet cleaning. These assistants may be divided into two classes. One class forms the so-called spirit soaps. These spirit soaps consist of normal soaps in conjunction with certain solvents. Carbon tetrochloride, trichlore-thylene, cyclohexanol, methylcyclohexanol and xylene find application as solvents in spirit soaps.

The second class include the so-called sulphonated fatty alcohols. These may be used alone or with the addition of a little ordinary soap. As already described in the chapter on "Water", these compounds are effective even in hard water.

The wet cleaning process is largely carried out by hand on a smooth slab or table. A slate or marble slab makes an excellent surface. A rotary washing machine of the usual laundry type is also a part of the wet cleaners' equipment. This machine must run very slowly, and when possible, should be fitted with an interrupter gear so that shrinkage of woollen goods is avoided by reducing movement to a minimum.

The amount of wet cleaning necessary with garments, such as suits, costumes and overcoats, is determined by the locality from which the work is received. In many cases, where it is not possible to obtain a satisfactory result after the normal dry cleaning and spotting a treatment with water alone may have the required effect. Whether the garments are brushed on the slab or "run" in the machine, skilful handling is necessary in order to prevent undue creasing ("Breaks") and alteration or running of colours. The final rinsing processes are very important in wet cleaning of garments. In the case of men's garments, it is customary to add salt to the final rinse to prevent alteration to the colour of linings.

After the rinsing, the garments should be very carefully packed into the hydro-extractor and the extraction time carefully controlled. The drying of the garments is best carried out in drying rooms in which

warm air is rapidly circulated by means of a large fan. The garments are placed on hangers and are pulled into shape as much as possible while still damp.

On certain occasions, the addition of a small amount of a mild alkali to the water as used in "water brushing" may be necessary to produce a satisfactory result. A few drops of ammonia added to the water is often very effective, or small amounts of the penetrants, such as the sulphonated fatty alcohols.

If the article requires further treatment, it is usual to apply a soap cleaning process. This is usually carried out cold with soft water in a machine or by hand. The essential feature of this process is that the soap should be made to penetrate the garment during the actual cleaning process and that the rinsing must be sufficient to remove all the soap.

During the summer, the cleaner receives many white or cream flannel trousers for treatment. It is common practice to "soap clean" these. Similarly, the greater proportion of blankets can only be successfully treated by a wet process.

The processes for white flannels and blankets are similar. The water must be soft. In many plants, condensed water is reserved for the flannels and blankets. The preliminary stage may be termed "the wetting-out" process. For this, the articles are immersed in a warm solution containing a small amount of ammonia, in order to neutralise any acidity. This acidity may arise from a previous cleaning or, in the case of new goods, from the sulphur stoving. Obviously, any acid retained in the goods would react with the soap used in the actual cleaning and cause precipitation of the fatty acid from the soap.

When the articles have been thoroughly wetted out, treatment with soap follows, either in a rotary machine or in special machines, such as the Waterleap machine and the Karntshrink machine. The special machines are designed to reduce the movement of the articles to a minimum. The first soap treatment in the rotary machine should be for five minutes with the soap in sufficient quantity to give a good lather. The articles are then passed through rubber squeezing rollers and given a second soap treatment, and the squeezing repeated. At this stage, flannels should be examined for stains or dirty marks that remain. These marks require individual treatment, but many of them are removed on brushing by hand with soap to which has been added "spirit" soaps.

Flannels are usually given a thorough rinsing in warm water to remove the soap, after which they are immersed in a weak solution of sulphuric acid. The acid treatment counteracts any tendency to "yellowing" and will also reduce many stains. It must be emphasised that all the acid must be removed in order to avoid risk of tendering of cotton linings or stitching, and also to preserve the soft handle of the material as much as possible. The flannels are rinsed in clear water, hydro-extracted and dried.

In rinsing of blankets, rinsing is carried out in a diluted soap solution. The amount of soap used must be carefully controlled, while the soap must be free from smell. The blankets are hydro-extracted and dried in a regulated current of warm air. The retention of a small amount of soap imparts a softness to the blankets.

