THE MANUFACTURE OF VARNISHES, OIL CRUSHING, REFINING AND BOILING

AND KINDRED INDUSTRIES

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THE MANUFACTURE OF

VARNISHES
OIL CRUSHING, REFINING AND BOILING
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KINDRED INDUSTRIES

DESCRIBING THE MANUFACTURE AND CHEMICAL AND PHYSICAL
PROPERTIES OF

SPIRIT VARNISHES AND OIL VARNISHES; RAW MATERIALS; RESINS;
SOLVENTS AND COLOURING PRINCIPLES; DRYING OILS, THEIR
EXTRACTION, PROPERTIES AND APPLICATIONS; OIL
REFINING AND BOILING; THE MANUFACTURE,
EMPLOYMENT AND TESTING OF VARIOUS
VARNISHES

TRANSLATED FROM THE FRENCH OF

ACH. LIVACHE
INGÉNIEUR CIVIL DES MINES

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TRANSLATOR'S PREFACE.

The translator, during his long experience as a teacher of the subjects dealt with in this English version of the work of M. Livache, has all along felt the want of a suitable English text-book.

He has, accordingly, enlarged and assimilated M. Livache's treatise to English practice, embodying the results of his own experience, whilst at the same time he has preserved strictly intact all the French author's theoretical views deduced from the results of actual experiments.

If, in some cases, the *quantities* operated upon may appear trivial to the English varnish-maker, the principle involved is the same whether the quantity be large or small, and, if the process itself be good, it is only a matter for an engineer to put it in actual practice on any scale.

The sections on resins, solvents and vegetable colours have been greatly extended. A short notice of the principal aniline dyes has been added. The crushing of oils and the manufacture of driers have also been introduced. Additional information has been given in regard to Australian sandarach, fair samples of which are now being regarded as equal to the best Mogadore.

Oil boiling by steam, originally introduced in this country by Vincent, has been more generally adopted of recent years. M. Livache's description of Vincent's process, drawn from a German source, has been extended.

These are a few of the many additions which have been embodied. The translation of the work has been free. The object of the translator has been to render M. Livache's ideas clear and intelligible to English readers, whilst, at the same time, he hopes that all the vivacity and verve of the original has been, to some extent, retained so as to present what may be to some a comparatively dull subject in as pleasant a manner as possible. It is hoped that in this way the information conveyed—in many cases reiterated—may be better retained, in the memory of the reader, than any mere dry statement of facts, condensed into a minimum of words. When these statements are repeated, one after another in quick succession, he who runs may read, but he may not understand.

As far as the additions are concerned, the translator returns his obligations and thanks to the numerous authorities he has consulted and is indebted to. It has not been found practicable to quote chapter and verse for every statement made, the more so as it is difficult, if not impossible, to trace the greater bulk to their original source.

The translator's apologies are tendered for the delay—due to ill-health and pressure of engagements—which has taken place in the completion of the work. This may, however, be to a certain extent counterbalanced by the fact that he has, in many cases, been enabled to embody the results of more recent researches, and by the many additions to render the work more serviceable to those for whom it is intended, *viz.*, the Members of the Oil and Colour Trade generally.

Although numerous recipes are given, slavish following of any recipe is to be deprecated. But perhaps in this branch of the trade, for very obvious reasons, the copying of other peoples ideas is not so prevalent as in the Colour and Paint department, where the indiscriminate imitation of each other's colours, show-cards of paints and enamels, and the matching of each other's samples are so prevalent. There is, however, abundant scope for the varnish-maker to strike out a new line for himself, and it is hoped that the present volume may, in some way, serve as a guide.

Starting with M. Livache's book as a basis, in embodying the additions he has made, the translator has not, with the limited time and space at his disposal, found it practicable to present the subject, in such a logical sequence, as he would have desired.

A very copious index is, however, appended, which will facilitate ready reference.

It is hoped in a future edition to render the volume still more serviceable to the English varnish-maker.

THE TRANSLATOR.

VARNISHES.

SECTION I.

DEFINITION, ETC., OF VARNISHES—RAW MATERIALS.

DEFINITION.

THE term "varnish" is used to designate any solution which, when spread by means of a brush, in a thin layer over the surface of an object, or applied thereto in a rational manner by any other suitable means, dries (either by simple evaporation of the solvent, as is the case with spirit varnishes, or by the combined evaporation of the solvent, and the more or less complete oxidation of the residue) to an adherent, smooth, uniform, lustrous, elastic, shining film, unaffected by air or moisture.[1]

UTILITY.

Articles, whether consisting of wood, metal, glass, stone, leather, paper, etc., are varnished because this impervious film or coating preserves them from wear and tear, and the oxidising action of air and moisture, and all those extraneous influences included under the term "weather," noxious gases, etc., whilst at the same time the uniform smoothness, transparency and brilliancy of the film is pleasant to the eye and the touch. Owing to the presence of antiseptic and toxic substances in the resins, oils, etc., which enter into the composition of certain varnishes, these, in addition to preserving the objects to which they are applied from ordinary wear and tear, also protect them from the ravages of insects, *e.g.*, wooden structures from white ants in tropical climates. "Dry rot" may also be prevented in a similar way; or varnishes containing toxic principles, whether present naturally in the

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[1] When varnish is used as a vehicle for pigments a so-called "enamel" is produced. Porcelain glazes are true enamels. As varnish enamels dry with a gloss they save labour by combining painting and varnishing into one operation.

resins or solvent, or purposely added thereto, may prevent undesirable lower forms of animal life from attaching themselves to the object to which they may have been applied, *e.g.*, antifouling compositions, some of which, however, partake more of the nature of a paint than a varnish.

When a coating of varnish has been given to any article it should dry rapidly to a uniform, durable film, possessing the properties just enumerated, neither crumbling nor cracking, but persistently adhering to the object to which it has been applied, and elastic enough to yield or stretch, with any bending action to which the article may be exposed, in its daily use. In addition to a preservative action varnishes have also a decorative effect. This is due to the fact that the application of a coat of varnish to any porous surface, dressed and smoothed in an appropriate manner, enhances its appearance by imparting thereto the uniform polished glassy lustre just referred to. Should the object to which it is applied consist of previously dressed wood, by filling up the pores thereof with a transparent media, it not only brings into bolder relief the more prominent structural features of the wood, but by a light and shade contrast (produced by the harder and denser fibres as against the softer and less compact) brings prominently into view all the characteristic modifications and ramifications of the woody fibre of different timbers known under the term "grain". The whole structure of the wood is, as it were, brought to the same level by actual contact with a glassy surface. By colouring the varnish a stained glassy surface is produced, by which poorer quality timber may be made to pass for better quality; the same result may also be arrived at by using naturally coloured resins, ex. 9 gamboge, dragon's blood, grass tree gum, etc.

CONSTITUENTS.

The principal substances which yield a coating fulfilling these requirements are resins and drying oils.

SPIRIT VARNISHES

Resins are sometimes applied to the object to be varnished in the simple state of solution in a volatile liquid such as alcohol. Such solutions are termed *spirit* varnishes. This class of varnishes yields, however, on drying a brittle coat, consisting practically of the unchanged original resin, presenting all its merits and defects. Spirit varnishes are, therefore, liable to crack, and but little calculated to withstand wear and tear and the action of the weather. They cannot, therefore, be used for outdoor decoration.

OIL VARNISHES.

When resins are dissolved by the aid of heat in a drying oil such as linseed oil[1] (so as to give the resulting coat more elasticity), and the product thinned down with a volatile solvent in which both oil and resin dissolve, such as spirits of turpentine, *oil* varnishes are produced. Drying oils play a double *rôle* : they not only act as solvents, but impart elasticity and durability to the varnish. Oil varnishes have consequently a much more varied and extended use, not only for outdoor but also for indoor decoration.

RAW MATERIALS.

As the quality of a varnish depends in great measure on the materials from which it has been manufactured, it is necessary, at the outset, to study the properties of these raw materials, and their adaptability to serve the special purpose for which the varnish is intended. Besides volatile oils and other solvents, drying oils and gum resins, several subsidiary products are used in varnish-making, such as driers and colouring principles.

Again, in the case of certain special varnishes, various hydrocarbons are introduced, such as bitumens, asphaltum,

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[1] Resina omnis dissolvitur olio.—*Pliny*, xiv., 20.

coal tar, bone, stearine and other pitches, inspissated juices, such as india-rubber and gutta-percha. Collodion and celluloid are also employed in varnish-making.

Resins.

The resins, gum resins, balsams, etc., employed in varnish-making are :—

Amber.
Copal.
Dammar.
Mastic.
Sandarach.
Elemi.
Benzoin.
Turpentines.
Rosin.

Shellac.
Japan Lac or Uruschi.
Chinese Lac.
Dragon's Blood.
Gamboge.
Accroides.
} principally used as colouring principles.
Asphaltum.
Pitches (coal tar, bone, stearine, etc.).

As resins play a part of supreme importance in varnish-making, it behoves us to study them with care, and the particular trend of this study should bear upon such properties as hardness, solubility in different solvents, etc.

Hardness.—Resins are the ingredients which impart hardness and brilliancy to varnishes. Knowing the hardness of any resin, we can to a certain extent determine beforehand the quality of varnish it will produce. In commerce, resins and gum resins are divided into classes, according to their hardness—*viz.*, hard, semi-hard, medium, and soft. The hard and semi-hard resins are furnished by plants belonging to the *Hymenæa* (a family of plants appertaining to the sub-order of *Cæsalpineæ*), the principal of which are the hard copals, or gum animés of Zanzibar, Mozambique and Madagascar, and the semi-hard copals of Sierra Leone, Benguela and Angola.

The medium hard resins are produced by coniferous trees, such are Manilla and Kauri copals.

Finally, the soft resins are produced by plants of the family of *Juglandaceæ*, amongst which are benzoin, sandarach, dragon's blood, and gamboge.

Solubility.—As a general rule, the softer the resin the more easily does it dissolve, and, *vice versâ*, the harder the resin, the more insoluble it is.

The hard resins and semi-hard resins are but very slightly soluble, but they are easily dissolved in spirits of turpentine, linseed oil, and other solvents, by previously heating them in such a manner as to cause them to lose 10 to 25 per cent. or more of their weight, according to the variety of resin dealt with.

Besides its solubility, we have to note, in the case of each resin, its origin, its composition, the principal commercial varieties met with in the market, also the adulterations to which each resin is subject, and the means whereby such adulteration may be detected, as well as the conditions to be observed, in the manufacture of good quality varnish from any given resin.

1. AMBER. (*Density*, 1·08.)

(Synonyms: Lat. *succinum*, Gr. *electron*, Fr. *succin*, Ger. *bernstein*.)

History.—Amber was known many centuries before Christ. The English word "electricity" is derived from the ancient Greek name for amber, *viz.*, *electron*. Theophrastes (300 B.C.) refers to its properties of electrical attraction. It is also referred to in different connections by Homer, Dioscorides, Virgil, Tacitus, Pliny, etc. The Latin term *succinum* is derived from *succum*, juice. Pliny states that it was supposed by their ancestors to be the sap of the pine tree. Homer in his *Odyssey* speaks of "a gold necklace ornamented with amber beads". Although amber is classed by mineralogists as a *mineral*, and generally, but erroneously, included by them in the class of bitumens, it is, in reality, a widely-distributed but, except in one or two localities, a far from abundant *fossil* resin—the product principally of a prehistoric conifer, *Pinus succinifer*, which flourished many thousand years ago upon the greensand beds of the cretaceous formation. The vast forest upon which these pines grew is supposed to have stretched from Holland, through Germany, Siberia, Kamtchatka to North America.

The cones of this tree are often found associated with the resin, so as to leave no doubt as to its origin; but, according to Goppert, it is not the product of any one single species, nor necessarily derived from a coniferous tree. According to some authors, it may, in some cases, be derived from the *Hymenœa*, a species of tree belonging to the *Cœsalpineœ*.

Localities.—Amber is met with in thin plates and nodules disseminated in the sands, clays and lignites of the greensand formation (lower tertiary) along the German shores of the Baltic. It is sometimes found in thin plates between the layers of lignites, but nearer the bark than the centre of the trunk, as in the case of resin-bearing trees of the present day. The district known as Samland, in Eastern Germany, is the chief amber-mining centre. It is from this district that the markets of the world are supplied. Beyond Germany the yield of amber is comparatively trifling and the working unprofitable. On the shores of Britain, especially on the Essex, Sussex, Norfolk[1] and Suffolk coasts, after severe storms, such as the autumnal and vernal equinoctial gales, as well as on the coasts of Denmark and Scandinavia, it is often thrown up on the beach. But in these cases there is every reason to believe it has been washed away and drifted from the Baltic

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[1] Specimens from Cromer may be seen in the Jermyn Street Geological Museum, London.

beds. This is the more credible when we remember that the specific gravity of amber is 1·08, and that of sea water 1·027, and that the force of the waves will, at such a time of wind and weather, easily overcome the small difference in gravity and cause the amber to drift with the current.

A sort of hybrid between amber and copal—copaline or Highgate resin—is mentioned as found in the London clay at Highgatehill and Kensington (London), but not in sufficient quantity to be of any use; moreover, it is as friable as common rosin, and its connection with amber is very problematical. Amber also occurs in the Paris Basin, associated with the bituminous deposits of the Paris clay, and in the departments of Aisne, Loire, Gard and Bas Rhin, and near Bâle, in Switzerland. On the Sicilian coast greenish or bluish-violet specimens are found.[1] It is also found on the Italian shores of the Adriatic. Near Prague, the capital of Bohemia, in sinking a well an extensive deposit of amber was come upon, some of the pieces weighing 2 to 3 lbs. Beds of brown amber occur in Roumania, in the mountains of Sibicio, Valley of Burgo, which, it is said, with capital, skill and energy, might be worked profitably. Although brown is the most prevalent colour, the tint passes through orange, yellow, red, blue, green and even black, often with veins of variegated hues and tints.

In North America amber is found at Gay Head, near Trenton, and Vincent Town, Camden, New Jersey, the specific gravity of the latter being said to be less than that of water; it also occurs at Capesable, Maryland. All the United States' specimens are found in the red clay, subjacent to the greensand or in the greensand itself.

Mining and Dredging.—Amber is mined in a greyish black, peaty earth, in the Hakong Valley, S.W. of the Mien Khorm Plains in British Burmah, at an altitude of

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[1] Fluorescent and other specimens from Catania, Sicily, are shown in the Jermyn Street Geological Museum, London.

1050 feet. It is mined by sinking pits to a maximum depth of 40 feet.

But the most extensive and productive amber-bearing deposits in the world are found in the district of Samland, Eastern Prussia. The amber is mined for systematically, and the working of the mines is a monopoly of the German Government. It is found at a depth of 108 feet below the surface and 46 feet below sea level, in the blue earth, a friable bluish freestone, constituting the lowest stratum of the chalk formation in that region. It varies in thickness from 10 to 30 feet, but only the lowest, 6 to 12 feet, are worked, and as the working is below sea level great care has to be exercised to prevent the shafts and tunnels being swamped by an inflow of sea water and sand. The earth sent up to the surface for examination was formerly washed through a wide-meshed screen or sieve into a long inclined trough, at distances of 6 feet apart; the valuable pieces of amber were collected by means of nets. The rubbish or tailings are run into the sea, through a sieve with meshes of about one-third of an inch. Latterly, however, the troughs and nets have been replaced by sifting machines or jiggers. These jiggers are fitted with sieves with a mesh of one-eighth of an inch, and can pass from 15 to 20 tons of earth through them hourly.

The amount of earth dealt with monthly in these mines is about 7500 tons to 12,500 tons, giving 3 to 6 tons of "large" and a ton to three-quarters of "smalls," the cost of production varying from 4s. 6d. to 7s. a lb.; 20 cubic feet of earth yields about $2\frac{1}{4}$ lbs. of amber of a mean value at the mines of 1s. 3d. for small and 7s. 6d. for large. The exact extent and geological distribution of these amber deposits of Eastern Prussia have not been definitely ascertained, as during hurricanes very appreciable quantities of resin are found amongst the *débris* washed from the sand hills that exist along the coast of a lower geological horizon than the mines. This variety is known as *fliesen* amber (*succin maritime*), that from the mines being termed *erd* amber (*succin terrestre*).

Fliesen amber is softer than *erd* amber, has a smooth, lustrous, rounded surface, whilst that from the mines, *erd* amber, is angular, rugose, and covered by a rather hard crust.

Marine amber is got by steam dredging at Schwarzort on the Kurischer Haff, close to Memel. The yield by dredging is close upon 100,000 lbs. annually, and this large yield has very materially reduced the price. It occurs in nodules, associated with lignite in the sand at a depth of about 10 feet. The sand, as it is dredged, is sent ashore and levigated in the same way as mine amber. In all, Eastern Prussia exports about 150 tons of amber annually. In Western Germany amber occurs under circumstances similar to those of Eastern Germany, *viz.*, in the sea and mines, but on land the mines are not at all profitable.

Insects of different genera living at the present day, but all the species of which are extinct, are often found embedded or entombed in amber, having found a living grave in the original oleo-resin as it flowed from this almost primeval pine, and amongst them several genera of spiders. Some genera of the various insects found in amber still live in America and Australia, but the species may be said to be extinct, but a single living insect, found in America, has been observed in amber, *viz.*, *Lepisma saccharinum*.[1]

Varieties.—According to M. Lebert the following are the different kinds of amber in the market :—

(1) *The coloured Amber of Kumst*, pale yellow and shining, or greenish and dull.