When bleaching of flannels or blankets is necessary for the removal of stains or discoloration (yellowing) hydrogen peroxide solution is the most suitable agent. Hydrogen peroxide is marketed in various strengths of solution, denoted by a number of volumes, i.e., 10 volumes; 12 volumes; 20 volumes, and even 100 volumes.

The commercial variety of hydrogen peroxide contains a small amount of acid (usually phosphoric acid), since in neutral or alkaline condition it gradually deteriorates. In contact with metals, hydrogen peroxide rapidly loses strength, and causes "blackening" or oxidation of the metal. This blackening effect causes dark stains on textile materials in contact with the metal. Because of this, all metal parts should be removed from articles or covered with white cotton material before the peroxide treatment. The treatment is carried out in a wooden vat without metal screws or bolts, or preferably, in a glazed earthenware tank.

The commercial hydrogen peroxide solution is diluted with sufficient soft water to give a two-volume solution. The acidity of the hydrogen peroxide is corrected by the addition of ammonia or silicate of soda, until the solution is just alkaline. The thoroughly rinsed goods are immersed in the peroxide bath, and in many cases, allowed to remain in the bath overnight. It is important that the goods should be kept below the surface of the liquor, and for this purpose, wood gratings are fixed in the vat. An additional precaution is to cover the whole vat with sheeting to prevent dust and dirt collecting on the liquor.

Bleaching by hydrogen peroxide is employed on flannels, straw hats, after cleaning, and on faded garments before dyeing.

Sodium perobrate is often used as an alternative to hydrogen peroxide. It has the advantage of being a stable powder, can be stored under proper conditions without deterioration, and can be easily handled. Sodium perborate may be considered as a chemical combination of hydrogen peroxide, borax and caustic soda. Its bleaching action is entirely due to the liberation of hydrogen peroxide. In order to prepare bleaching baths of effective strength, about 20 lbs. of sodium perborate are dissolved in 100 gallons water. This solution is then neutralised by the addition of acid, sulphuric, hydrochloric or acetic acids, until blue litmus paper is just turned red. The liberation of hydrogen peroxide is then increased by the addition of a small amount of ammonia. The same precautions with hydrogen peroxide are necessary with sodium perborate.

The yellowing of white woollen articles, especially blankets, is often corrected by sulphur stoving, although this process is not employed to anything like the extent of the pre-war period. Sulphur stoving is carried out by hanging the damp articles in an atmosphere of sulphur dioxide overnight. The sulphur stove is a chamber that can be sealed airtight. The sulphur dioxide is produced by burning sulphur within the chamber. The damp articles absorb the sulphur dioxide, which bleaches the wool and produces a good white. After sulphur stoving the articles may have a harsh feel and exhibit an acid reaction. The acid retained after this operation would cause precipitation of soap in subsequent wet cleaning, unless ammonia or similar alkali is used in the "wetting-out" stage. The affect of stoving is counteracted by the oxygen of the air and in consequence the whiteness obtained is not permanent, as in the case of that obtained with hydrogen peroxide or perborate.

Since dry cleaning solvents dissolve rubber, garments containing this, either as an adhesive or as a water-proofing, can only be treated by a wet process. The need for careful inspection of such articles is very important in order that any defect might be detected before treatment. The handling of rubbered garments and articles is so much of a lottery that it is a rule in the dyeing and cleaning industry that these are only accepted for treatment at owner's risk.

Garments such as mackintoshes or raincoats are cleaned by brushing

with soap solutions by hand. After cleaning they are rinsed, passed through a bath containing salt or acetic acid to prevent colours "running" and lightly hydro-extracted. The drying must be at ordinary temperature, since heat would tend to melt the rubber. Many waterproof materials are prepared by sticking together two lengths of material by means of a layer of rubber between them. If at any time the rubber comes away from the material, a "pocket" for water may be formed, and this may develop during the wet cleaning to such an extent that the rubber peels away and the proofing is destroyed beyond repair.

Lace and casement curtains are usually wet cleaned, except in the case of curtains with coloured appliqué trimmings. The appliqué trimming is liable to break away from its stitchings when wetted. Such curtains can only be treated with safety by dry cleaning.

Before curtains are passed into process for wet cleaning, careful inspection is necessary in order to detect if any have been tendered from exposure to the atmosphere. Generally, curtains hold a considerable amount of loose dust and dirt. This is removed by steeping the curtains in cold water for as long as possible. In many works, steeping is carried out overnight. The cleaning proper may be done in an ordinary washing machine. For this, the curtains are placed in large mesh net bags, in order to reduce the strain on the curtain net during the rotation of the machine. The removal of old starch from the curtains liberates still more soiling matters.

Many reagents are now available for this purpose. Malt extract tends to be superseded by such reagents as Diastafor, Nova Fermasol, Rapidase and several others. The more modern reagents may be employed at high temperatures, in contrast to malt extract, which must be used cold. These compounds convert the starch into soluble starches which are easily removed from the curtains. When the starch has been removed, the nets containing the curtains are cleaned in the rotary machine using soap and alkali in equal proportions and sufficient to produce a good lather. This first wash is carried out usually at moderately high temperature, 140°F.—150°F., for 15 minutes. The second wash is merely the first wash repeated. Many cleaners convert the second wash into a boiling process, by running the machine for a few minutes and then gradually bringing the washing solution to the boil. Probably a separate boiling process would give clearer results.

White lace curtains are invariably bleached in order to counteract yellowing. The bleaching reagent is sodium hypochlorite. This may be added to the second wash or the boil, although the best result is obtained if the bleach is added in the rinsing stages. This procedure, however, entails some risk, that some of the bleach may be left in the curtains and cause serious tendering and damage. After the boiling process, the curtains are given a series of rinses beginning with very hot rinses and finally becoming cold. The curtains are then hydroextracted and are prepared for starching.

Curtains are starched with boiled starch solutions made from Potato Starch (Farina), Wheat Starch or Maize Starch. The addition of a small amount of borax to the starch solution increases the stiffening effect.

Coloured curtains are wet cleaned at low temperatures with soap alone. If the colour be cream or ecru, the curtains may be tinted by the addition of suitable dyes to the starching solution.

Window blinds are usually cleaned by hand. Owing to the long exposure to atmospheric conditions blinds are often extremely weak, so much so that they cannot withstand even a wetting-out process. This applies particularly to the bottom portion of the blind. The original starch is removed as with lace curtains, after which the blinds are brushed with soap solution on a table. Blinds require heavy starching and because of this are usually starched twice. The blinds are treated with what are known as "thin boiling" starches. Thin boiling starches are made by treating raw starches with a regulated amount of acid so that the nature of the starch is modified. In this condition the starch gives, with boiling water, a thin paste, which remains liquid even on cooling. The advantage of thin boiling starches is the greater rapidity with which their solutions penetrate textile fabrics. The blinds are dried between the two starching processes. The dry fabric absorbs a greater amount of starch than when wet.

The treatment of cretonne and chintz material is an important part of wet cleaning. Previous to wet cleaning, cretonne and chintz materials require careful examination in order to determine whether the colours will withstand the process. In cases of doubt, the articles should be dry cleaned, but in certain cases it will be found that many articles are too much soiled to be made satisfactory in dry cleaning.

Many of the colours on these fabrics will withstand a treatment by hand, while others may be safely wet cleaned in the rotary machine. Coloured materials in general are treated with soaps free from alkalies. The so-called spirit soaps are very suitable for cretonne cleaning. The rinsing processes are in a series of luke-warm water, with the addition of acetic acid in the final rinse. The acid serves to brighten the colours. Sulphuric acid may be used, but subsequent rinsing is necessary to remove all traces of the acid.

A stiffening effect may be obtained by adding a small amount of starch either with the acid rinse or in a further rinse.

The stamping-off of colours during hydro-extraction is prevented by wrapping suspected materials in sheetings. After extraction, the cretonnes are dried in preparation for the finishing stage.

Chintz materials are given a preliminary de-starching treatment to remove all traces of old starch. After cleaning, chintz is specially starched, usually being given two treatments, the fabric being dried after the first starching. The boiled starch solution must be thoroughly worked into the material, in some cases being worked in a punch tub and then being rubbed into the fabric by hand. After extraction, the chintz is examined on a slab and any unevenness or surface layers of starch rubbed in by hand. The chintz is dried by hanging in the drying room, all linings being separated from the face of the fabric since separation when dry, would be difficult and entail risk of damage.