(2) *Bastert Amber*, a deeper, opaque yellow, citron to dark yellow.

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[1] Specimens of amber, enclosing remains chiefly of *Neuroptera* and *Diptera*, from the Prussian shores of the Baltic, are exhibited in the Jermyn Street Geological Museum, London.

(3) *Bone-coloured Amber*, dull, milk-white, opaque yellow in parts, rich in succinic acid.

(4) *Agate-coloured Amber.*

(5) *Schlaubig Amber*, greenish yellow, transparent, but containing organic *débris.*

(6) *Transparent Amber*, from pale yellow to deep yellow, or even red.

Properties—Colour, etc.—Amber is of various tints and degrees of opacity, transparency and translucency, the same specimen often showing marked variations in this respect. Blue is due to the presence of vivianite, ferric phosphate, $Fe_3P_2O_8$. The milk-white, opaque, bone-like specimens owe their appearance to the presence of an excess of succinic acid in the free state. Sunlight darkens the lighter-tinted varieties. Cloudiness, caused by enclosed water, may be removed by boiling in linseed oil. The *fracture* of amber, which is conchoidal (shell-like) is more or less shining and vitreous. Its consistency is solid, hard, brittle. It has no smell, and its taste (if any) is insipid. However, when amber is strongly rubbed it gives off a peculiar odour, and acquires electrical properties, becoming strongly negatively electrified by friction on a woollen cloth.

Fusibility.—The melting-point of amber varies from 287° to 290° C. (about 549° F.) When heated, it decomposes and gives off a pleasant aromatic smell, and burns with a bright flame. When gradually heated in linseed oil to the "boiling"-point of the latter, and kept at that temperature for twenty hours, it becomes soft, pliable and ductile, but does not melt nor decompose. It must, however, be cooled slowly in the oil, or it becomes as brittle as glass. Opaque spots may in this manner be rendered translucent, and the amber moulded to any form, and pieces can even be cemented together.

Distillation Products.—When distilled, amber yields an acid liquor containing acetic and succinic acids; crystals of solid succinic acid next form in the neck of the retort, and then a disagreeable smelling oil—amber oil—comes over; and when stopped at this stage there is left in the retort a more or less brown resin, which is used in the manufacture of varnish. The more control and attention to the action of the heat, and the more skill displayed in stopping it at the proper stage, the finer, brighter and lighter-coloured is the residual resin and the varnish made therefrom. The amber oil as it comes over first is thin and yellow, but afterwards becomes brown and thick, and if the heat be prolonged a volatile product, of theoretical importance only, having the appearance of wax, passes over, to which Berzelius gave the name of crystallised pyretine, but Gmelin that of amber camphor. It is, in fact, identical or isomeric with Borneo camphor $C_{10}H_{18}O_2$, the product of the *Drybalonops aromatica.*[1]

The volatile oil of amber, after rectification, is an excellent solvent of refractory resins, and can be used for this purpose in varnish-making, but its bad smell restricts its use. (See "Amber Oil," p. 144 *et seq.*)

Composition.—The proximate constituents of amber consist of a volatile oil, succinic acid, a resin insoluble in all solvents, and two other resins soluble in alcohol and ether. In alcohol of specific gravity ·840 only one of these latter resins is soluble in the cold. Its ultimate analysis showed it to contain, according to two different analyses:—

	Per cwt.	Per cwt
Carbon	80·59	70·68
Hydrogen	7·31	11·62
Oxygen	6·73	7·77
Ash	3·27	9·93
Loss	2·10	
	100·00	100·00

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[1] Described on p. 147 *et seq.*

Solubility.—Amber is almost entirely insoluble in water, ammonia, benzol, petroleum spirit, acetic acid, carbon disulphide, essential and fixed oils. If finely pulverised and then heated with alcohol, ether, chloroform, benzene, spirits of turpentine, it swells but does not dissolve but very partially indeed. In alcohol containing a little camphor it seems to dissolve slightly. It is also said to dissolve in a mixture of alcohol and spirits of turpentine, heated in a closed vessel. On the other hand, if amber be treated in the manner indicated under "Distillation Products," the residual resin dissolves easily in all the solvents used in the manufacture of both spirit and oil varnishes. In the fixed oils, especially in raw or boiled linseed oil, this residue very easily dissolves with the aid of heat.

Hardness and Density.—Amber which has a density of 1·08 is the hardest known resin, *viz.*, 2 to 2·5 on Moh's scale. After fusion its hardness is slightly less.

Adulterations.—Amber is sophisticated with soft copals, common resins, rosin, and with amber-coloured fluorides.

The first test to apply is that of smell. All other resins, even copal, give off a characteristic odour when rubbed between the fingers. The second test is the behaviour of the sample when heated. Soft copals melt between 180° and 200° C. A fragment of the substance when held in the candle flame, if pure amber, will melt without running. Should it contain copal it will run down in drops. Real amber gives off sulphuretted hydrogen when strongly heated. Amber is insoluble in cajeput oil, etc., whilst some copals are said to be quite soluble. The addition of copal may also be recognised by the development of the smell characteristic of copaiba balsam on treatment with potash solution. If hard, better quality copal has been added to the sample, it may be detected by a test depending upon the difference in hardness between amber and copal. A crystal of rock salt, as pure as possible, is cleaned so as to present a well-polished and brilliant face. The operator then tries to scratch this face with the sharp point of a piece of the sample. If the face of the crystal, on inspection with a lens, shows scratches, the sample is pure amber; whilst hard copal would not produce any impression.

Common resins and rosins may be detected by softening in boiling water, or by their solubility in alcohol. Fluorides are easily detected by testing with a metallic point.

Uses.—The finest kinds of amber are used in the manufacture of articles for personal adornment, and other articles of ornamental turnery, beads,[1] trinkets, etc., as well as for mouthpieces for pipes and cigar-holders, which has given rise to the mineralogical witticism that it is usually associated with meerschaum. Orientals regard amber trinkets with more favour than Europeans. When intended to be used in the manufacture of ornaments, the amber is split on a leaden plate on a lathe, and then ground to the necessary shape on a Swedish whetstone, and afterwards polished with chalk and water or vegetable oil, and finished by rubbing with a woollen rag. Owing to the friction developed in grinding and polishing the amber becomes very hot and highly charged with electricity, even to such a degree as to produce electrical currents in the arms of the operator, giving rise to nervous tremors, like those from a galvanic battery. These can easily be obviated by the operator working the pieces one after the other, so as not to allow any one of them to get unduly heated. The German towns engaged in the working of amber and the

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[1] A string of amber beads of Chinese workmanship, and a necklace of amber beads as worn in Finnland, are on exhibition at the Jermyn Street Geological Museum, London. Several magnificent amber mouthpieces as used in Constantinople may also be seen there.

selling of both crude and finished articles are Danzig, Königsberg, Stolpen, Breslau and Lübeck. The chief markets are Constantinople, London, Paris, Moscow, Vienna and New York.

Points to be observed in Buying, etc.—Buy only the best quality. Inferior samples yield dark varnishes—difficult to sell. It is very questionable whether amber is at the present day used in England to any great extent as an ingredient of varnishes There are, however, violin varnishes on the market, the resinous constituent of which is guaranteed to consist wholly of amber. Formerly, no doubt, when copals were not so abundant, it played an important part in the manufacture of high-class coach varnishes and japans.

2. Copal and Animé.

Definition.—The term "copal" is not applied to any one species, but to a whole class of resins of different origin, which may agree in properties one with another in a more or less marked manner. These resins are, or have been, produced by different species of plants whether living or extinct belonging to the family of *Hymenæa*, a genera of plants included in the natural order of *Cæsalpineæ*. However different the various copals may appear to the eye, they all agree in being more or less hard and in having a high melting-point, and in yielding by dry distillation a volatile oil and a residual resin which readily dissolves in all varnish solvents.

Classification.—The various kinds of copal may be divided into three different classes:—

(1) *Hard Copals* or *East Indian Animé*, *Bombay Copal*, *Calcutta Copal*, comprising the copals produced on the *east* coast of Africa and India.

(2) *Semi-Hard* or *West African Copal.*

(3) *Soft Copals, American Animé (copal tendre)*; including Kauri, Manilla and Borneo copals.

General Properties—Physical Appearance.—Copal is met with in the form of round tears, stalactites, or in flat pieces the size of which is generally in inverse proportion to the hardness. The hard copals rarely reach the size of a man's fist, while masses of soft copals are found weighing 60 to 120 lbs. and over.

The surface of copal consists almost always of a very characteristic crust of greater or less thickness, which varies in colour. This crust can be removed mechanically by a sharp tool, or by washing with alkaline solutions. In the case of hard copals, this crust may have an appreciable thickness, whilst it is very thin in that of soft copals.

When this crust is removed, certain copals show a smooth surface, whilst others are covered with mammillary asperities of an irregular polygonal form, whilst some are ramified by striæ all over their surface. (See also "Goose-Skin," p. 24.)

The colour varies much, especially with soft copals; often in the same piece several colours are observed; in fact, all colours are met with, blue excepted.

Transparency.—Transparency is almost invariably absent, with the exception of South African copal.

Fracture.—The fracture is generally conchoidal, except in the case of some hard sorts, which are flat and dull. Generally, however, the fracture is lustrous or resinous, especially with soft copals. In the case of the best Zanzibar sorts, the fracture gives dull and lustrous alternations. The fracture of a hard copal, on exposure to the air, does not become dull, whilst soft copals become duller and duller.

Smell and Taste.—With the exception of Zanzibar copal, which is perfectly insipid and inodorous, they have all a more or less agreeable aromatic smell, which varies with each sort. Borneo and Manilla copals have a very decided and characteristic fragrant and pleasant odour. Kauri copal has a pleasant aromatic taste and fragrant smell, whilst the smell of South American copal is unpleasant and repugnant.

Friability and Plasticity.—Between the teeth copal breaks to a powder, which does not reagglutinate.

Hardness.—Hardness is the most important property of copal. Andés, and also Bottler, after carefully studying and establishing the hardness of each variety, arrange them as follows, starting with the hardest and finishing with the softest variety :—

Andés Scale of Hardness (*Copal*).

1. Zanzibar.
2. Mozambique.
3. Madagascar.
4. Sierra Leone fossil.
5. Red Angola.
6. Benguela.
7. Bastard Angola.
8. Globular or Glass.
9. Akra.
10. Benin.
11. Loango.
12. Gaboon.
13. Congo.
14. Sierra Leone ordinary.
15. White Angola.
16. Kauri.
17. Manilla.
18. Borneo.
19. Singapore.
20. South American (Brazil and Guiana).
21. Cochin China.

Bottler's Scale of Hardness (*Copal*).

1. Zanzibar.
2. Red Angola.
3. Sierra Leone fossil.
4. Do. ordinary.
5. Benguela yellow.
6. Benguela white.
7. Congo.
8. Manilla.
9. White Angola.

According to Bottler, all copals are scratched by rock salt—No. 2 of Moh's scale—but Zanzibar, Angola and Sierra Leone fossil only very faintly so. According to Wiesner, Zanzibar and Mozambique copals scratch rock salt, but are scratched by sulphate of copper (blue vitriol).

Again, Bottler states that Manilla, Congo and Sierra Leone cleave (split or shell off) when a needle-point is directed against any one of the smooth faces. The other copals, on the contrary, do not shell off in this manner.

All copals are scratched by calc spar—No. 3 of Moh's scale—but all, with the exception of fetchy sec, scratch talc—No. 1 of Moh's scale.

Fusibility.—However different may be the appearance, size and hardness of the different variety of copals, they all possess a melting-point, which, although very different in the case of each, is nevertheless very high in all; the melting-points usually quoted lie between 180° and 340° C. But recent determinations by Bottler fix them between 90° and 315° C., as shown in the following table:—

	Degrees C.
Copal hymenœa	90 to 95
Manilla yellow	115
Benguela yellow	180
Do. white	185
Congo	190
Sierra Leone	195
Do. fossil	230
White Angola	245
Zanzibar	275
Red Angola	315

Heated in the candle flame, they burn with a smoky flame, melt and run down, drop by drop.

Copal Oil.—Under the action of destructive distillation, they act like amber and yield a volatile oil and a resinous residue, which dissolves, at a low temperature, in the usual varnish solvents.

Specific Gravity.—The hard copals are of less density than the soft copals—*i.e.*, the gravity is in inverse proportion *to* the value. Bottler attributes this difference to the air included in the cavities of the harder varieties. By reducing the copal to fine powder, and thus expelling enclosed air, he found their density to increase, rising, for example, from 1·068 to 1·082, and from 1·069 to 1·111, and the harder the copal the greater was the increase in density. He, however, found some hard copals denser than softer varieties. Zanzibar copal, for instance, has a density of 1·0621, whilst that of white Angola was only 1·035. Other authorities, however, give a higher density for white Angola. Meich and Stinghl, for instance, give 1·062.

Solubility.—The different varieties of copals vary much in their solubility, and the different data given in books are of no value, except in regard to the variety of copal which is referred to; and this is exactly the point which investigators have almost invariably omitted to supply.

The solubility of copal is regulated by the proportions and the nature of the different resins of which it is composed, these resins behaving very differently under the action of different solvents.

In fact, Unverdorben separated five different resins from certain South African copals, which he respectively named A, B, C, D, E.[1]

Resin (A) melts at 100° C., and dissolves in all proportions in 72 per cent. alcohol.

Resin (B) does not melt at 100° C., is insoluble in alcohol of 67 per cent., spirits of turpentine, fixed oils; soluble in absolute alcohol and ether.

Resin (C) is separated from the residue insoluble in absolute alcohol by saponification with alcoholic potash and decomposition with sulphuric acid. It simply froths at 350° C., is insoluble in absolute alcohol and spirits of turpentine; it is insoluble in ether and cajeput oil.

The resin (D), extracted from the unsaponifiable residue by exhaustion with alcohol and decomposition with hydrochloric acid, undergoes no real fusion at a temperature exceeding 300° C.; it is insoluble in absolute alcohol, ether, spirits of turpentine, unless it has been softened by heat.

Finally, resin (E), which resists all the preceding treatments, forms a compact infusible mass insoluble in alcohol,

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[1] This fractional separation by solvents is of little or no value to the practical varnish maker. It is, however, a typical illustration of the methods of analysis of resins adopted by the older chemists.

ether, spirits of turpentine, fatty oils, and neither combines with acids nor alkalies.

It will be readily understood that different copals will comport themselves differently in any given solvent, according as one or other of the above resins predominates in its composition.

Influence of Age and Origin on Solubility.—Further, the origin and age of a copal influence its properties, the more so as the air ought to have a marked influence on the resin as it runs from the tree which yields it. This is confirmed by the fact that resins (D) and (E)—covered with alcohol or, better, a mixture of alcohol and ether—absorb oxygen and change into resins (A), (B) and (C).

It will thus be seen that any data given in regard to the solubility of copal in books or memoirs is of no value whatever unless the variety of copal in question be mentioned.

It may be stated as a general rule that hard copals are almost completely insoluble in the usual solvents, whilst soft copals are partially or completely soluble.

Action of Ether.—According to Berzelius, copal gives a jelly which, according to Vogel, dissolves on treating with successive additions of hot alcohol, but he does not state upon what variety he wrought. With a soft copal, such as Manilla, complete solution may be effected by exposing it for some weeks to the air, in rough powder, or by torrefying it slightly.

Action of Alcohol.—Hard Zanzibar copal softens in alcohol, but can hardly be said to dissolve. Forty per cent. of Sierra Leone copal, which is softer, dissolves in 96 per cent. alcohol, leaving a viscous greyish mass, which may be dissolved by the addition of a little spirits of turpentine.

Action of Camphor.—According to Berzelius, certain kinds of copal dissolve in alcohol in the presence of a small quantity of camphor. Vogel states that this is also the case when they are exposed to the vapour of either pure or camphoretted alcohol.

According to Bottler, Manilla copal, after being exposed to the air, dissolves in alcohol. In a close vessel perfect solution is obtained. Likewise it can be completely dissolved, after torrefaction, in a mixture of equal parts of absolute and amylic alcohol.[1]

Cajeput Oil.—This oil, possibly owing to its containing a species of camphor, seems to possess very marked solvent properties for copal, and in particular for Angola, which it is said to dissolve completely. *Oil of Rosemary* and Lavender oil act similarly, and probably from the same cause.

Amylic Alcohol.—According to Vogel, this should be a very active solvent, but it is scarcely probable that it attacks the hard copals. With the aid of heat the copal swells rapidly, and dissolves completely on boiling; the clear solution spread on a glass plate gives, on drying, an almost colourless coating of a very transparent bright varnish. This solution is partially precipitated by absolute alcohol, but clarifies on the addition of amylic alcohol and chloral. The value of amylic alcohol as a solvent is greatly counterbalanced by its dreadful toxic effects.

Nitric Ether.—Nitric ether, which is easily procured cheaply, dissolves Manilla copal completely. It commences by softening the resin, finally giving a green solution, passing to brown. This solution may be brought to the requisite consistency by the addition of alcohol.

Aniline acts almost instantly upon all the copals, completely dissolving soft copals, but this solution is very dark, and its smell prevents it being generally adopted as a solvent.

Spirits of Turpentine.—Andés observed no immediate action, but after a time complete solution was effected. Angola copal dissolved completely after ten or twelve years, an

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[1] Certain varieties of Manilla dissolve in alcohol without any previous treatment whatever. Many shellac varnishes are grossly adulterated with Manilla and rosin, the rosin counteracting the tendency of the Manilla to string.

effect probably due to the oxidation of the spirits of turpentine, in fact, when copals are treated with artificially-oxidised oil of turpentine, all the copals are more or less attacked, Sierra Leone copal dissolving completely; the others do not dissolve completely, but may be made to do so by a series of successive treatments with the oxidised spirits of turpentine.

Action of Oxygen.—Moreover, oxygen has an appreciable action upon the solubility of copal. It would appear that the solubility varies with the percentage of oxygen in the resin, and it is probable that if the different sorts of copal were reduced to powder, preferably under water, and afterwards exposed for a sufficiently long time to the air, that the powdered resin would be found to dissolve the more easily the more oxygen it would absorb.

Mechanical Agents.—In this connection Andes made interesting experiments. He reduced the resin to very fine powder, as no appreciable results are obtained with coarsely-ground resin. He also used mechanical agents to increase the solubility. Thus, in crushing the gum with fragments of fluor-spar or sulphide of antimony in the proportion of one to seven, the solubility increases in a marked manner, because in breaking copal with substances harder than itself the fineness is increased, and, consequently, the action of the air is facilitated. Moreover, these substances isolate the finely-ground particles of copal, thus preventing agglutination and enabling the solvent to act upon a larger surface.

Alkaline Solutions.—All copals are, more or less, attacked by alkaline solutions. Soft copals are easily saponified without using a high temperature; hard copals are strongly acted upon, and complete saponification takes place with Angola copal. (See under "Resinates," p. 211.)

Heat.—Submitted to the action of heat, and after a partial decomposition has been undergone, resulting in the evolution of succinic acid and a volatile oil, even the hardest copals dissolve in the majority of solvents.

Adulterations.—The chief sophistication consists in mixing inferior with superior qualities. This may be detected in the way described under "Amber"—*viz.*, by the lowering of the melting-point, which, for the better sorts of copal, varies from 275° to 315° C., according to some authorities, or 340° to 360° C., according to others.

Buying Precautions.—The resin should be free from dust, which gives bad results. When running Zanzibar copal, well-known brands should be chosen, the pieces being equal in size, and the colour as uniform as possible throughout the mass.

Angola and Benguela copal ought to be chosen in as large pieces as can be obtained, especially if it is intended to wash them. Care must be taken that the pieces are as hard as they are big, so that the resultant varnish may be hard and durable.

It is advantageous not to buy unwashed West African copal (Sierra Leone, Angola, Congo). They eventually run cheaper washed, because when unwashed they contain much dust, impurities and vegetable *débris*.

Many sorts of Borneo and Manilla copals are on the market. It is best to shun those of a deep brown dull colour and non-resinous lustre. Those with a dull fracture yield soft varnishes, which do not bind well.

Varieties of Copal.

GROUP I. EAST AFRICAN.

This group consists of the hard fossil *Zanzibar* copal, including *Animé*, *Mozambique* copal, and *Madagascar* copal.

Zanzibar copal, known also as East Indian, Bombay or Calcutta copal, because these were the ports from whence it was shipped to this country, is found chiefly in the district in and around Zanzibar.

There are two sorts: fossil, or ripe (*sandarusi*), and recent or raw (*sandarusiza mti*, commonly called *chakazi*: hence corrupted to *jackass*). The particular species of tree which yielded the fossil or ripe copal is undecided, but is supposed to be allied to, if not identical with, the species which now yields the recent or virgin.

(1) *Raw Zanzibar Copal.*—The recent or *raw* copal flows from the *Trachylobium verrucosum* (Klotsch) [*Hymenæa verrucosa Lam.*] and *Trachylobium mossambicense* (Klotsch). This tree, called by the Arabs *Shajar-el-sandarus*, still grows on the island of Zanzibar and on the mainland, often attaining a height of 20 to 25 feet to the first branch, and 3 to 5 feet in girth. It not only grows on the coast, but extends inland beyond the coast ridge, where it is said to be more abundant. It dwindles out as it approaches the limestone formation. The resin is either collected on the tree or is found unchanged at a very small depth in the loose soil. Attempts have been made to acclimatise the tree in India and Australia. Like resin-bearing trees of the pine tribe, whenever a natural puncture has been made on the bark, there does the resin flow. Every organ of the tree is saturated with resin; even the fruit legumes are dotted with excrescences of bright transparent resin enclosed in a thin cuticle. The recent or raw resin is collected either by picking it from the tree, or it is dug out of the earth, as on Zanzibar island, where it has penetrated to a shallow depth, but undergone no change. It finds a market in Bombay, from whence it is transhipped to other parts of India and China, where it is made into low-class varnish.

(2) *Fossil Zanzibar Copal.*—The true fossil ripe Zanzibar copal, the *sandarusi* proper, is supposed to have been the product of the same species as now yield the raw or recent copal. Sometimes it is found when sinking hut

piles: at other times it is left by a high tide on the foreshore. According to Burton, the copal coast extends along the seaboard from Ras Gomani in south latitude 3° to Ras Delgado in 10°41′, stretching inland with an average breadth of thirty miles. The whole of this district contributes its quota of the copal sent to market. The greater bulk, if not the whole of this district, would now appear to be included in German East African territory. But even a small section of this tract, from the mouth of the Panguni river to Ngao (Monghou), would, if systematically exploited, meet all demands. Even in the most abundant deposits it is only found in nests or patches, thus probably indicating that it was the product of isolated individual trees. The natives work the deposits very unmethodically. Unless they strike resin at the first trial, they go no further, but dig fresh holes in the loose, sandy soil in which it is principally found, until finally they come upon resin. As a rule, the redder the soil the better is the copal.

On the *island* of Zanzibar the copal consisting entirely of *chakazi* is found in the vegetable mould overlying a subsoil of blue clay.

The finest and best fossil copal on the mainland is found on the banks of the Rufiji river, particularly in the north district of Wandé, now German territory, and is brought to the coast by travelling dealers or the natives who dig it.

"Goose-Skin."—Some authorities hold that the peculiar puncturing of the surface of Zanzibar copal known as goose-skin is due to the liquid resin falling upon sand, and the peculiar hollows which give rise to the appearance are simply the imprints of the sand. It is contended, on the other hand, that these markings are due to the conformation of the woody fibre of the tree itself. It has also been pointed out that the flow of resin, always a sign of weakness on the part of the plant, increases with age and decay, and that it is the tissues of the wood surrounding the aggregation of resin which imprints the goose-skin structure thereon. As an actual fact this process goes on in the case of trees existing at the present day. As the trees which yielded fossil copal decayed, and in the course of time became completely converted into mould through the ordinary natural agencies, the resin which resisted all natural destructive forces became imbedded in the drifting sands, which swamped the district on which the primeval forest stood. When first dug up the resin does not show the goose-skin, owing to its being covered with a crust produced by the action of the soil and natural elements. This skin can be easily removed by washing in an alkaline ley, bringing the goose-skin into bold relief.

Method of Collecting.—The method of collecting the resin is very simple. A hole about 6 ins. wide is dug out of the earth with a sharp stick, and the earth removed by hand as far as the arm can reach. The mean quantity gathered in a day by a native is about 1 lb., but he could easily gather ten times that quantity. Digging cannot be carried on in the dry season, as the resin is then very brittle, and the rude native tools unsuitable, owing to the hardness of the ground: hence working is confined to the rainy season (April to October).

Washing.—When the rainy season is over, the unsorted copal is taken to Zanzibar, where it is sifted from extraneous matter before exportation. It is also sometimes cleansed here. This operation is performed by brushing the resin in vats, into which a current of water runs, or, better still, by washing with a dilute alkaline ley (containing 2 per cent. of potash), and then rinsing well in pure water, so as to leave no trace of alkali on the resin. It has now lost about 20 per cent. of its bulk. The resin is then sun-dried for some hours, taking great care to avoid dust and dirt being incorporated with the resin. It is then brushed sufficiently hard to remove the outer crust, but leaving the goose-skin intact. The dirt still lurking in any hollows is removed by a knife.

Garbling.—This is the next operation; it requires skill and knowledge on the part of the workman. The gum is sorted out into pieces of the same size, colour, hardness, etc. Hence the necessity of experience, and the ability to recognise the different sorts at sight, the different degrees of hardness, and shades of colour passing from white and transparent through lemon-yellow to bright red, blackish and greenish. It is, however, often exported in the crude state to the East Indies and America, there to be washed and garbled.

The copal which comes to the European market is all cleansed on the spot before exportation. The best brands are those of Oswald & Co., of London, Zanzibar and Hamburg. Colour is represented by the initial letter W = white, R = red. Size of the pieces by A, B, C, and so on; thus W A = white large; R B = red medium. The semi-transparent plates, varying in size from that of a threepenny bit to that of a crown piece, and in thickness from $\frac{1}{6}$ to $\frac{1}{2}$ of an inch, are in general the best. The darker varieties consist of irregularly shaped rounded masses or angular lumps with occasional globules, the size varying from that of a pea to that of a man's fist. The weight of individual pieces of the darker sorts varies from 2 to 3 ozs., whilst sometimes lumps weighing from 5 to 35 lbs. have been unearthed. The *melting*-point of Zanzibar copal is about 280° C., at which temperature it gives off 10 per cent. of an essential oil—copal oil. According to Sir John Kirk the exports of copal from Zanzibar sometimes reach 800,000 lbs., value £60,000.

(3) *Mozambique Copal.*—In 1883 the British consul at Mozambique reported the discovery, at Inhambane, of a tract of copal forest 200 miles long, extending inland for

100 miles parallel with the coast. Samples of this new fossil copal were sent to England, and upon practical tests being made upon its suitability for varnish-making was favourably reported on and valued at from £80 to £100 per ton. It is very similar to Zanzibar copal, even to the so-called goose-skin. The natives call the gum *stakate* and *staka*, the Zulu *inthlata*. Some of these samples are now contained in the museum at Kew.

An extract from a letter from Inhambane, of date 5th February, 1886, states: "Many tons of copal have been exported from Inhambane. For some pieces I have received as high as £13 10s. per cwt. The average price realised on large lots has been £7 per cwt. The forest containing the trees extends from the River Sabia, in a south-westerly direction, as far as Beleni."

Properties.—This resin is met with in the form of thin plates or flattened globules, varying in *size* from ¼ of an inch to 2 inches; the *colour* is sherry yellow, often inclining to red. Pale yellow pieces are seldom met with; the fissures running through the resin are red, so also is the crust or efflorescence on the surface of the resin when freshly dug up. The surface is warty but without the regularly-shaped facets of Zanzibar copal. The *fracture* is conchoidal, the *lustre* vitreous, and the resin has neither *smell* nor *taste*. Its density is 1·069.

Fresh seeds were also received at Kew, and several hundred plants raised from them, which have been distributed from Kew to India, Singapore, Jamaica, Trinidad, Demerara, Dominica, and tropical parts of Australia.

(4) *Madagascar Copal.*—This variety is said to be the product of a species of *Trachylobium*. It occurs in the form of plates, cylindrical or oval pieces of a deep yellow colour, with a smooth weathered white crust. It is not faceted like Zanzibar copal. When washed the surface is

lustrous, but the resin itself is much contaminated with plant *débris*.

GROUP II. WEST AFRICAN COPALS.

Copal is still more abundant on the west than on the south-east coast, being dug over a coast line of over 700 miles in length, between 8° N. and 14° S. latitude, including therein the coast lines of Sierra Leone, Ashanti, Dahomey, Gold Coast, French Congo, Congo Free State and Angola. The copal is found, at various depths, up to 10 feet from the surface, embedded in layers of marl, sand and clay.

The principal varieties distinguished by the names of the localities from which they are derived are *Sierra Leone*, *Angola*, *Benguela*, *Akra*, *Benin*, *Loango*, *Gaboon*, *Congo*. The young copals of Sierra Leone and Akra are the products of species of *Gibourtia*. Angola, Benguela, Congo, etc., copals, are generally fossil resins. The water-rubbed, rolled and rounded forms of Sierra Leone copal—the so-called Pebble copal—found between the rivers Pongas and Malaenzi, would seem to indicate that at some time or other the resin had been transported by running water from some considerable distance inland to the coast. The young copal, specific gravity 1·06, is found in bright nut-shaped pieces, sometimes in tears, with a strong aromatic odour; it melts at 195° C.

(1) *Pebble Copal* (*Sierra Leone*), *specific gravity* 1·055, is found principally in the form of small pebbles in the neighbourhood of Cape Verde, not unlike amber, colourless or white to a bright yellow, transparent or translucent, sometimes but rarely partially covered with a fine-grained opaque whitish crust. The pebbles vary in size from that of a pea to a large-sized pigeon's egg. Larger pieces are occasionally, but seldom met with. It is the hardest of the West African copals, ranking, in this respect, next to Zanzibar copal. It

melts at a high temperature, and by dry distillation yields an essential oil so neutral that even concentrated potash solution does not attack it.

(2) *Gaboon Copal, specific gravity* 1·073, is found in rather large size, flattened or rounded pieces $\frac{1}{3}$ to 3 inches in diameter. Its yellowish red colour passes sometimes to white with red streaks in it.

Its surface is, in general, smooth white, with a very thin chalk-like crust. The fracture is conchoidal to splintering and of a vitreous lustre; when fresh, the scratch on new surfaces is smooth, on old surfaces splintering.

(3) *Loango Copal, specific gravity* 1·064, which is found in sticks sometimes 12 to 15 inches in length, and which must have been of an indefinite length originally, is much esteemed because of its hardness and homogeneity. The colour is white, yellow and red, and these colours may be again subdivided. There is, however, on the surface a thin reddish layer. The surface is smooth, clean or resinous with a slight efflorescence easy to rub off, or entirely smooth or covered with excrescences. The fracture is conchoidal with glistening surfaces. The red variety being the most uniform is the most esteemed.

(4) *Angola Copal, specific gravity* 1·065.—This kind is met with in several varieties which differ from each other as much in colour as in hardness. The coloured varieties are brighter, more transparent and homogeneous than the duller and paler kinds. It occurs as rounded or globular, rarely flattened, pieces of about 1½ to 2 inches in diameter, and covered naturally with an earthy dirty white to brown crust to a greater extent than any other copal.

The *yellow* variety is covered with a reddish efflorescence. It is transparent, and hard and distinguished for its facets similar to those of Zanzibar copal. It is found in the three forms named above.

The *red* variety is rounded, irregular, with a hard opaque red crust to a depth of almost ⅓ of the diameter. It is sometimes irregularly faceted, or studded with crevasses. This red variety is one of the hardest copals of the west coast of Africa.

White Angola, which is not so much esteemed and is softer than the red, occurs in flattened pieces, or as small nuts with yellowish or blackish portions, and covered with a white efflorescence.

(5 and 6) *Congo and Benguela Copals.*—These are so similar to Angola copal that the one description practically applies to the three.

(7) *Akra Copal* occurs in shapeless pieces of a brown-like appearance, whitish in colour, and covered with a very fine whitish efflorescence, although sometimes presenting a uniform cloudy surface, especially in the case of those pieces of a pronounced bone-like form. In hardness it comes next to Angola.

(8) *Benin Copal*, found in irregular white, yellow, or red pieces covered with a yellow or red crust, often contains impurities. It is similar to Akra copal but softer.

Ogea Gum.—A hard fossil resin, said to be of the nature of copal, was introduced to notice in 1883 by Captain, now Sir Alfred, Moloney, from the gold coast. It is described as being the produce of a leguminous tree closely allied to *Daniella thurifera.* For lack of proper material, however, its species has not yet been determined. The gum is used by the natives for lighting fires and for illuminating purposes; it is also used when powdered as a body perfume by the women. It exudes from the trunk either from wounds or from holes caused by the boring of insects. The gum has not yet appeared in commerce, and would thus appear to be more of the nature of gum than that of copal.

Kauri.—This is a semi-fossil resin of New Zealand, known to the trade as gum kauri. It is secreted by a gigantic coniferous tree, *Dammara australis*, highly esteemed for its timber as well as its gum. The best quality is dug from the ground beneath the trees or where the trees do not at present exist. In 1853 the total exports of kauri amounted to only 829 tons, valued at £15,971; in 1883 this had risen to 6518 tons, valued at £336,606. It is said that over two-thirds of the produce goes to the United States. Though the quantity imported into this country is very large there are no available statistics.

The semi-fossil kauri is found in the province of Auckland in open bushland, where no vestige of the original tree remains. This conclusively shows that it is the product of a prehistoric forest. In former days kauri was dug by the natives, but a large number of white people—generally of the lowest orders, composed principally of Austrian immigrants or refugees—are now engaged in this occupation, recently therefore the number of persons engaged in the industry has been swelled to about 10,000 by the unemployed.

Consequently the supply of gum brought to the Auckland market has greatly increased. As a result of the Austrian immigrants returning to their mother country as soon as they have made a competency, the New Zealand Government have recently imposed an export duty of £3 per ton on kauri resin.

Kauri gum has a conchoidal fracture and a greasy lustre. It is scratched with a metallic point without the scratch radiating. Heated between the fingers it gives off a disagreeable smell. Its taste is feebly aromatic and rather pleasant.