Fancy cleaning refers particularly to the wet cleaning of ladies' dresses. The great variety of materials used for dresses demands great skill on the part of the wet cleaner for their successful treatment. The danger of shrinkage and loss or alteration of colours calls for special care. The experienced wet cleaner readily detects doubtful colours. In general, fancy cleaning is carried out at low temperature by hand. A preliminary treatment in clear water removes loose dirt and any acid that may be in the articles. Soiled and dirty places are brushed with neutral soap solution. The articles are rinsed by passing through several waters until clear of soap. A final rinse containing diluted acetic acid is given. In the case of silk articles, acetic acid has the double effect of brightening the colours and imparting "scroop". The brushing of silk garments must be very carefully carried out with special soft brushes. Too much friction would cause chafe or "face" marks on the silk.

The wet cleaning of "artificial" silk garments calls for even more care than the treatment of silk garments. In the case of cellulose acetate materials, the selection of the cleaning reagents must be made with the object of avoiding those which would injure or alter its nature. Certain spirit soaps contain solvents which dissolve cellulose acetate. The use of alkalies must be carefully controlled particularly with regard to temperature. The temperature of the cleaning solution should be kept below hand-heat. The wet cleaning of artificial silks should always be by hand. The chief danger with this type of material is that of causing creases or "breaks" which are almost impossible to remove. To avoid this, the articles must not be twisted or wrung out. Hydro-extraction must be carefully and quickly carried out, and the articles spread out flat to dry.

The British Celanese Co. have produced a soap, "Celascour" which has been prepared specially for the wet cleaning of "Celanese". The delustring of artificial silks may be easily disturbed if the wet cleaning is carried out under unsuitable conditions such as high temperatures, hard water and harmful reagents.

When dry cleaning has been unsuccessful in the treatment of carpets and rugs, wet cleaning may be resorted to in many instances. The process must be carried out at low temperature in order to avoid risk of affecting the colours, and because of this the selected soap must be soluble without gelatinisation at all temperatures. Such soap is readily rinsed out. The oleine soaps used in the wet cleaning of flannels and blankets are employed for carpet cleaning.

The carpets are laid on a cement floor with a slight fall in order that the water may drain away. Before wetting, the carpets should be thoroughly beaten so that all loose dust is removed. They are then brushed with soap solution. The brushing may be by hand, but it is more customary to use mechanical brushing machines such as the Finnell Circular Brushing Machine or the Connersville Carpet Machine. The soap lather is removed from the carpet by means of a squeegee. Several brushings may be necessary until a clean lather is obtained. Rinsing is by flushing the carpets with water from a hose and removing the excess water by means of the squeegee. Acetic acid is often used to brighten the colours. Mineral acids may be used, but these must be thoroughly rinsed away to avoid tendering of the

vegetable fibres, cotton or jute which may have been used in the making of the carpet foundation or back.

Owing to the risk of shrinkage when carpets or rugs are "wetted out" completely, a compromise is effected in a process known as "shampooing". This consists in brushing the surface of the carpet with strong soap solution so that the pile is merely damped rather than wetted completely. Mechanical brushes are used, and excess soap and water removed by vacuum suction machines.

Carpets may only be hydro-extracted in specially constructed large extractors. Alternatively, they should be allowed to drain for several hours. After extraction or draining, the carpets are hung in drying rooms, the temperature of which is carefully regulated, while the changes of air are maintained as high as possible.

The preparation of garments for dyeing is closely related to the processes used in wet cleaning. Every article must be thoroughly cleaned before dyeing. Imperfect scouring is one of the most frequent causes of failure in the dyeing of new goods. In the case of worn garments, perfect scouring or cleaning is absolutely essential. Although many garment dyers dry clean their goods as a preliminary to dyeing, the general practice is to wet clean them.

Silk articles are treated in lukewarm soap baths with soft water, great care being taken during handling in order to prevent face marks. There is always the risk that weighted silks may drop during the soaping, especially if the temperature rises above hand-heat. After the soap treatment, silk goods are rinsed several times in warm, soft water and finally in cold water. It is important that the soap should be thoroughly removed before dyeing.