According to Bottler, kauri gum, after being torrefied very slightly, dissolves in greater part in alcohol, to a rather less extent in a mixture of ether and alcohol by digesting it at a temperature between 20 and 25 C. With acetone it partially dissolves, becoming limpid on prolonged digestion at 30° C. This torrefied resin may also be dissolved in a mixture of 12 parts of ether and 1 part of camphor. Its chief use is as an ingredient of "oak" varnish.[1]

3. DAMMAR.

Origin.—The term "dammar" is more generic than specific, *i.e.*, it is applied to several more or less closely allied resins, *e.g.*, East Indian dammar, sal dammar, black dammar, rock dammar.

Commercial Varieties—(1) *East Indian Dammar.*—This variety (dammar properly so called), also known as "white," "Singapore," "Batavian," or "cat's-eye" dammar, is the product of the gigantic Amboyna pine, *Dammara orientalis* or *Dammara alba.* This tree is a native of Java, Sumatra, Malacca, Borneo and the Moluccas. The resin is mostly obtained from Amboyna. It exudes from large excrescences on the lower part of the stem, in the form of a "tacky" liquid, which soon consolidates into a mass of resin. The wood of the tree abounds so much in resin that in certain localities, as in Sumatra, the natural secretion from the excrescences is so copious that it is not necessary to stimulate it by incisions. This is, however, done in other places, near the foot of the tree, and the resin collected in suitable vessels. The exuded resin forms large, transparent, colourless stalactites, with a vitreous lustre which gradually assumes a yellowish hue.

East Indian dammar is met with on the market in the form of nodules or tears of various sizes, generally about $\frac{1}{4}$ inch or so in diameter, but often larger. It is usually coated on the surface with a white powder, from the mutual rubbing of the nodules against each other. This dust deadens the otherwise bright lustre of the gum, and masks its transparency and translucency. The usual

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[1] Hard Manilla copal resembles kauri but is inferior in quality. Soft Manilla of a pale yellow colour very much resembles East Indian dammar.

colour is pale white, with a very faint tinge of yellow. It is very friable, brittle and pulverulent. It is scratched by copal, and (some varieties) by mica. Its fracture is conchoidal; its lustre is vitreous. When pierced with a metallic point it yields radiating cracks with splinters. It has an agreeable balsamic odour, but loses its smell on storing. The heat of the hand disengages a faint smell. It softens at 75°, is pasty at 100°, and fluid at 150° C. It only adheres but feebly to the hand. It is easily broken up between the teeth, and the particles only agglutinate together but very slightly. It has a feeble balsamic flavour.

(2) *Sal Dammar*, a product of different species of sal tree, *Shorea* [*Vatica*], growing in British India, Borneo and Sumatra, is found in the market in brittle, pale, cream-coloured, striated, stalactitic pieces presenting the appearance as if each stream of resin had coagulated and solidified before the secretion and deposition of the next. Its density is 1·097 to 1·123, it melts easily, dissolves partially in alcohol and almost completely in ether, whilst it is perfectly soluble in spirits of turpentine and fixed oils, it is more freely soluble in benzol.

(3) *Black Dammar, specific gravity* 1·090, is the produce principally of *Canarium strictum*, indigenous to, and cultivated in, British India. The tree is killed in the hot season by making a number of incisions in the bark in the lower part of the stem and then setting fire to the tree below the incisions, and then leaving it for two years before collecting the resin. Black dammar occurs in big stalactitic lumps, opaque when viewed by reflected light in the case of thin pieces, but translucent and deep reddish brown by transmitted light. Its structure is homogeneous, its lustre vitreous. Like ordinary dammar it is freely soluble in spirits of turpentine. It is insoluble in cold alcohol, but dissolves in hot alcohol to a certain

extent on the addition of camphor. It is used in India for making varnishes and bottling wax, and in the caulking of ships, but is said to be scarcely better than common rosin.

(4) *Rock Dammar* is the product of two species of *Hopea*—the *Hopea odorata* of Rangoon, etc., and the *Hopea micrantha* of Sumatra, etc. The *odorata* resin comes to market in straw-coloured to colourless nodules about the size of walnuts, with a lustrous resinous fracture, and altogether very much like ordinary East India dammar, equal to it in every respect, and even rather superior to it in point of hardness. It is soluble in spirits of turpentine and benzol, yielding a clear, bright, quick-drying uniform varnish. The resin of *Hopea micrantha* scarcely differs from the above, but is rather darker in colour, and not so brittle.

Composition.—Dammar may be separated into two resins, one of which is insoluble in cold alcohol. The insoluble portion varies from 13 to 16·8 per cent., and the soluble portion from 83 to 86·7 per cent. It contains traces of gum (0·1 per cent.) and of mineral matter (0·2 per cent.).

When submitted to destructive distillation it yields 78 per cent. of an oil very much resembling ordinary rosin oil.

Solvents.—Dammar is more easily dissolved than copal. It does not dissolve completely in hot nor in cold alcohol, but it dissolves easily in boiling alcohol, the solution taking an acid reaction. It is not completely dissolved by chloroform, benzol, coal tar naphtha, rosin oil, or fixed oils even with the aid of heat.

Dammar dissolves completely in spirits of turpentine, both in the hot and the cold, yielding a cloudy liquid which clarifies completely on the addition of a little absolute alcohol, but the solution becomes several shades deeper in colour.

The best solvents for dammar are acetone, or a mixture of ether and absolute alcohol.

Saponification.—Heated with potash ley, dammar only dissolves very partially, but if the ley be added to a solution of dammar in spirits of turpentine, and if this solution be kept boiling until the whole of the spirit be boiled off, complete solution is effected. This alkaline solution yields, by the addition of solutions of several metallic salts, precipitates of metallic resinates which, after drying, dissolve partially in ether, and under this form find an interesting use in varnish-making.

Hardness.—Dammar is very soft and is ranked below the softest Manilla copals; it is simply a little harder than rosin.

Adulterations.—In buying care should be taken to select parcels made up of large colourless nodules as free from dust as possible; the latter is apt to be sophisticated with rosin, which may be detected by the increase of the solubility of the sample in alcohol and the higher acid nature.

4. MASTIC.

History.—Mastic was well known to the ancient philosophers. Galen, 131-210 A.D., physician to Marcus Aurelius, the elder Pliny, 23-79 A.D., and Dioscorides, Patriarch of Alexandria (died 454 A.D.), make mention of it in their writings.

Origin.—The mastic of commerce is secreted by the *Pistachio lentiscius*, a shrubby tree indigenous to the Levant and the Greek Archipelago, but cultivated chiefly in the island of Chios. Each tree yields annually 10 to 12 lbs. The greater part of the annual production is sent as tribute to the Turkish Government and the rest sent to market.

Commercial Varieties.—There are two kinds on the market, which only differ in the mode pursued in collecting the resin. *Mastic in tears*, or officinal mastic, is produced by making slight vertical incisions on the trunk and branches of the tree and collecting the resin which adheres to the bark in the form of small rounded tears, whilst the larger drops, falling upon stones arranged on the ground to prevent the resin from being soiled, constitute mastic in bulk, or common mastic.

Mastic in tears or officinal mastic consists of rounded, elongated tears from $\frac{1}{5}$ to $\frac{1}{2}$ inch in length, either spherical or flattened, and irregular according to their size. They are covered with a coating of a whitish dust. When freed from this coat they are brilliant and transparent. Their fracture is vitreous, and their colour when recently collected is greenish-white, soon changing to yellowish-white. This coloration, according to Fluckiger, is due to chlorophyll. At the ordinary temperature mastic is very hard. Its fresh fracture is conchoidal and the lustre glistening. It has a feeble balsamic odour, its taste is aromatic, slightly bitter but rather pleasant. It softens at 99° and melts at 103° C., giving off an agreeable odour, but the melting-point of mastic stored for a long time rises as high as 120° C. It breaks between the teeth, then softens whilst the pieces agglutinate together, a property which distinguishes it from sandarach, which it somewhat resembles in appearance. The density varies between 1·04 and 1·07.

Composition.—Mastic is composed of two resins, one of which is soluble in alcohol with an acid reaction, *mastic acid* with the formula according to Johnston of $C_{40}H_{64}O_4$, whilst the other, *masticin* $C_{40}H_{62}O_2$, is insoluble therein, but soluble in ether and essential oils. The weight of the latter amounts to from 8 to 20 per cent. of the weight of the mastic. There are in addition to these traces of an essential oil in freshly collected resin.

Solubility.—Mastic only dissolves partially in ordinary solvents; alcohol of 95 per cent. strength dissolves about 90 per cent., and bisulphide of carbon 95 per cent. On the other hand, it dissolves completely in ether, *absolute* alcohol, amylic alcohol, and also, but with the aid of heat, in spirits of turpentine and acetone, but it is insoluble in petroleum spirit.

The portion of mastic which is soluble in alcohol is precipitated from its alcoholic solution by chlorine as a viscous elastic mass, which then behaves in every way like the portion insoluble in alcohol. The latter is white and very viscous, but by desiccation and fusion it becomes transparent and yellowish like mastic, and can then be pulverised, but so long as it is in this condition it only dissolves in absolute alcohol. It may, however, be rendered soluble in dilute alcohol if left a rather long time in contact with it in this pulverulent condition.

Hardness.—Mastic is ranged in regard to hardness between dammar and sandarach.

Adulterations.—Mastic is but seldom adulterated with other resins, with the exception of sandarach, but the tears of the latter are more elongated, and it pulverises between the teeth without softening; moreover, sandarach is completely soluble in alcohol, and only partially so in ether and spirits of turpentine.

It is adulterated more frequently with certain gums analogous to gum-arabic from the *Antractylis gummifera* and the *Echinops tiscosus*, but this fraud is easily detected by treating the resin with water, which dissolves out the gum and separates it from the resin in the form of a jelly.[1]

It has on occasion been found to contain common salt, which is easily recognised under the microscope or by the usual chemical tests. Other impurities are soil, sand, *débris* of leaves, bark, etc.

Selection.—Mastic should be chosen in the form of large tears free from dust and other impurities.

5. SANDARACH.

History.—Sandarach was known in ancient times. The Arabs used it against diarrhœa and to lull the pain of hæmorrhoids. The Chinese used one kind, *C. sinensis*, as a stimulant in the treatment of ulcers, to promote the growth of flesh, as

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[1] This mucilaginous solution may be reprecipitated by alcohol. Distinction between a true gum and a true resin: a resin is dissolved by alcohol and reprecipitated by water; a true gum is dissolved by water and reprecipitated by alcohol.

a deodoriser, and to preserve clothes from the attack of insects. In Europe at the present day it is used very little in medicine.

Origin (*Mogadore*).—Sandarach, also erroneously known as gum juniper, is a resin which runs naturally from the bark of the *Callitris quadrivalvis* (Rich.), *Thuya articulata* (Desf.), a small tree met with in the north-west of Africa, especially in the neighbourhood of Mount Atlas. The yield is increased by making incisions on the trunk near the bottom analogous to those used in obtaining mastic. As it exudes it has obviously high refractive power, and in appearance much resembles ordinary freshly exuded turpentine resin in taste, smell and general appearance. In this condition it is very irritating to a cut or wound. It is shipped to this country from Mogadore.

Origin (*Australian*).—In Australia several species of *Callitris* also yield sandarach, and, according to Maiden, given similar circumstances in regard to size and age of tree, season of flow, climatic conditions, etc., the sandarachs from all the species are precisely similar in chemical and physical properties. Conversely it follows that if two specimens of sandarach are of different qualities the explanation is to be found in the circumstances above enumerated. The best season to collect sandarach or to bleed trees in a particular district is only to be learned by experience.[1]

Commercial Varieties.—(1) *Choice Sandarach*, which is in tears or small thin rods of not more than $\frac{1}{5}$ of an inch in thickness and 1 inch in length, is transparent pale yellow and covered with a whitish dust.

(2) *Common Sandarach*, in deeper coloured tears, going as far as brown with a slight reddish tinge, less transparent and often impure.

(3) *Australian Sandarach*, recently put on the London market, consists of much larger but non-transparent tears.

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[1] The timber of some of the species withstands the ravages of white ants (*Termites*).

According to Mr. Clark, the Australian resin is very similar to Mogadore, and a very average sample of the former might, according to that authority, if properly washed, etc., with a weak solution of potash, equal the finest picked Mogadore sandarach selling at the date of Mr. Clark's report (October, 1894) at 70s. per cwt.

There can be no doubt but that Australian sandarach has not hitherto at the hands of varnish makers received the attention which its intrinsic merits entitle it to, and which have only to be known to be appreciated.

Properties.—Sandarach is hard but brittle, the slightly conchoidal arched fracture has a glistening lustre when fresh, it is easily crushed under the teeth, becoming reduced to a finer and a finer powder, the particles of which do not agglutinate together as in the case of mastic. It has a faint smell of turpentine and a slightly bitter balsamic taste. It commences to soften at 100° C., melts at 135° C., catching fire and at the same time emitting a characteristic pleasant smell. Its density is 1·092 according to Brisson, 1·05 according to Pfaff, and 1·066 according to Fluckiger. Old samples have a mealy appearance, but this is merely superficial, and has been accounted for as follows :—

Dust.—The surface of the tears appears to be covered more or less with powder, but this is not due to the mutual attrition of the fragments one against another, but to the unequal contraction of the resin in drying, resulting in a mass of fissures that form, as in the case of several kinds of copal, facets that gradually separate from the mass and constitute the powder of many authors. In support of this view it has been pointed out that resins of the *sandarach* class are nearly white on the trees, showing that the appearance is brought about by exposure to the alternations of temperature.

Composition.—Sandarach is composed of three resins which may be separated, but they differ so slightly from the point of view of their behaviour with the usual solvents that their study presents no interesting feature.

Solubility.—Sandarach is soluble in ether, and it dissolves completely in hot absolute alcohol; it also dissolves in amylic alcohol and in acetone. It is partially soluble in spirits of turpentine, in chloroform, and bisulphide of carbon, and only a very small quantity dissolves in benzol and petroleum ether.

Hardness.—Sandarach is equal in hardness to kauri, but superior in this respect to that which it scratches.

Adulterations.—It rather serves for the adulteration of other resins than for being itself adulterated. It is better, however, to make sure that it has not been sophisticated with German sandarach, gum juniper, a resin which is exuded exclusively by old juniper bushes. On heating it gives off a characteristic odour, by which it is easily recognised.

6. ELEMI.

Origin.—The resin elemi is produced by several species of *Terebinthaceæ*, of the genera *Icica elaphrium* and *Canarium.* It comes principally from Manilla, although it is also found in other countries—Brazil, Mexico, the East Indies, etc.

Commercial Varieties.—The principal kinds used in varnish-making are:—

(1) *Manilla Elemi* is a product of a *Canarium* indigenous to the Philippine Isles, especially Luzon, known under the name of the bread tree. It is met with in soft mass of a granular structure containing disseminated crystals. The colour varies from greenish white to yellow. It often contains impurities. Its odour is pleasant, recalling that of essence of lemon and terebenthene. It is exported to this country from Manilla.

(2) *Brazilian Elemi* is produced by the *Icica cariba* (D.G.) or *Amyris ambrosiaca.* It is met with in very irregular pieces, which, although soft in the beginning, become glued

together, and harden in the course of time. They are covered with vegetable *débris*, are yellow in colour, with portions of a greenish yellow.

(3) *Mexican Elemi*, secreted by the *Elaphrium elemiferum* (Royl.), is met with in greasy rounded masses. It also is of a blonde yellow with greenish patches and mixed with vegetable *débris*.

(4) *Elemi in cakes*, the product of the *Icica carana* (Humboldt) of New Grenada, also called *Caraque*, comes to market in the form of masses of 1 lb. to 2¼ lbs. in weight. The colour is white or yellowish green.

Properties.—Elemi is a balsam; when fresh its consistency is soft, but it hardens on storing, and can then be ground easily. It has a crystalline structure, which is especially pronounced in the case of Manilla elemi. Its colour varies from greenish white to yellow. The resin, hardened by keeping, possesses a deep citron yellow colour. The odour is fragrant, recalling that of fennel, carraway, dill and anise, but drawing nearer to that of turpentine as the resin ages and becomes harder.

Its behaviour with heat is characteristic: it softens at 80° C., it becomes partially fluid at 100° C., and completely so at 200° C. Its density varies between 1·018 and 1·083.

Even the better kinds of elemi are dirty, through the presence of vegetable *débris*, but may be very easily purified by melting the resin and filtering through a coarse cloth.

Composition.—Fresh elemi consists of 10 to 12·5 per cent. of an essential oil, and variable proportions of both an amorphous and crystalline resin. The latter is of great importance in identifying and distinguishing elemi. If, in fact, we place a little elemi on a glass slide and melt it at a gentle heat, and then examine it under the microscope after cooling, the appearance is but slightly characteristic, but if the substance be moistened with a little alcohol, it becomes rapidly crystalline, often presenting the form of long needles.

Solubility.—Elemi is partially soluble in cold alcohol, but entirely soluble in hot alcohol, ether and spirits of turpentine.

Hardness.—Elemi is a very soft rosin, softer even than ordinary rosin. It may be cut with a knife like steatite soap-stone, a property peculiar to itself alone amongst all the resins.

Adulterations.—Elemi is almost exclusively adulterated with galipot, which has the same consistency, but this sophistication is easily recognised by observing the crystalline form under the microscope, or by the smell of spirits of turpentine which is given off when the resin is heated. The principal use or function of elemi is to diminish the brittleness of, and to impart elasticity to, spirit varnishes.