Garments and articles containing wool are cleaned with lukewarm soap solutions to which a slight amount of ammonia has been added. It is possible to clean many of this type of garment in the rotary machine, but every precaution must be taken to prevent shrinkage. The use of wetting out and penetrating agents is an advantage in the preparation of woollen garments for dyeing. In all cases, thorough rinsing is essential, particularly with articles with thick seams. The retention of soap in thick places such as seams will prevent even penetration of the dye.

Cotton and linen goods may be cleaned with soap and alkali at high temperatures, but in the case of articles which have been starched or

heavily dressed previously, the starch and dressing must be removed first by treatment with malt extracts.

In all cases, the garments should be carefully examined for stains which have not been removed by the wet cleaning. Paint stains are the most prevalent.

# Chapter V

#### GARMENT DYEING

It is the golden rule in garment dyeing that all articles must be thoroughly cleaned in the preliminary stages. Particular attention must be paid to stains due to grease or paint.

After the cleaning process, the dyer must decide whether the required shade can be obtained direct from the original colour of the article. In accepting garments, curtains, and other articles to be re-dyed, the dyer should always recommend colours, which are darker than the existing shade.

The manageresses of receiving depots should be well trained regarding the possibilities of re-dyeing over all colours. At one time nearly 75% of the work undertaken by the garment dyer was for dyeing to the standard shades of navy blue, brown, or black, while the remaining 25% was for special colours. The present tendency is for the proportions to be reversed, so that 75% of the work is for special and fancy colours. This means that the number of garments that can be passed straight to the dye vats is becoming less, while the number that require further preparation is increasing.

When the original colour prevents the required shade being dyed directly, the dyer resorts to "stripping" or bleaching processes, in order that the colour is removed or "lightened" sufficiently to allow the required shade to be obtained. Stripping is also necessary when the colour of the treated article has faded in the exposed portion, or in any other way been made uneven.

The stripping method adopted is dependent upon the nature of the article. In the case of woollens, or half-wool goods, sufficient colour may be removed by a treatment with a weak solution of ammonia at 120°F. Wool can often be treated with a warm solution of Glaubers salt. (Glaubers salt is sodium sulphate.) Care must be taken to reduce the time and temperature of the Glauber salt treatment to a minimum. It will be found that treatment beyond 15 minutes at

high temperature (near boiling point) causes the wool to lose handle and become harsh and increases the tendency to shrink. In the case of the vegetable fibres and the artificial silk fibres, stripping may be carried out by means of sodium hypochlorite solutions, but in this case, no other fibre must be present. The bleaching is obtained by immersing the articles for several hours in a solution of  $1\frac{1}{2}$ ° Tw. strength. It must be emphasised that all traces of hypochlorite should be removed by thorough rinsing. In some cases the goods are given an acid bath after the bleaching process, but this should be avoided if possible, because of the greatly increased tendering action which results from it.

Goods containing silk are often stripped by treatment in hot solutions of Marseilles soap, while mixture of wool and silk may be treated with lukewarm soap solution containing a little ammonia.

Stripping by means of hydrosulphite compounds is widely adopted by garment dyers. Hydrosulphites are powerful reducing agents which chemically alter the chemical composition of many dyestuffs and so change them into colourless compounds. It is interesting to note that the colouring matter is not removed from the fibre by the process, nor is any tendering action exerted on the fibre.

Hydrosulphite can be obtained in a great variety of forms and, as such, is marketed under many names. The simplest form is sodium hydrosulphite. This is a fine white powder, which readily decomposes, particularly if allowed to become damp. It is soluble in water, and is used in strength of  $1\frac{1}{2}$  lbs. to 2 lbs. per 100 gallons of water at boiling temperature. It will be found that the solution is a powerful reducing agent, and will completely alter or strip the original shade of many dyestuffs.

Stripping is best carried out in clean wooden or porcelain vats, in which steam pipes are covered with perforated boards or wrapped with suitable material to prevent the goods touching the metal.

In contact with the common metals, hydrosulphite produces the dark sulphide of the metal, which marks off on goods touching the metal. This causes dark stains, which are very difficult to remove.

In the case of vegetable fibres, the addition of soda or caustic soda to the stripping bath increases the action considerably.

A more stable form of hydrosulphite consists of a complex combination of zinc and sodium hydrosulphites, known as Formusol. This form is marketed under various names as Decroline, Hyraldite, and

Rongalite C., and is particularly useful for the stripping of woollen articles. The stripping bathis prepared at 120°F. with 3% hydrosulphite (zinc compound) and 3% acetic acid or formic acid, the percentages being calculated on the weight of the treated goods. The goods are entered at 120°F., and the temperature slowly raised to the boil. The temperature is maintained at or near the boil until the original colour is sufficiently reduced, when the goods must be thoroughly rinsed to remove all traces of the stripping agents.

In many cases it may be found that the original colour is stripped almost entirely or completely altered while in the stripping bath, only for the colour to be re-developed in the rinsing baths. In these cases the dyestuff of the original colour is of such chemical composition that its reduction product is readily re-oxidised to its original state by the oxygen of the atmosphere.

The chemist classifies dyestuffs into categories according to their chemical constitution, while the dyer classifies them according to the method of application in the dye-bath.

Based on their general methods of application, dyes are divided into groups as follows:

- (1) Acid dyes; (2) basic dyes; (3) direct dyes; (4) mordant dyes;
- (5) sulphur dyes; (6) vat dyes; (7) developed dyes; (8) oxidised dyes;
- (9) mineral dyes; (10) dispersed dyestuffs.

The line of demarcation is not always clearly defined, and examples of dyes belonging to more than one group are known. The eosins, erythrosins, phloxines, rose Bengals, and rhodamines may behave as basic or acid dyes.

The acid dyes and the direct dyes are the classes most generally used in garment dyeing, although the basic dyestuffs and the developed dyestuffs may find limited application. The dispersed dyestuffs are of recent introduction, and are specially prepared for the dyeing of cellulose acetate silks.

The acid dyes are used on the animal fibres.

The characteristic of the acid dyes is that they are dyed on wool and silk in an acid bath. They do not as a general class dye the vegetable fibres. The classification of acid dyes may be further subdivided into those, which will dye only from acid baths, and those which will due from weakly acid baths or even neutral baths. To the garment dyer, the property of level dyeing is essential in the dyestuffs employed.

Dyestuffs that "rush" on to the materials must be avoided. The makers of dyestuffs publish lists showing the properties of their dyestuffs, and in many cases classify them as (a) those which are level-dyeing in strongly acid baths, (b) those which are level-dyeing in weakly acid baths, or in neutral baths.

The general method of dyeing with acid dyestuffs is to prepare the bath with the required amount of dyestuff with about 10% of Glaubers salt and about 3% of sulphuric acid. The percentages are calculated on the weight of the goods being dyed. The function of the Glaubers salt is to slow down the rate of dyeing, so that the dyestuff penetrates the materials thoroughly and evenly. In contrast to this, the function of the acid is to "force" the dye on to the fibre.

To prevent the dyestuff rushing on to the material too quickly, and so causing uneven dyeing, it is advisable to commence dyeing at a temperature of 120°F. to 150°F. After the goods have been worked in the bath for a time, slowly raise to the boil. It is essential to successful dyeing that the goods should be kept open and moving in sufficient volume of dye liquor.

When dyeing from acid dye-baths, it will be found that in time the whole of the dye is transferred to the wool, so that the liquor becomes absolutely colourless. In this state the bath is said to be exhausted. The rate at which dyes are exhausted varies considerably. Those which exhaust slowly give good penetration and level dyeing, while those which exhaust quickly are likely to give surface dyeing without penetration causing uneven dyeing. It is important in using more than one dye, as in the case of dyeing to sample, that the dyes should be carefully selected. The best results are obtained when the various dyes have similar rates of exhausting.

Where it is necessary to use dyestuffs, which are known to exhaust quickly, acetic acid or formic acid may be used in the early stages, and complete exhaustion brought about in the last stages by the addition of  $\frac{1}{2}\%$  sulphuric acid. Because of the expense of acetic acid or formic acid in comparison with sulphuric acid, the use of these acids is not widely adopted by garment dyers.