7. Benzoin.

Benzoin, corrupted in Britain to "Benjamin," has but a relative importance for the varnish-maker, who principally adds it to varnishes on account of its pleasant smell.[1] It is above all used for varnishes to be applied to objects which have eventually to be submitted to a rather high temperature, or as an ingredient of *finishing* polishes for furniture.

Origin.—Benzoin is indigenous to the Indies, Cochin China and the Indian Archipelago. It is the product of the *Benzoin officinale hayne* of the family of the *Styraceæ.* These trees are cultivated with the sole view of producing resin. The oldest trunks yield an inferior product. In Siam the first incisions are made on the tree when seven years old, by which time the trunk has attained a diameter of about 7 inches. The whitish resinous juice which exudes during the next succeeding three years is termed "Head"; it is softer, more aromatic, and contains more yellowish-white tears than that obtained during the next seven to nine years, which is inferior, and termed "Belly". The average annual yield

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[1] Or some other special purpose.

is 3 lbs. The tree is at last cut to pieces and the resin scraped off and collected. This constitutes the "Foot," which is naturally still darker and contaminated with wood scraps and other extraneous matter. The "Head," or first quality, after softening by the heat of the sun, is run into cubical boxes at the coast and shipped to Europe, whilst the inferior qualities find a market in India, Persia, etc. It has been transplanted to Brazil and the Isle of Bourbon.

Commercial Varieties.—These may be divided into three groups.

(1) *Benzoin in Tears* or *Siam Benzoin*, consists of flattened elongated milk-white homogeneous pieces, having a diameter up to an inch, agglutinated together, the mass covered with a brownish opaline crust, white on freshly broken surfaces, but assuming in the course of time a coloration varying from white to brown.

(2) *Amygdaloid Benzoin* or *Sumatra Benzoin* is only differentiated from the preceding by the form of its almond, which, covered with a greyish or brown crust, presents a reddish yellow colour outside and white inside. Its smell is not so strong as that of Siam benzoin.

(3) *Common Benzoin* or *Calcutta Benzoin.*—This variety, which is prepared in Calcutta, comes to market in large blocks, and contains the two preceding kinds amalgamated together into a resinous mass.

Properties.—Benzoin is a balsam with a peculiar, agreeable odour, intensified by heat. Some kinds—particularly those from Singapore—have a smell quite analogous to that of vanilla. Its taste is at first sweetish and then becomes acrid. It melts between 80° and 95° C., but in the case of Siam benzoin the melting-point may be as low as 75° C. Heated it disengages benzoic acid. Distilled with water it yields a small quantity of a pleasant smelling volatile oil. Its density varies between 1·063 and 1·092.

Composition.—Benzoin contains three different resins, some authorities say five; moreover it contains a small quantity of essential oil and a mixture of benzoic acid and cinnamic acid or cinnamic acid alone. Kolbe and Lauteman met with some specimens of benzoin which did not contain benzoic acid. From one sample of the best quality brought from Sumatra they extracted an acid altogether different from benzoic acid, fusible in hot water, and giving oil of bitter almonds when oxidised by potassium permanganate, and supposed to be identical with alpha-toluylic acid which Müller and Strecker obtained by heating vulpinic acid with baryta.

Analysis of Benzoin (Ludz).

	Per cent.
Cinnamate of benzo-resinol	5
Cinnamate of resinotannol	64
Cinnamic acid	30
Vanillin	trace

Solubility.—Benzoin is entirely soluble in alcohol; it dissolves completely in ether and in fixed and essential oils.

Hardness.—Benzoin yields a product, the hardness and fracture of which is very similar to that of sandarach.

Adulterations.—Benzoin is often mixed with impurities, from which it may be separated by dissolving it in alcohol. Further, it may be freed from the greater part of its benzoic acid, but then it only retains a very feeble smell, and on breaking its fracture does not show any white portions.

Selection.—Good benzoin always shows an abundance of milk-white almonds.

8. TURPENTINES.

The turpentines employed in varnish-making are:—

1. Common Turpentine.
2. Venice Turpentine.
3. Boiled Turpentine.
4. Burgundy Pitch.
5. Rosin.

(1) COMMON TURPENTINE.

Origin.—Turpentine is the product of several species of pines in the Landes and in the neighbourhood of Bordeaux: it is extracted from the *Maritime pine*, *Pinus pinaster*, *Solander pinus*, *Maritime* (Lam.). In Germany it is obtained from the *Pinus austriaca* (L.), *Sylvestris* (L.), and *Rotundata* (Link.). Finally, in America it is obtained from the *Pinus toeda* (L.) and *Australis* (Mich.).

General Characteristics.—Turpentine varies much in *colour* and *consistency*. The prevailing colour is yellowish-white often bordering on brown. The odour of each is characteristic and pervades the atmosphere in the neighbourhood of the pine forests and is said to be very remedial in phthisis. French turpentine has an unpleasant smell, American a pleasant lemon odour.

N.B.—Spirits of turpentine is generally but erroneously known as *turpentine*.

Commercial Varieties.—(1) *Bordeaux Turpentine* is a balsam of the consistency of thick honey which separates in time into two layers, the one transparent and semi-fluid, and the other resinous and of a crystalline aspect.

(2) *German Turpentine* has almost the same appearance as the preceding.

(3) *American* or *Boston Turpentine* is yellowish white, of the consistency of honey, but does not separate in time into two layers.

Properties.—When examined under the microscope common turpentine generally shows crystals of sylvic acid; the odour is strong and hardly pleasant, and its taste is bitter and acrid. The melting-point, according to Andés, is, on the average, 130° C., and the density about 0·856.

Composition.—Besides sylvic acid, turpentine contains 15 to 30 per cent. of spirits of turpentine, which is disengaged when the turpentine is distilled with water.

Solubility.—It completely dissolves in alcohol, ether, bisulphide of carbon, and in fixed and essential oils.

Hardness.—Common turpentine is a more or less thick balsam, and is more solid the less spirits of turpentine there may be present.

Adulterations.—As the price is lower than that of other resins, far from being adulterated, on the contrary, it is itself used for adulterating purposes.

(2) VENICE TURPENTINE.

History.—This oleo-resin was known to Pliny, who describes it as follows: "*Plusculum huic erumpit liquoris malleo colore atque lentiore nunquam durascentis*" (This resin which is honey coloured issues slowly from the larch tree, but never becomes dry). Again, Dioscorides states: "There are liquid resins also from the pine and pitch tree. These are brought from France and Etruria. They vary in colour, as some are like oil, others white, and some like honey, as the larch." Moreover, the *atramentum* of Pliny "which" (he states) "was applied so thinly over the picture when finished that it brought out the colours in all their brilliancy and preserved them from dust and dirt". "*Quod absoluta opera atramento illinebat ita tenui ut idipsum repercussa claritatis colorum excitaret custodietque a pulvere et sordibus.*"

Origin.—It is obtained from the common larch—*Pinus larix* (L.), *Larix decidua* (Mill.)—which is grown for resin-producing purposes in the Tyrol, Piedmont, and in France in the environs of Briançon.

Commercial Varieties.—There is only one kind, known under the name of Venice turpentine, Briançon turpentine, turpentine in tears.

Properties.—It is thick, and varies in colour from bright yellow to greenish yellow. It is nebulous (cloudy), but when freed from the water which it contains it is bright and clear. Its smell recalls that of spirits of turpentine and essence of lemon. Its taste is bitter and aromatic.

It does not, like other turpentines, separate into two layers with time, and it does not in time harden on the surface, nor become crumbly when kept for a long time in a cold place. It shows no crystals when viewed under the microscope. It becomes fluid at a low heat, and then runs easily.

Composition.—This oleo-resin contains 15 to 25 per cent. of essential oil.

Solubility.—It is soluble in alcohol, ether, and in essential and fixed oils. It melts at 130° C. Its specific gravity is 0·856.

Hardness.—It is thick like honey, and does not in the course of time become covered with a dry, brittle pellicle.

Adulterations.—It is frequently adulterated with common turpentine. This sophistication is detected by heating gently so as to drive off the volatile oil, and then moistening the residue with alcohol; if this residue presents a crystalline appearance common turpentine is present. The smell also serves as a guide.

(3) Boiled Turpentine.

Boiled turpentine, or *résine à l'eau*, consists of the residue obtained after distillation of the spirits of turpentine from common turpentine, and calls for no special comment further than that it always contains a certain quantity of water—a fact of some significance.

(4) Burgundy Pitch.

Origin.—Burgundy pitch is extracted from a species of pine—the *Pinus abies* of Linnæus, the *Abies excelser* of Lamarck, which is very abundant in the Vosges and the Alps.

Commercial Varieties.—There is only one kind, which is known under the name of Burgundy pitch, Vosges pitch, and yellow pitch.

Properties.—It is opaque, yellowish brown, solid, rather brittle ; when cold it softens easily under the action of heat, even that of the hand. It has a strong odour of spirits of turpentine and a sweet aromatic taste.

Composition, etc.—It is prepared by melting the solid turpentine collected from the trees with water. It therefore contains a larger quantity of spirits of turpentine than the residue obtained by heating the turpentine with a fire heat rosin and a larger quantity of water, which takes away its transparency and brilliancy. When heated it cracks or spurts on account of the proportion of water which it contains.

It is important to determine the amount of water in this resin, which is done by pulverising it very finely and placing it over concentrated sulphuric acid in a desiccator, until of constant weight.

Solubility.—It is soluble in ether, spirits of turpentine, fixed oils, but only partially soluble in alcohol.

Hardness.—It is brittle at ordinary temperatures but easily softened.

(5) Canada Balsam.

Origin.—Canada balsam is simply an oleo-resin exuded from the Canadian balsam pine, *Pinus balsamea*, indigenous to North America. This tree frequents wet, bleak, hilly districts, and grows to a height of 30 to 40 feet with a diameter of 8 or 9 inches. Its timber, as may be inferred from its habitat, is of little value, and the tree is grown solely for the sake of the balsam which exudes from it. The oleo-resin segregates into vesicles on the exterior of the bark, and is collected during the summer months. Each tree exudes annually about 1 lb. of resin.

Properties.—Canada balsam when fresh is a transparent, straw coloured, viscous fluid which soon hardens on exposure to the air. The dried film retains its transparency to a very great extent. Its smell is strongly and characteristically aromatic though not unpleasant. Its density is said to be ·998 at 15° C.

Composition.—Bonastre obtained the following results:—

Analysis of Canada Balsam.

	Per cent.
Essential oil - - - - - -	18·6
Resin (soluble in alcohol) - - - -	40·0
Resin (difficultly soluble) - - - - - -	33·4
Elastic resin - - - - - - -	4·0
Extractive and salts - - - - -	4·0
	100·0

Solubility.—It is soluble in ether, benzol, chloroform and warm amylic alcohol. Benzol is most esteemed as a solvent. When it dries from solution in the latter or chloroform it leaves a perfectly transparent, faintly yellow and beautifully lustrous film. Its index of refraction is 1·532. It is dextro-rotatory in use. It is principally for cementing lenses and mounting microscopic objects.

Points to be observed in buying.—There is sometimes substituted for this product a mixture of spirits of turpentine and galipot. The latter is Bordeaux turpentine, which has run from the tree during winter, and which, poor in spirits, has dried on the trunk of the tree. This substitute is detected by its smell of common turpentine, its taste, and especially by its complete solubility in alcohol.

9. ROSIN (COLOPHONY).

Origin.—Rosin is the residue obtained from the steam distillation of turpentine, and principally of Bordeaux turpentine and American. The essential oil which they contain is carried over by this distillation and condensed apart. The water is also driven off. This process is fully described in the chapter on "Solvents," p. 90.

Commercial Varieties—(1) *Bordeaux Rosin* or *French Rosin*, consists of brittle, friable masses, varying in colour from bright yellow to deep brown, according as it has been submitted to a greater or less heat.

(2) *American Rosin* is transparent, varies in colour from window-glass through pale amber to dark brown. It possesses a peculiar fragrant smell not possessed by the French variety. As this smell is retained by yellow soap, into the composition of which it enters, it is highly prized by British soapmakers; so much so that during the American Civil War its price rose above that of tallow, French rosin not being available on account not only of its bad smell, but also of the limited production which barely meets the wants of their own home consumption. When American rosin enters into the composition of an oil or spirit varnish it modifies the smell of the solvents. These, however, having a distinct smell of their own—more or less pungent—the smell of the rosin is not so perceptible as in the case of soap.

Properties.—Rosin is hard and friable at the ordinary temperature. It softens at 80° C., and melts between 90° and 100° C. Density, 1·045 to 1·100.

Composition.—Rosin is never completely free from spirits of turpentine, the better varieties still retaining 1 to 2 per cent.

Refining.—Several processes have been indicated for refining rosin. In view of the numerous attempts which have been made to refine resin so as to produce therefrom a good drying varnish, the following processes may be pointed out without expressing but a limited amount of belief in their efficacy:—

(1) It has been proposed to pass a current of chlorine through melted rosin; the mass is acidified with sulphuric acid, washed with boiling water, and finally with hot water containing nitric acid.

(2) The rosin is heated with a solution of common salt; it is then brought to the boil for a few minutes after adding a solution of chromic acid, or a solution of bichromate of potash and sulphuric acid, and the operation finished by washing with water rendered slightly ammoniacal.[1]

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[1] Both nascent chlorine and oxygen would appear to be developed by this mixture.

(3) Another process consists in heating rosin with a mixture of chalk, black oxide of manganese, bichromate of potash, and filtration through sand.

(4) Or the rosin is heated with zinc dust, with or without the addition of bisulphate of soda.

(5) Attempts have also been made to use chloride of zinc and sulphuric acid at a high temperature.

(6) The best process would appear to be to previously filter the melted rosin, then to heat it to 150° C. with 5 per cent. of zinc chloride for one or two hours, and then to add about 12 per cent. of bichromate of potash in powder. The whole is filtered after allowing the temperature to fall to 100° C.

(7) Another process of purification consists in the use of sulphuric acid under pressure and at a high temperature. The process is conducted in an iron autoclave furnished with a steam jacket heated with superheated steam and capable of resisting a pressure of 12 lbs. About 2 cwt. of rosin are run into the autoclave, and heat applied until the whole is melted, and when the pressure has reached 9 lbs. the sulphuric acid is introduced. The whole is heated to 100° C. for an hour, then allowed to cool, and the product washed with boiling water.

(8) Rosin may be obtained as colourless and transparent as glass by distilling it with superheated steam. (Hunt & Pochin's patent.)

Solubility.—Rosin does not dissolve in water, but dissolves easily and completely in alcohol of 95 per cent. strength. It is very soluble in benzol, bisulphide of carbon, ether and chloroform, coal tar, petroleum and shale naphthas, and its solutions in these solvents may be thinned down with gasolene. It is soluble also in spirits of turpentine and in fixed oils.

Saponification.—Rosin dissolves in solutions of both caustic and carbonated alkali, 1000 parts of rosin requiring on an average about 170 parts of pure caustic potash for complete saponification. Its solution fluoresces or "blooms". See also under "Resinates," p. 211, for further details as to its solubility in alkalies.

Hardness.—Rosin is harder than elemi. It has been pointed out that this hardness increases when it is heated in an atmosphere of carbonic acid; when it is afterwards broken it does not crumble as before, and its fracture recalls that of copal.

(1) *Hardening Rosin.*—The following process has also been proposed for obtaining products of greater hardness than ordinary rosin. The resin is heated from 180° to 210° C., and 8 to 10 per cent. of glycerine stirred in with constant stirring. By working in a vacuum a temperature of 310° C. may be used; there is afterwards added 3 per cent. of pulverised lime, and the whole heated until the mass becomes homogeneous. Working in this manner, according to Dr. E. Schaal, a very hard product—a rosin ester—is obtained after cooling.

(2) It has also been proposed to heat to a temperature of 180° to 240° C. a mixture of 4 parts of cane sugar with 5 to 6 parts of glycerine and 100 parts of rosin. The whole is then heated to 208° C. after the addition of 3 parts of linseed oil and three parts of lime until the mass becomes homogeneous.

But it does not appear that this hardened rosin, or rosin "ester," can be altogether substituted for the hard copals, even for those of inferior quality.

Adulterations.—The only point to be attended to is the amount of spirits of turpentine, water and grit present. Austrian rosin should not be bought, it is too soft and "tacky".

10. SHELLAC.

Origin.—Shellac comes from the East Indies, and is produced by an insect, the *lac cochineal coccus lacca*, which lives upon different trees, but principally on the *Ficus relegiosa L.*, *Ficus indice L.*, *Rhamnus jujuta L.*, *Croton lacciferum.* The insects are enshrouded by the resinous juices which run from the surfaces punctured by the insect These juices dry upon them, and principally upon the female insects, which are far more abundant than the males, the proportion being 1 male to 5000 females. Inside the burrowings thus formed the larvæ are developed which consume the juice, and which after development pierce the envelope to escape. These burrowings are coloured reddish brown and constitute the shellac, which consists of a colouring matter, wax and resin. The best shellac is that which is collected previous to the escape of the insects.

Commercial Varieties.—Shellac is met with under six forms:—

(1) *Stick Lac*, or *Crude Lac*, which adheres to the branches upon which it has been produced, is met with as an irregular transparent crust of a dark red colour with a lustrous fracture.

(2) *Lac Dye* and *Lac Lake* are made by passing the preceding product through a mill and sifting the ground lac, returning to the crusher everything that does not pass through the sieve. The sifted product is stirred in a revolving cylinder with a weak solution of caustic soda, which dissolves the colouring matter; the alkaline solution is decanted, and a solution of alum added which precipitates this colouring matter and the fraction of shellac dissolved by the alkaline lye.