The cotton linings or stitchings remain undyed in acid dyeing, and consequently must be treated in a second bath, using colours which dye the cotton only. This process is known as "filling in" the cotton. Between the acid bath and the "filling in" bath, the goods must be

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thoroughly rinsed in order to remove all traces of acid. The tendency of garment dyeing practice in recent years is to avoid "filling in" as much as possible, as obviously it involves a further process with a proportionate increase in expense. It is found, however, that acid dyeing gives the most satisfactory results on faded woollen articles.

The direct dyestuffs derive their name from the fact that they will dye cotton direct from the dye-bath.

Previous to the discovery and application of this class, the vegetable fibres could only be dyed after mordanting. Direct dyes are also known as salt dyestuffs and as cotton dyestuffs. The reason for the latter name is obvious, but the former arises from the fact that they are dyed from baths containing salt.

The dye-baths are prepared with the required amount of dyestuff with 10% to 20% salt and 1% to 2% alkali. The dyeing is commenced at the boil and the bath allowed to cool down slowly. The direct dyestuffs in general do not dye to full shades at high temperatures, but the temperatures at which these dyestuffs give the best results vary considerably. In the case of mixed dyestuffs it is important that the temperatures of dyeing for each dyestuff should be close together.

The direct dyestuffs have the very important property of dyeing all fibres, with the exception of cellulose acetate. This enables the garment dyer to treat many articles in a single operation. By careful selection certain direct dyestuffs are obtained which dye equally on mixed fibres. The dyestuffs makers' publish lists of these from time to time. The shades obtained by direct dyeing are usually duller in comparison with those obtained by acid dyeing.

The desirability of shortening the time and expense of dyeing has led to the introduction of the process known as Union dyeing. In this one bath method, articles containing cotton, wool and silk can be dyed in solid shades by correct selection of dyestuffs. The Union dyestuffs employed in the process are generally a combination of acid dyestuffs, which dye the wool to full shades from a neutral bath and direct dystuffs, which give full shades at relatively high temperatures. The temperature of dyeing is varied according to the rates of dyeing on the various fibres. If the wool is the paler, the temperature is raised, while if the cotton is too pale, the bath is cooled down. Since after direct dyeing or Union dyeing the handle of the wool may be

impaired, a small amount of acetic acid is often added to the bath, where better handle is desired.

In contrast with the acid dyestuffs, the direct dyestuffs do not exhaust from their dye vats, so that a considerable amount of dye remains unabsorbed.

In order to economise in dyestuffs, garment dyers endeavour to keep the unexhausted dye bath for re-use. These baths are known as standing baths. They are particularly effective with dark shades, but of course, can only be maintained where there is regular demand for particular shades. In the case of navy blue or black, the amount of dye required for fresh baths would be 6-10% of the weight of the treated garments. When the bath is kept standing, only about 3% of dye is necessary for each subsequent bath. The amount of salt added is reduced to about half. Eventually, the concentration of salt becomes such that dye is "salted" out of the solution and "bronzy" dyeing results. "Bronziness" means a marked tendency to rubbing. Before this stage is reached, the bath must be run off and made up afresh.

The basic dyestuffs command a certain amount of attention because of the exceptional brightness of the shades obtained from them. They possess great disadvantages as a general class is not being fast to light or rubbing. Wool and silk are dyed direct by this class of dyes, but the vegetable fibres can only be dyed after mordanting. The mordanting of cotton may be with tannic acid, Katanol, oil mordant or a direct dyestuff. The basic dyestuffs form an insoluble combination with many of the direct dyestuffs. Advantage is taken of this in topping direct cotton dyestuffs with basic dyestuffs. Many direct cotton blacks have an unsatisfactory red tone, but this may be corrected by topping with the basic dyestuff, Methylene Blue 2B.