The precipitate is pressed into cakes which are dried in the sun. A product is thus obtained which contains 50 per cent. of colouring matter.

(3) *Shellac properly so called* is obtained from the resin purified by the preceding operation. This resin is melted and run into hot flat moulds, from whence it is taken by the workmen dipping in a piece of bark and spread over a hollow cylinder of galvanised iron filled with tepid water. The layer is smoothed by passing a leaf over the surface, and soon solidifies, assuming a fine shagreen texture; the thicker

parts are separated and remelted, whilst those portions, which are sufficiently thin, are suspended in an airy place and afterwards exported in the form of shellac.

(4) *Seed Lac* consists of the grains directly detached from the branches.

(5) *Button Shellac* is obtained by roughly breaking the crude lac and stirring it for several hours with hot water, which separates a portion of the colouring matter. The resinous mass is again treated with hot water, melted and run into buttons.

(6) *Shellac Rods* are obtained by drawing out the melted shellac into rods.

Properties.—Shellac is a brittle substance varying in colour from reddish brown to a golden yellow, according to the amount of colouring matter which has been removed from it by previous treatment. When heated it becomes soft and gives off a peculiar, pleasant smell. If the heat be sufficient it melts completely and catches fire.

Composition.—The following analyses are due to Hatchett :—

	Crude. Per cent.	Button. Per cent.
Resin - - - - - -	68	85·5
Colouring matter - - - -	10	2·5
Wax - - - - - - -	6	4·5
Nitrogenous matter - - - -	5·5	2·0
Impurities - - - - -	10·5	2·5

The wax consists of a mixture of several myricic ethers, with more than 50 per cent. of free myricic alcohol and a small quantity of free or combined cerylic alcohol. But the proportions of these different ingredients varies with the treatment which the crude lac has undergone; lac dye contains as much as 50 per cent. colouring matter, whilst orange shellac only contains 0·05 per cent. for 91 per cent. of resin.

Solubility.—Shellac is only very partially soluble in ether, which only dissolves 6 per cent. of the wax ; the same remark applies to chloroform, which only dissolves 10 per cent. of the wax, and also to bisulphide of carbon. For the same reason it only dissolves partially in cold alcohol, although it gives a clear solution with hot alcohol; the solution becomes cloudy as it cools from separated wax. It does not dissolve in oils. Shellac dissolves easily in solutions of caustic or carbonated alkalies as well as in a solution of borax; it is precipitated from these solutions by acids.

The colouring principle is soluble in water and also in dilute sulphuric or hydrochloric acids.

Hardness.—Shellac although tough is not at all hard; it is particularly brittle and friable when rosin is present to any extent.

Adulteration of Shellac and its detection.—Rosin is the most general adulterant, and this fraud is very difficult to detect owing to the behaviour of both substances with the usual solvents being very similar.

Ether in which rosin is completely soluble may, however, be employed for this purpose, or petroleum ether which only dissolves 1 to 2 per cent. of pure gum lac and 3 to 15 per cent. of seed or stick lac, whilst rosin is completely soluble.[1]

A process based on the difference in density of the two resins has been given by Wiesner. It consists in making a solution of common salt or sugar with a density of 1·08 to 1·09 at 15° C., in which resin floats whilst shellac falls to the bottom. The finely pulverised sample is briskly shaken with this solution, and after being allowed to stand for some time the liquid is decanted along with the floating resin. After sufficient washings the resin is weighed, and the proportion in which the mixture was made is thereby ascertained.

BLEACHING OF SHELLAC.

As the colour of shellac is often objectionable, attempts have been made to bleach it. Chlorine may be used for the

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[1] The merest tyro may detect rosin in orange shellac by crushing it between the fingers and the palm of the hand. Rosin also increases the acid value.

purpose. The shellac is dissolved in a weak alkaline lye and a current of chlorine passed through the solution. But chlorine acts very energetically, and often so modifies the resinous matter that it becomes altogether altered and unfit for use owing to its becoming very difficultly soluble in alcohol. Moreover, great trouble is experienced in removing all the chlorine from the bleached shellac, even after repeated washing with water. The former afterwards acts injuriously when the resin is extended into a varnish, especially upon metals, and to such an extent as to exclude it from employment thereon.

(1) *Willstein's Process.*—500 parts of chloride of lime are mixed with 2000 parts of water, and a solution of 500 parts of carbonate of potash in 1500 parts of water is run in so long as a precipitate is formed. Into the filtered liquid there is run, a little at a time, a solution of 500 parts of orange shellac in 2000 parts of alcohol of 90 per cent. strength. After the whole has been well stirred, it is allowed to stand, preferably in the sunlight. After half an hour to an hour the liquid containing the resin is run in a thin stream into dilute hydrochloric acid, 1 part of acid in 5 parts of water. As soon as the resin is completely precipitated it is washed with water until free from acid. It is then churned with hot water until the latter is no longer coloured; finally, the hot resin is run into sticks which are generally piled together. If the action of the chlorine has been too energetic, and if the shellac has been so altered as to become insoluble in alcohol, this may be remedied by breaking the shellac into small pieces and immersing these for twenty-four hours in ether, where it swells and is afterwards easily dissolved in alcohol.

Sauerwein has suggested the addition of sulphite of soda to the resin precipitated by hydrochloric acid.

(2) *Another Method.*—Willstein's method is costly owing to the use of alcohol. The following method may be

used instead: 40 parts of shellac are heated with a solution of 10 parts of carbonate of soda in 150 parts of water until complete solution is effected. The liquid is allowed to clarify by standing and then decanted, after which there is added to it a filtered solution of 40 parts of chloride of lime in 40 parts of water, leaving the whole to stand from twenty-four to forty-eight hours, and then precipitating the resin by hydrochloric acid as in the preceding method.

(3) *Alkaline Method without Chlorine.*—In the following process an attempt has been made to altogether suppress the use of chloride of lime. The shellac is added in small quantities to a dilute solution of boiling soda by taking care not to make a fresh addition until the previous lot has been dissolved, stopping short when there is a slight excess of free soda.

When all is dissolved, the whole is boiled for a few moments with constant stirring and then allowed to cool. The wax which solidifies on the surface is removed and the liquid filtered, if need be, so as to obtain a clear solution, which is treated with hydrochloric acid as before. But this process yields a product which still contains an appreciable amount of colouring matter.

Care has to be taken not to prolong the boiling with alkaline lye even although very dilute, for Benedikt and Ehrlich have shown that the shellac would become converted into a substance of a balsamic consistency which has a similar centesimal composition as the primitive resin, and which they have named liquid gum lac.

(4) *Elsner's Method.*—Elsner has proposed the use of animal charcoal, which bleaches the resin without altering it. He agitates an alcoholic solution of shellac with coarse-grained animal charcoal which has been previously washed with acid, then with water, and dried. A quantity of

animal charcoal is added to the alcoholic solution sufficient to make therewith a rather fluid paste and the whole exposed to sunlight for several days, taking care, however, that the temperature is not too high. When it is ascertained by filtering a small fraction of the liquid that the solution is sufficiently bleached, the whole is thrown on a filter and the alcoholic filtrate evaporated. This process yields a remarkable product, but it can only be available in certain exceptional cases because it is too costly. The charcoal, moreover, retains too large a portion of the solution and has to be washed with alcohol, which still further increases the cost.

(5) *Sulphurous Acid Method.*—It has been further proposed to dissolve the shellac in alkaline lye and to afterwards neutralise it with a current of sulphurous acid which precipitates the bleached shellac.

11. JAPANESE LAC.

Origin.—The gum lac of Japan or *urushi* is the milky juice of the *Rhus vernicifera*, a tree cultivated in Japan at different altitudes, at Dewa Aizu, Hiroshima and in the environs of Tokio. The largest yield of juice is obtained when the trees are fifteen years old. Two kinds of liquid are obtained from one and the same tree, one of which is called *Ki urushi* and the other *Seshime urushi.*

To obtain the former shallow incisions are made on the stem of each tree, which yields on an average about forty grains of this particular kind. The second variety is obtained by macerating the branches for several months in water and afterwards heating. The seshime urushi thus obtained is only used for the preparation of oil varnishes.

Commercial Varieties.—(1) *Pure Urushi* is a thick greyish liquid, having the appearance of an emulsion.

(2) *Ordinary Urushi.*—This is a mixture of 60 per cent. of pure urushi and 40 per cent. of mokuyiki or wood sap, but

inferior qualities do not contain more than 30 per cent. of pure urushi.

Properties.—Pure urushi is a liquid with a sweet characteristic odour. Its density is 1·002 at 20° C. If exposed to the air in a thin layer it assumes a deep colour and dries to a brilliant translucent varnish, which is very stable and resistant, even under the action of nitric acid.

Composition.—According to M. Ishimatsu, who has published a very complete research upon Japanese lac, the pure urushi of Yoshino contains in 100 parts :—

Matter soluble in alcohol (urushic acid)	85·15
Gum analogous to gum-arabic	3·15
Diastasic matter	2·28
Water and volatile matter	9·10
	99·98

Ordinary urushi has the following composition :—

Soluble in alcohol	58·24
Gum	6·32
Diastasic matter	2·27
Water and volatile matter	33·17
	100·00

Urushic acid, which forms the greater part of urushi, is a deep coloured paste possessing the same characteristic odour. It has a density of ·9851 at 23° C. It resists a temperature of 160° and decomposes slowly at about 200° C. It is not affected by atmospheric influences.

Urushic acid only dries under the influence of the diastasic matter which is coagulated by heat and loses its action about 63° C. It is therefore necessary to work at a comparatively low temperature, about 20° C. In contact with air it is converted into oxyurushic acid—the dried varnish—which corresponds in composition with the formula $C_{14}H_{18}O_3$.

According to M. G. Bertrand, *Comptes Rendus de l'Académie des Sciences*, t. cxviii., p. 1215, the portion soluble in alcohol, which he calls laccol, becomes oxidised as easily in the absence as in the presence of the ferment which he calls laccase, but in the first instance the laccol only yields a viscous resinous mass—soluble in alcohol—which remains tacky for a long time, and not the black insoluble product characteristic of the lac. It is under the alternate action of the oxygen of the air and the diastasic principles that the lacquer is produced.

Solubility.—Urushic acid is soluble in ether, alcohol, benzol, bisulphide of carbon; it does not dissolve so easily in petroleum, and is insoluble in water.

Adulterations.—Japanese lac is adulterated by the addition of mokuyiki or wood sap, and also by the addition of a drying oil, generally linseed oil.

12. CHINESE LAC.

Origin.—Chinese lac, or Chinese varnish, is a natural product which, according to Father d'Incarville, is composed of a gum resin which flows from a tree known to botanists as *Angia sinensis*, but called by the Chinese *Tssi-chou* or varnish tree, which grows in several of the meridional provinces of China. It grows *wild* on the mountains. The diameter of the trunk is often a foot or more. But the tree is also *cultivated* for the sake of its varnish, which it then yields more abundantly and of finer quality. During the summer cuts are made in the bark in the form of a triangle, and a resin exudes of the consistency of liquid pitch. An oblong river shell is inserted in the base of the triangle to receive the resin. The natives collect the varnish into bamboo pails hanging from the loins by scooping it out of the shell with a finger previously moistened with the tongue to prevent the varnish from adhering. Many use a wooden spatula moistened with water. The varnish is stored in barrels, the mouths of which are closed by a sheet of paper made of hemp fibre. The varnish which flows from the wild trees is collected in a very crude way. Cuts are made at different heights on the tree and the varnish is collected at the foot. In the Tonkin section of the Paris Exhibition of 1889 natives might have been seen manipulating this varnish and its application to different objects viewed from first to last. This substance dissolves in the different solvents—ether, alcohol, spirits of turpentine. It can thus be brought to any desired consistency. Macaire Princeps states after investigation that its properties are similar to Venice turpentine or copaiba balsam. Exposed to the air it solidifies, becomes of a red colour, then black, and forms a brilliant coat, susceptible of taking a beautiful polish. The resplendent lustre and durability of the lacquers used by the Chinese in the decoration of articles of vertu, ornaments, etc., have for centuries been the admiration of Western Europe. At first sight their method would appear to be altogether different from our own. But on closer inspection it will be found that the principle of both is identical, the only real difference being in the materials used.

The Chinese use three different kinds of varnish—*Nien-tsi*, *Si-tsi*, and *Kouang-tsi*. The two first are used for black varnish, and the latter, which is the most highly esteemed, as a vehicle for colours. For this purpose it is mixed with a kind of oil called tong-yeou, the drying properties of which have been intensified by exposure to air and sunlight. The most remarkable difference between our varnish and Chinese is the fact that the quicker the former dries the more brittle does the coating become and the greater its tendency to scale off. The Chinese varnish, whilst drying quicker, does not possess this fault. Their method of manufacture is very simple. The varnish is first reduced to half its original bulk by exposure to the sun. It is then mixed with about ¾ oz. of pig's gall and about ½ oz. of Roman alum per lb. of pure varnish. For black varnish they add ivory black and tea oil rendered drying by boiling with arsenious oxide or sulphide. Further, as a vehicle for pigments, the Chinese use a special varnish called *Kao-kin-tsi*, formed by a mixture of equal parts of *Tshao* and of *King-tsi*, made, the first by adding a drying oil to *Kouang-tsi*, and the second by the addition of the same oil to *Si-tsi*, and in both cases a certain quantity of camphor.

The varnish thus made is applied in a very systematic and judicious manner. Thus the varnishes instead of being mixed are applied one above the other and gold bronze strewed between the two layers, thus producing the pretty ground works on the art objects which they export.

The varnish is repeatedly filtered before use, four or five times in succession, so as to obtain a perfectly limpid and homogeneous fluid, which is supposed to be applied in very thin successive and contiguous layers.

The Chinese colour their varnishes as follows:—

Yellow—Orpiment.
Red—Vermilion or safflower.
Violet—Tai-tsi.
Green—A mixture of indigo and orpiment.
White—By incorporating silver leaf in the body of the varnish.

13. ASPHALTUM.

Origin.—Asphaltum belongs to the class of resins which are by some petrologists included amongst minerals. It is met with along the shores of the Dead Sea, in the bituminous earths of the Isle of Trinidad, and in the bitumens of Britain, France and the Tyrol, etc. It is a solid bitumen, consisting of a mixture of solid, liquid or gaseous hydrocarbides. It is usually associated with rock salt in localities where rapid evaporation and concentration by the heat of the sun has taken or still is taking place.

Commercial Varieties.—In the manufacture of varnishes the following sorts are employed:—

(1) *Syrian Asphaltum* or bitumen of Judæa, a deep black sub-

stance by reflected, but yellowish-brown in their sections by transmitted, light, with a strong smell and a glistening fracture. This substance, which constitutes the bulk of the asphaltum of commerce, is generated as a form of crude petroleum by springs in the neighbourhood of the Dead Sea, on the dense water of which it floats, and in course of time, owing to the powerful and concentrated action of the sun's rays, it assumes a greater and greater consistency, and is eventually driven on shore by the wind and collected. During its inspissation the toxic effect of the gases given off has been said to kill birds in their flight across the sea.

(2) *American Asphaltum*, Trinidad pitch, glance pitch, gilsonite, or grahamite, a black product almost without smell.

(3) *Barbadoes Pitch* or *Manjak.*—This mineral is of a bright black colour, and occurs at a very slight depth, sometimes on the surface in beds 1 to 2 feet thick; it generally appears under an angle of about 40 degrees and in the immediate vicinity of rock. It is presumed to be solidified petroleum, which is often seen there exuding from the earth or floating on the water. In its composition this mineral is similiar to the pitch of Trinidad, to the gilsonite of Utah and the Canadian albertite, but it is of a much better quality. The best varieties of "manjak" contain 2 per cent. of water, 70·85 per cent. of volatile organic substances, 26·97 per cent. of fixed carbon, and 0·18 per cent. of mineral matter. A more general grade showed 5 per cent. of water and mineral substance. Trinidad pitch contains 21 to 30 per cent. of water and about 38 per cent. of ash: hence the "manjak" mineral is much richer in natural bitumen. It is used, among other purposes, for the insulation of electrical conduits, for varnish, bituminous concrete, and for fuel, mixed with peat, and possibly might be used as an insulating medium instead of gutta-percha.

Properties.—Asphaltum is met with in compact masses, the colour of which varies from deep black to deep brown; the fracture is conchoidal, appearance greasy. It is not translucid. In thin plates it is very faintly transparent. It is insipid, becomes negatively electrified by friction, and gives off a slight smell of petroleum. It melts between 1·07 and 1·17. The melting-point is 100° C.

Solubility.—Ether dissolves about 70 per cent., spirits of turpentine and fixed oils about 50 per cent., and essential oils about 33 per cent.; absolute alcohol only dissolves about 5 per cent.

Hardness.—Asphaltum has about the same hardness as gypsum.

14. Coal Tar Asphaltum or Coal Tar Pitch.

In varnish-making it is more profitable to employ coal tar pitch than natural asphaltum. It very much resembles the latter and is considerably cheaper. It softens more easily, and is a bitumen varying from a solid to a semi-liquid consistency.

It dissolves more readily in a certain number of solvents than natural asphaltum, but it is indispensable that it should have been previously well freed from heavy oils.

It is but slightly soluble in alcohol; ether dissolves about 50 per cent. It is soluble in chloroform, bisulphide of carbon, benzol, amylic alcohol and acetone. It is slowly soluble in spirits of turpentine, and partially soluble in petroleum ether. Alkalies partially dissolve it.

Manufacture of Coal Tar Pitch.

When coal tar is distilled it may be separated by fractional distillation into :—

1. Light oils.
2. Heavy oils and soft pitch.
3. Anthracene oils and hard pitch.

(1) *The Light Oils* include all those volatile substances which pass over below 200° C., such as benzene, toluene and

their higher homologues, some hydrocarbides of the ethylene series, basic substances and phenols. The ethylenic hydrocarbides and basic substances are eliminated by treatment with concentrated sulphuric acid, and the phenols by washing with alkaline solutions. The oil thus treated is then redistilled, collecting only the products which distil between 80° and 120° C. This liquid—the commercial benzol—contains variable proportions of benzol, boiling at 80° C., and toluene, boiling at 110° C., together with xylenes and other hydrocarbides boiling above 110° C. It can, according to the use to which it is to be put, be used as it is or it may be again fractionally distilled (see "Solvents," p. 90).

(2) *Heavy Oils.*—These distil between 200° and 300° C., and being rich in solid naphthalene solidify to a crystalline mass in cooling. The naphthalene is separated by filtration and pressure, and the heavy oils freed from naphthalene may be used in the manufacture of disinfectants, or it may be used for the manufacture of phenol and carbolic acid.

(3) *Anthracene Oils.*—The residue from the distillation of the heavy oils is known as soft pitch. It consists of a mixture of solid hydrocarbides, anthracene, phenanthrene, pyrene and chrysene, together with several substances the nature of which is but imperfectly understood. By redistilling this oil at a dull red heat anthracene oil, from which anthracene is abstracted, is obtained.

(4) *Soft Pitch*, softening at 40° C. and melting at 60° C., is the residue from the distillation of the heavy oils, and is obtained by stopping the distillation before the anthracene oils begin to come over, that is to say, when the specific gravity of the oil as it distils is about 1·090. This pitch when well boiled and thinned down with naphtha or light oil is the most serviceable for varnish makers. Mixed with coal dust and pressed into briquettes it forms block fuel.

Hard Pitch, softening at 100° C. and melting at 160° to

200° C., is very insoluble, so much so that it is of but little or no service to the varnish maker. It is even too brittle for use in the manufacture of briquettes, and for this purpose has to be mixed with the oil from which the anthracene has been eliminated. It is supposed in fact to contain free carbon, due, perhaps, to coking going on in the still to a greater or less extent. Behrens obtained from pitch freed from all oils up to specific gravity 1·120, by successive digestions with benzene, carbon disulphide, boiling benzene and boiling alcohol, 23·54 per cent. of a black powder, quite similar to South Wales anthracite, of the following composition :—

	A Per cent.	B Per cent.
Carbon - - - -	90·836	91·921
Hydrogen - - -	3·058	3·157
Ash - - - - -	0·398	0.872

Good hard pitch, according to Habet, consists of :—

	Per cent.
Carbon - - - - -	75·32
Hydrogen - - - - -	8·19
Oxygen - - - - -	16·06
Ash - - - - - -	0·43
	100·00

Medium Hard Pitch, as its name implies, is obtained by stopping the distillation between the gravities 1·090 and 1·120.

Soft Pitch may be kneaded between the teeth. Medium pitch is less easily masticated. Hard pitch crumbles to a powder.

INDIA-RUBBER OR CAOUTCHOUC.

Origin.—India-rubber is the dried milky juice which runs from a great number of South American, African and Indian plants, such as the *Siphonia elastica* (Pers.), the *Ficus elastica* (L.), the *Castilloa elastica* (Cav.), etc.

Commercial Varieties.—These are distinguished according to the names of the country from which they have been imported.

(1) *Brazilian Rubber.*—Para (the most highly esteemed), Maranham, Ceara, Bahia, Pernambuco.

(2) *Central American Rubber.*—Panama, Guayaquil, Carthagena, Central America.

(3) *African Rubber.*—Gaboon, Sierra Leone, Liberia, Mozambique, Madagascar, Nossi-Be.

(4) *Asiatic Rubber.*—Assam, Rangoon, Singapore, Borneo, Java, Sumatra.

Properties.—India-rubber is soft, flexible and almost colourless in thin sheets; it possesses a remarkable elasticity, but it hardens with cold. It melts at 235° C.

Composition.—India-rubber is a hydrocarbide with a composition corresponding to the formula C_4H_7; it moreover contains small quantities of fatty matters, nitrogenous substances, sulphur and chlorine.

Solubility.—India-rubber is insoluble in water and alcohol; it is soluble—or rather swells whilst presenting the appearance of a real solution—in ether, bisulphide of carbon, coal tar naphtha, petroleum spirit, and essential oils. Heavy coal tar oils dissolve about 5 per cent. of their weight, whilst the light naphthas dissolve about 30 per cent. The best solvent for rubber is a mixture of 100 parts of bisulphide of carbon and 5 parts of absolute alcohol, which yields a perfectly limpid solution. Pinene hydrochloride reacts energetically on rubber, quickly effecting complete solution. See also "Caoutchoucine," p. 137.

It is necessary to remark that if the evaporation of the solvent be slow, the rubber yields a tacky, pitchy coat.

India-rubber withstands the action of alkalies and dilute acids.

GUTTA-PERCHA.

Origin.—Gutta-percha is the product resulting from the drying of the sap of the *Isonandra gutta* (Hooker), a tree indigenous to the Malay Peninsula.

Properties.—Gutta-percha is a greyish white substance, with great suppleness but little elasticity. It begins to melt at 48° C. It is ductile at 50°, pasty and malleable at 100°, and fusible at 130° C. Left for a long time in contact with the air, it alters and becomes brittle.

Composition. — According to Payen it contains three proximate principles, which he named gutta, albane and fluavile.

Solubility.—It is insoluble in water; alcohol only dissolves 15 to 22 per cent. It partially dissolves in the hot in spirits of turpentine, shale naphtha and in fatty oils. It also dissolves fairly well in benzene, and is completely soluble in bisulphide of carbon and chloroform, yielding after filtration perfectly limpid and colourless solutions.

It resists the action of alkalies and most acids.

BALATA.

Origin.—Balata is the concrete milk of the bullet or bully tree of Central and South America (*Mimusops balata*).

Extraction.—After stripping off the bark a number of oblique cuts are made with [illegible] knife extending to about seven feet up the trunk. The [illegible] caught in a clay ring encircling the tree below the cuts, and is collected in calabashes.

Yield.—A tree gives from 6 to 30 ozs. of milk, giving when dry ¾ to 1 lb. of balata. But when the trees are cut down, and circular incisions made thereon about one foot apart along the trunk and a receptacle placed below each incision, 5½ to 11 lbs. of better quality gum are obtained from an average tree, whilst large trees yield as much as 45 lbs.

Properties.—The crude resin dries rapidly to the consistency of leather in two to three days. Its specific gravity is 1·042. It is intermediate in properties between india-rubber and gutta-percha, possessing the elasticity of the one and the ductility of the other without the intractability of the former or brittleness of the latter, thus possessing advantages for many purposes not shared by either.

Solubility.—It is freely soluble in benzol and carbon disulphide in the cold, and when heated in spirits of turpentine, chloroform and petroleum spirit. It, however, only dissolves but partially in absolute alcohol and ether. Neither caustic alkalies nor hydric chloride have any action, but concentrated sulphuric and nitric acids attack it energetically. It is easily vulcanised.

Uses.—It is used extensively as a chewing gum in the United States, and as an electrical insulator, and in varnish making.

Chicle is analogous to, if not identical with, balata. Its uses are similar.

CAMPHOR.

Camphor is obtained from several trees of the laurel family. It is a semi-transparent white substance, which is easily pulverised after being moistened with a few drops of alcohol or ether. Camphor dissolves in alcohol, ether, acetone, acetic acid, wood spirit, bisulphide of carbon and oils. The addition of camphor to the solvents used to dissolve refractory resins greatly facilitates the solution of the latter. For a full description see "Camphor Oil," p. 143.

GURJUN BALSAM OR COCHIN CHINA WOOD OIL.

Origin.—This oleo-resin, notwithstanding its synonym, has no connection whatever with Japanese wood oil. The latter is a fatty oil belonging to the class of drying oils. Gurjun balsam is an oleo-resin secreted by different trees belonging to the order *Dipterocarpæ*.

Trees and Localities yielding Gurjun Balsam.

Species of Diptero Carpus.	Country.
Turbinatus - (*lævis, indicus*)	E. Bengal, Chittagong, Pegu, Singapore, French Cochin China.
Trinervis -	Java and the Philippines.
Incanus -	Chittagong and Pegu.
Alatus - -	Chittagong, Burmah, Tenasserim, Andamans, Siam and Cochin China.
Crispalatus -	French Cochin China.
Zeylanicus -	Ceylon.
Hispidus -	Ceylon.
Gracilis -	Java.
Littoralis -	Java.
Retusus - (*Spanoghei*)	Java.

Habitat.—These *Dipterocarpæ* grow in the forests of mountainous regions, and more especially on the borders of the clearances.

Yield.—The collection of the oil is one of the chief industries of French Cochin China, where the trees are now felled in a systematic manner under Government supervision. The annual value of the oleo-resin of a *Dipterocarpus* in that country is estimated at about 16s. to 17s., and the trees continue to yield for about 100 years.

Collection—Forestry Technology.—The oil is collected in the following manner: The woodman first of all makes a round hole in the wood with an auger about 2 inches in diameter and reaching to within a short distance of the opposite side of the tree. It is at the mouth of this hole that the font is made to receive the oil. At this point a deep, almost horizontal, cut is made in the tree by means of an axe, and higher up another vertical cut meeting the other at an acute angle. A sort of font or basin-shaped hole is thus scooped out of the trunk of the tree about the height of a man from the ground where the oleo-resin collects.

When the collection of the resin commences glowing, charcoal is placed in the font, or leaves saturated with oleo-resin, and enflamed; the surface of the wood chars and the fluid oil commences to flow. The fire is then extinguished, and three or four days afterwards the oil is collected.

At intervals of three to four weeks the charred surface of the font is chipped off and the fresh surface again fired. The same font may be fired eight to ten times, and it is not uncommon to find upon the same tree and at the same height four or five cuts, each of which are exuding oleo-resin.

The oil is collected by means of a sort of spoon, formed of meshes of cocoa-nut, and fixed to a bamboo handle; occasionally by means of a gutter with a wide mouth, from which the liquid runs into a receptacle at the bottom of the tree.

Some species of *Dipterocarpe* secrete balsam spontaneously from a cut made on the trunk which is collected in the cold. This "cold-drawn" quality is much esteemed by the Annamites for the manufacture of lacquer.

The collection of balsam constitutes the principal if not the sole occupation of the Annamites of the province of Ben Hoa. The product is of a mixed character, varying much in character and quality owing to the resin of different species being collected and stored in the same vessel.

Commercial Varieties.—Two qualities of oleo-resin are met with:—

(1) *Pale Yellow* (*Blonde*).—The product principally of *Dipterocarpus turbinatus*, and which on standing separates into two layers. The *upper* layer is clear limpid amber, yellow and slightly fluorescent. The lower layer is very abundant, viscous, and of a greyish white colour.

(2) *Black.*—The product principally of *Dipterocarpus alatus.* On standing it separates into a thick, viscous and very fluorescent upper layer, deep brown red by refracted light, greenish by reflected. The lower dark greenish black layer

of less bulk separates more rapidly. The density of these oleo-resins is not definite; it would appear to oscillate between 0·960 and 0·966. The pale oil is always the lighter in gravity.

Both the blonde and the black oleo-resin when distilled yield a volatile oil which has been rectified over chloride of calcium.

M. Rigal obtained the following results:—

Rectification—Fractionation of Pale Yellow Oil.—A litre of blonde oil mixed with six litres of water yields 290 grammes of a straw yellow volatile oil, lighter than water, whilst a white material occupies the space between the water and the oil, which would appear to be the hydrate of the volatile oil. This hydrate is decomposed at a slight heat (176° C.) in contact with calcium chloride.

By fractional distillation M. Rigal has obtained 242° C. as the point at which the liquid boils. The different fractions pass over as under:—

1st fraction between 258° and 262° C.
2nd fraction between 267° and 275° C.
3rd fraction between 284° and 295° C.

The third oil is greenish in colour.

Black Oil Fractionation.—The black oil, 320 grammes per litre, commences to boil at 248° C. and yields:—

1st fraction between 252° and 257° C.
2nd fraction between 260° and 262° C.
3rd and final fraction between 262° and 266° C.

Properties.—These oils rotate the plane of polarisation differently. This is due to the fact that they are the product of different species. (French and American spirits of turpentine—one obtained from *Pinus maritima* and the other from *Pinus toeda*—behave similarly.) They detonate with fuming nitric acid. Nitric acid gives a violet coloration, sulphuric acid a purple red, hydrochloric acid a violet red. Iodine attacks them energetically but without explosion.

The oil resembles copaiba in smell, flavour and therapeutical properties, but it imparts no unpleasant smell to the breath. It is completely soluble in benzol, cumol, chloroform and carbon disulphide, and partially in the other varnish solvents. Gurjun balsam is sold in Albay, Philippine Isles, at 4½d. a pint. It is extensively used in England in skin diseases, and in India as a substitute for copaiba. In the far East it is principally used in varnishes, lacquers and paints, with the special object of protecting the wood against the ravages of white ants.

Termites.—It is also applied generally to the seams and bottoms of ships.

In the crude state it is said to have but little body and to dry slowly, but when the essential oil is driven off it dries better.

PARAFFIN WAX. *Melting-Point*, 58° C.

Properties.—Paraffin is a solid, white, brilliant substance, soft to the touch, inert, inodorous and insipid; it dissolves in 28½ parts of boiling alcohol, from which it separates in beautiful pearly lamellæ on cooling. It dissolves in petroleum and shale, naphtha, spirits of turpentine and fixed oils. It dissolves in hot alcohol but crystallises out on cooling.

Source.—It is obtained by distilling crude petroleum and shale oil. It concentrates in the heavy oil distillates, from which it is separated by a process of refrigeration, sweating and consecutive refining by acid and alkali.

Uses.—It is sometimes used to diminish the brittleness of some varnishes. It is likewise used as an encaustic, and finds a special use in mural decoration by artists to protect and preserve delicate tints without injuring their purity of tone. It has the disadvantage of becoming plastic and liable to bend considerably below its melting-point, but its indifference to acids and alkalies recommends it for many purposes; a minor application of this property being to coat the labels used on chemical laboratory reagent bottles so as to preserve them from acid fumes.

Composition.—A mixture of solid hydrocarbides of the marsh gas or C_nH_{2n} series.

OZOKERIT (EARTHWAX, CERESIN).

Ozokerit is a naturally occurring crude paraffin wax of high melting-point. Its colour is generally black from bituminous impurities, but it may be refined into yellow and white ceresin.

PYROXYLINE OR GUN COTTON.

Manufacture.—(1) Pyroxyline or gun cotton is obtained by immersing cotton wool, or other finely divided cellulose material, in a mixture of 3 volumes of nitric acid and 5 volumes of sulphuric acid. The cotton becomes nitrated to a greater or less extent according to the length of time it has been left in contact with the acids. It is well washed in running water to free it from all trace of acid, and then dried at a very gentle heat (below 100° C.).

(2) The above mixture may be replaced by a mixture of 200 parts of sulphuric acid at 66° C. and 100 parts of well-dried nitre, and the cotton, 5 parts, is left therein for fifteen minutes. This yields a mixture of mononitrated and binitrated cellulose, which is much more soluble than the trinitrated cellulose.

(3) Pyroxyline soluble in alcohol and ether may also be made as follows: 4 volumes of fuming nitric acid are diluted with an equal bulk of water, and 18 volumes of concentrated sulphuric acid added. The whole is well stirred, and when the temperature, which will have risen to about 70° C., has fallen to 55° C., the cotton wool is dipped in the liquid contained in a stoppered vessel, or in a precipitating flask

covered with a glass plate. The cotton wool as it is being immersed is pressed against the sides of the vessels with a stirring rod so as to eliminate air-bells. The vessel is covered with the glass plate or the stopper inserted, and the whole allowed to stand for twelve minutes. The cotton is then quickly withdrawn and plunged into a basin full of water and the washing of the cotton is gone on with in such a manner as will free it as speedily as possible from the greater bulk of the acid, the final traces of which are got rid off in a current of water.

Solubility.—Pyroxyline is soluble in a mixture of alcohol and ether, in methylic alcohol and acetate of amyl.

Pyroxyline should not be stored by the inexperienced. It should be kept moist with water.

CELLULOID.

Celluloid is a body obtained from a mixture of pyroxyline, camphor, etc. This substance swells and dissolves in a mixture of alcohol and ether; acetate of amyl may also be used as a solvent.

Sometimes pyroxyline is dissolved, and 25 to 50 per cent. of camphor added to the solution. Celluloid waste, old combs, etc., can also be economically used in spirit varnish manufacture, amyl acetate being again used as a solvent.

METALLIC RESINATES.

The resinates of magnesia, lime, baryta, zinc and aluminium may be used as gum resin substitutes. They are made by dissolving rosin in a diluted solution of carbonate of soda and precipitating by a weight of the salt (the metal of which is required in the form of resinate) equivalent to the amount of soda used to saponify the rosin.

SECTION II.

RAW MATERIALS (*continued*).—COLOURING PRINCIPLES.