When cellulose acetate first appeared on the market the garment dyer had considerable difficulty in handling it, particularly in obtaining satisfactory black or navy blue shades. At first, the only possible way of obtaining reasonably satisfactory results was by submitting the cellulose acetate to a treatment with caustic soda. This causes partial "saponification" of the cellulose acetate, so that a film of regenerated cellulose was formed on the surface of the fibre. The regenerated cellulose was then dyed with direct dyestuffs. This process was not possible in the case of mixed fabrics and it became gradually obsolete. The dispersed dyestuffs are of particular interest in the case of cellulose

acetate dyeing. It has been established that dyestuffs most suitable for the dyeing of cellulose acetate are of low solubility in water. Such dyestuffs can be made to form colloidal solutions or suspensions with certain emulsifying agents, which are stable, and in which the particles of dyestuffs are so small that they can pass through the pores of a filter paper. In this way, many dyestuffs have been made available for the dyeing of acetate silk. These dyestuffs are classified as the Dispersed dyestuffs. The British Celanese Co., Ltd., have produced a special range of dyestuffs for the dyeing of Celanese under the series name of S.R.A. In these, the emulsifying or dispersing agent is Sulpho-Ricinoleic Acid. Other examples are the Dispersols and the Duranois, the Celatine colours and the Cibacete colours.

The dispersed dyestuffs are particularly applicable to the dyeing of mixed fabrics. The cellulose acetate is first dyed to the required shade using dispersed dyestuffs, and the wool or cotton dyed afterwards with suitable direct dyes or in the case of wool alone with neutral dyeing acid colours. In many cases, it is possible to carry out the dyeing in the same bath.

Cellulose acetate may also be dyed by developing the colour on the fibre. This method has the great disadvantage of being a three-bath dyeing process, but it is widely adopted for the dyeing of acetyl silk to black.

The dyeing by means of the Ionamine colours is typical of this method. For the dyeing of black, the articles are dyed with Ionamine A.

The acetyl silk garments are dyed with 10–12% Ionamine, i.e., as with direct dyeing. This dyes the material to a dull brown shade, which in itself is of little use as a dyed shade. After dyeing, the goods are rinsed to remove excess dye and are then treated with nitrite of soda and hydrochloric acid. This produces a chemical reaction known as diazotisation. The diazotising bath is freshly made each time with 3–4% nitrite of Soda and 9–12% hydrochloric acid. It is important that the diazotising bath should be kept acid, and quite cold. The goods are kept well open and constantly moved in this bath for about 20 minutes. After one rinsing bath, the black is developed on the fibre by treating in a third bath with a solution of Boxynaphthoic acid S. The treatment consists of working the material in the developing bath containing 3–4% Boxynaphthoic acid S., and a small amount of acetic acid for  $\frac{1}{2}$  hour at 140°F.

The process dyes the cellulose acetate to a full deep black, and when the conditions of dyeing in its "various" stages have been carefully controlled, the black is fast to light, water and rubbing. In many cases, the black may appear heavy or bronze, but this is readily corrected by a warm soap treatment.

When cellulose acetate is present with other fibres, the garments are dyed in the normal manner so that all the fibres, except the acetate silk, are dyed. This dyeing is then completed by developing the black on the cellulose acetate.

The suitability of dyestuffs for garment dyeing depends upon many factors. It is obvious that many garments are sent to the garment dyer to be re-dyed either to the original colour or to darker colours because their original colours have faded in wear. Fades on woollen garments have always been a source of difficulty in re-dyeing. Dyestuffs, which dye faded and unfaded wool to equal shades, are particularly attractive to the garment dyers. As has already been indicated, faded wool garments are best dyed in acid bath (hard dyed) and the cotton subsequently "filled in".

Level dyeing is also very desirable in garment dyeing, and this is an important factor in the selection of suitable dyestuffs.

The factors controlling the selection of suitable dyestuffs for the dyeing of viscose silks appears in contradition to those affecting the selection of other fibres.

It has been determined that when hanks of viscose silk standard size are suspended in standard solutions of dyestuffs, the hanks are coloured by the colour rising up the fibres. By measuring the height to which the hank is coloured in a fixed time, a range of values for individual dyes may be obtained. Contrary to anticipation, those dyes showing least tendency to rise up the hank give the best results on viscose silks.

In addition to the selection of dyestuffs for level dyeing, care should also be taken that the selected dyestuffs are of good fastness to rubbing and light.

# **NOTES**

# **NOTES**

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