Colouring Principles.

The principal substances used to colour varnishes are:—

1. Dragon's Blood.
2. Gamboge.
3. Gum Accroides.
4. Seed Lac.
5. Sandalwood.
6. Safflower.
7. Annatto.
8. Turmeric.
9. Saffron.
10. Indigo.
11. Aniline Dyes.
12. Coloured Resinates.

1. *Dragon's Blood.*[1]—Dragon's blood (specific gravity, 1·2) is a term used to designate a balsam obtained from different species of plants, but principally from the fruit of the *Calamus draco* (Willd.) of Indo-China and the Moluccas.

After collection of the ripe fruit, the brittle resin occurring as a bright red exudation thereon is separated by shaking or beating the fruit in sacks or baskets. Impurities are separated by sifting. It is so far melted by exposure to the heat of the sun or by boiling as to become plastic enough to be moulded into sticks, balls, etc., after which it is wrapped in a palm leaf, in which form it is exported to this country.

Commercial Varieties.—The principal varieties of dragon's blood are:—"globular," "stick" or "reed," "cake" and "lump". The stick or reed, occasionally 12 to 15 inches long and ½ to 1 inch in diameter, is the best dragon's blood (jirnang).

Properties.—The colour of the resin is dark red brown by reflected light, but deep crimson when viewed in the form of

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[1] This substance is probably the dark resinous red colour brought from India in ancient times and called by Pliny the true cinnabar.

thin transparent sections by transmitted light. It is soluble in varnish solvents with the exception of spirits of turpentine, and is only slightly soluble in ether. When heated to 120° C. it melts, emitting the vapour of benzoic acid. At one time it was extensively used to colour spirit varnishes a deep red, but is now almost entirely replaced by the cheaper aniline colour, Bismarck brown.

African Dragon's Blood, though known in medicine from the earliest historical times, now rarely finds its way into commerce, and until recently nothing has been known of its history. In 1871, however, Mr. Baker described a species of *Dracæna* found in Somaliland, and yielding dragon's blood, under the name of *D. schizantha*, and he also identified a species of *Dracæna* found in Socotra as *D. ombet.* This tree is described as growing at an elevation of 1500 feet, attaining a height of twenty feet, with spreading, drooping branches of a mushroom form. The resin is obtained by scraping the bark, and after about fifteen to twenty days it exudes. It is exported from Aden chiefly to Bombay, where it is used by goldsmiths.

Adulterations.—Dragon's blood is adulterated with dammar and rosin, which are added at the time the resin is being prepared for the market. In poor qualities the sophistication is so obvious as to be perceptible to the naked eye.[1]

2. *Gamboge.*—Gamboge, a gum resin produced by plants of the family *Guttiferæ*—the *gutta fera vera* and the *Cambogia gutta*, which grow in the peninsula of Cambodia and the island of Ceylon—is used to colour varnishes.

Commercial Varieties.—The principal kinds are:—

1. Gamboge in cylinders and sticks.
2. Gamboge in cakes or in lumps.
3. Gamboge in bulk.

Composition.—Gamboge contains, according to the kind, 35 to 80 per cent. of yellow resin and 14 to 19 per cent. of

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[1] Dragon's blood fades on exposure to sunlight but is not affected by foul gases.

gum soluble in water. The following proximate analyses of different varieties of gamboge are by Castelloe:—

	Lump per cent.	Pipe per cent.	Powder per cent.
Resin - - - -	67·6	79·30	76·6
Gum - - - -	27·4	19·45	22·5
Impurities - - -	3·8	0·15	0·7
	98·8	98·90	99·8

Ure quotes an analysis showing 89 of resin and 10·5 of gum.

The resin consisting of gambogic acid, $C_{30}H_{35}O_6$, may be separated from the gum and impurities by digestion in strong alcohol. By evaporating the latter pure gambogic acid is obtained of a deep brown red colour, which, when pulverised, becomes converted into a beautiful yellow.

Solubility.—Gamboge is very soluble in ether, less soluble in alcohol. It is often adulterated with starch, sand and tinctorial barks, a fraud which is detected by digesting the finely ground resin with 60 per cent. alcohol and examining the residue with the microscope.

Gamboge as met with on the market varies greatly in quality. That from Ceylon, presumably from the *Stalagmites*, a pseudo gamboge, is said to be very inferior.

Physical Properties.—The fresh fracture is deep brown red which soon changes to liver colour, the surface becoming coated with a dark green layer. The streak is lemon yellow changing to orange yellow. Fresh fractures have a greasy lustre which soon becomes dull. It has no smell and hardly any taste, but leaves an acrid feeling in the throat. When beaten up with water gamboge forms a fine yellow emulsion—the gum holding the resinous matter in suspension for a very long time.[1]

Uses.—Gamboge is used to colour golden lacquers, but it cannot be used as a pigment except in water-colour painting. Moreover, it is highly poisonous, being a drastic purgative.

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[1] Gamboge is reddened by acids and bleached by sunlight.

3. *Gum Accroides* (Synonyms: Black-boy gum, Botany Bay gum, Xanthorrhea resin, Grass-tree gum).—These resins are the product of several species of *Xanthorrhea*, indigenous to Australia, but more especially of the *Xanthorrhea australis* (of Victoria), *X. hastilis*, and *X. arborea*. Although produced so abundantly in Australia, these were, until comparatively recently, practically unknown in this country. But lately their importation has become more general, and at the present day they, to a certain extent, replace dragon's blood, gamboge and other tinctorial agents in the colouring of pale lacquers.

The resin exudes naturally, but a further quantity is obtained by crushing the stems and sifting, but, as may naturally be expected, the resin obtained in this way is much contaminated with plant *débris*, and is consequently inferior in quality. There are two varieties:—

(1) *Yellow Gum Accroides*, the product of *X. hastilis*, contain, according to Hildebrand, free acids, cinnamic and paracumaric. The latter is also present in the form of an ester combined with xanthoresinotannol, an alcohol with the formula $C_{43}H_{49}O_9OH$. This ester constitutes as much as 80 per cent. of the resin. Xanthoresinotannol is an alcohol containing a hydroxyl group. No methoxyl group could be detected by the Zeisre method. The behaviour of the acetyl and benzoyl derivates pointed to the presence of a hydroxyl group only. This resin alcohol may be converted by direct nitration into picric acid. In addition to the above, styracin, cinnamic acid, phenol propyl, ester, paroxybenzaldehyde, and possibly vanillin, were also present.

(2) *Red Gum Accroides* differ in composition from the yellow variety in containing paracumaric but no cinnamic acid; parabenzaldehyde is present, but no vanillin, styracin, nor the phenol propyl ester of cinnamic acid. The greater bulk of the resin consists of an ester of paracumaric acid,

conjugated with erythroresinotannol. The tannic resin alcohol has the formula $C_{40}H_{39}O_9OH$, and is readily converted by nitration into picric acid. Benzoic acid, but only in the combined state, is also present. The xanthorrea resins thus belong to the same class as Peru balsam, storax and benzoin. A concentrated solution of the red resin, containing a little castor oil or copaiba balsam to give it elasticity, has a magnificent red colour, which is employed to coat the windows of photographic laboratories so as to exclude the active rays.

4. *Seed Lac.*—This resin is described under " Shellac ".

5. *Sandalwood.*—There are three kinds of sandalwood, *viz.*, white, yellow and red. It is the latter variety (*Pterocarpus santaline*), the red sanders (*Lignum santali rubrum*), that is used in varnish staining. It is a solid, compact, dull heavy red coloured wood imported from the Coromandel coast and the mountainous parts of India. It is met with in the market as large logs, or in the shape of chips, or in the form of either coarse or fine powder. Its taste is neither sweet, bitter nor astringent.

Santaline.—The chief colouring principle, which is very permanent, is santaline. It is present to the extent of 16·75 per cent. ; it is a crystalline red powder, melting a little below 212 F., soluble in alcohol, ether, acetic acid, and caustic alkalies. It may be extracted and isolated from the wood as follows: The finely-powdered sandalwood is completely exhausted with alcohol, and the alcoholic solution treated with an excess of hydrated oxide of lead (made by precipitating sugar of lead with caustic soda). The precipitate is collected on a filter, washed with alcohol and dissolved in acetic acid. To the solution an excess of water is added which precipitates the colouring matter. The solution of acetate of lead may be used to make new hydrate of lead. The precipitated colouring principle (pure santaline) is washed and dried at a low temperature. In beauty and brightness it is nearly equal to carmine, and is of great interest to painters, who find it to be a very solid and fast colour. The carriages of Napoleon III. were painted with it, and nine years afterwards were as bright as when it was first put on.[1]

Adulterations.—The powder is said to be frequently adulterated with red raddle, a fraud which may be detected by triturating 2 of the powder with 10 of water, and afterwards shaking with chloroform. The wood floats on the chloroform.

6. *Safflower*, or *Carthamus tinctoria*, sometimes confounded with saffron, is an annual plant, cultivated originally in the Levant, but afterwards in Persia, which furnishes the best quality, and other parts of Asia, Egypt, America and Europe. There are two varieties of it, one with large the other with smaller leaves. It is the last which is cultivated in Egypt, where it forms a considerable article of commerce—the saffron grown in that country rivalling in quality that of Persia. Spain produces a fair quality, whilst that of France, Mexico and Germany is inferior.

Composition.—Safflower contains two colouring principles, one yellow, the other red. The yellow principle is alone soluble in water. Its solution is always turbid, giving with reagents the precipitates common to yellow colouring matters. The alkalies render it lighter, the acids deepen it in shade, giving it more of an orange hue; both produce a small dun precipitate which clarifies it. Alum gives a slight deep yellow precipitate. The precipitates with the solution and other metallic salts are not characteristic.

Alcohol takes but a slight dye out of those flowers from which the yellow substance has been previously extracted with water. But such flowers yield a yellow liquor, with caustic alkaline solutions, which on neutralising with acid becomes turbid and reddish, and deposits a slight reddish-

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[1] Some authorities, however, describe it as fugitive.

yellowish precipitate. Solutions of alum, zinc and tin yield a yellow, and those of iron a copper greenish tinted, precipitate. If a carbonate of an alkali has been used, the acids produce an abundant and a redder precipitate, but the shade differs according to the acid employed. Alum gives with the carbonated alkaline solution a red precipitate, which is so light that it usually floats on the surface of the liquor. This colouring matter is so delicate that it must not be treated with hot solutions, otherwise the precipitates have no longer the same beautiful colour.

The petals of safflower have a fine flame colour. It should be gathered only when it begins to fade; and it is better when it has received rain in this state, although there is a prejudice to the contrary. The rain may be supplied by an artificial watering of the flowers morning and evening. The seeds may still be left to ripen after the blossom is cropped.

These directions are given with the view of separating the yellow substance, a redundance of which may constitute the difference between the carthamus of Western Europe and that of the Levant. It is proper to keep the carthamus in a moist place, for too much drying might injure it.

It has been grown successfully at Göttingen and Amiens. The yellow matter of safflower is not used, but in order to extract this portion the carthamus is put into a bag which is trodden under water till no more colour can be pressed out. The flowers which were yellow become reddish and lose nearly one half in weight.

These are now treated with alcohol, which extracts almost pure carthamine, a substance which is soluble in fatty oils, yielding a rose red or orange red liquid. According to Guignet, carthamine is very dear, rising as high as 3000 francs the kilogramme, but it has great tinctorial powers. It is extensively used in the preparation of vegetable rouge, which has the advantage of colouring the skin without dyeing it.

7. *Annatto* is a somewhat dry hard paste, brown without and red within. It is usually imported in cakes of two or three pounds' weight, wrapped up in leaves of large reeds packed in casks from America, where it is prepared from the seeds of the *Bixa orellana* of Linnaeus, the *Rocouyer* of the French.

The pods of the tree being gathered their seeds are taken out and bruised, and it is from the resinoid pulp that the annatto is produced. They are now transferred to a vat, covered with water, and left for several weeks or even months. The substance is now squeezed through a sieve placed over the vat so that the water containing the colouring matter may return thereto. The residue is covered with banana leaves and allowed to ferment, after which the process is repeated, and so on till no more colour remains.

The substance thus extracted is passed through sieves to separate the remaining seeds and the colour is allowed to subside. The precipitate is boiled in coppers till reduced to a consistent paste; it is then suffered to cool and dried in the shade.

Another and simpler method consists in simply washing the seeds of annatto until they are entirely deprived of their colour, which lies wholly on the surface, and precipitating the colouring principle by lemon juice or vinegar, and to boil down or to drain in bags. The annatto produced in this way is of quadruple value to that made by the previous process; moreover, it dissolves more readily and gives a purer colour.

Annatto contains two colouring principles, *viz.*, orelline and bixine. Orelline is yellow, soluble in water and alcohol, and almost insoluble in ether, whilst bixine is red, very slightly soluble in water, but soluble in alcohol, ether and alkaline solutions.

According to Dr. John, annatto contains an aroma, an acid, resin combined with the colouring matter, vegetable mucilage fibrine, coloured extractive, and a peculiar matter which approaches to mucilage and extractive. This analysis explains why an alkali is added to annatto when used in dying. The alkali combines with the resin and forms a soap which dissolves in water. It likewise acts on the colouring matter, rendering it more lively.

Annatto is often adulterated by sprinkling and mixing it with urine, which can be recognised by the smell of ammonia which it gives off when heated with caustic soda. It is sophisticated with ochre and sand, which are recognised on treating with alcohol.

A decoction of annatto in water is turbid, has a strong peculiar odour and a disagreeable taste. Its colour is yellowish red, turning orange yellow with alkalies; at the same time the liquor clarifies and becomes more agreeable, while a small quantity of a whitish substance is separated from it which remains suspended in the liquid. If annatto be boiled along with an alkali it dissolves much better than when alone, and the liquid has an orange hue.

With the liquor (1) acids yield an orange coloured precipitate, soluble in alkalies to a deep orange coloration.

(2) Alum yields a deeper orange precipitate; the liquid is of a pleasant lemon yellow colour verging to green.

(3) Sulphate of iron forms an orange brown precipitate: the liquor retains a very pale yellow colour.

(4) Sulphate of copper gives a precipitate of a yellowish brown colour a little brighter than the preceding: the liquor preserves a greenish yellow colour.

(5) Solution of tin produces a lemon yellow precipitate which falls slowly. The colouring principle of annatto is soluble in oil as well as in alcohol. A solution in olive oil is used in France to impart a butter yellow tint to margarine.

8. *Turmeric.*—The colouring principle of turmeric is

furnished by the root (*rhozome*) of *Curcuma tinctoria* (Gub.) (*lepra merita*) which flourishes in the East Indies. This substance is very rich in colour, yielding a brilliant orange yellow, which is not however permanent. It is soluble in ether, alcohol and coal tar naphtha. It is insoluble in water. It dissolves in caustic alkaline solutions from which it is precipitated by acids.

Turmeric powder is often adulterated especially with pea flour, which can easily be distinguished under the microscope.

9. *Saffron* consists of the stigma of the flower of the cultivated saffron, *Crocus sativa*, indigenous to the East, but now cultivated generally throughout Europe. It requires a million flowers to make 2¼ lbs. of saffron.[1] It contains a yellow colouring principle, *safranine* or *crocine*, which, when isolated, consists of a rose powder that dissolves with a yellow coloration in boiling water, alcohol and alkalies.

Saffron is often sold after being exhausted with alcohol and dried, but in that case instead of having a greasy lustre its surface is dull. It is often mixed with other flowers, with the fibres of dried beef, etc. These sophistications may be detected under the microscope.

Certain formulæ for spirit varnishes still retain saffron, but it is principally used in cooking, confectionery and medicine.

10. *Indigo* is a vegetable dye obtained by a process of steeping, fermentation and oxidation from the leaves of *Indigofera tinctoria* and *I. cerulea*, natives of the East Indies and other parts of Asia. Indigo is met with in commerce in the form of small cubes or in flat irregularly shaped pieces of a bright black or greenish blue colour, and consisting of a dry paste containing among other matters a peculiar colouring principle, *indigotine*, which may be isolated by sublimation.

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[1] Neither of these must be confounded with the aniline dyes of the same name.

Indigo is used in spirit varnishes, but only the very light kinds so as to avoid precipitation. When indigo is treated with sulphuric acid, and the product neutralised with soda, a blue colouring substance is obtained called indigo carmine, which is soluble in water and oil and finds a use in miniature painting.

When indigo carmine is used to colour varnishes it is first ground upon a slab with a small quantity of the varnish and then incorporated with the bulk.

11. *Aniline Colours, used in Lacquer Making, etc.*—(1) *Magenta.*—Crystals which have a greenish metallic lustre by reflected light, but when viewed in thin sections by transmitted light their colour is red. It dissolves rather sparingly in water, imparting to it a crimson colour without fluorescence. It is precipitated by tannic acid from its aqueous solution. It dissolves readily in alcohol, and also in amylic alcohol (fusel oil, etc.). Only a comparatively small proportion may be used to colour spirit varnish, otherwise it will dry with a bronze reflection which will mask the true colour.

(2) *Safranine* is met with in commerce as a brown red powder. It forms when pure red crystals with green reflection, which dissolve in water a red colour. It is precipitated by tannic acid from its aqueous solution. Its solution in alcohol is red with a fine orange fluorescence, and when mixed with auramine it colours spirit varnish a fine scarlet. It does not dissolve in ether.

(3) *Acid Magenta.*—Commercial acid magenta is a green powder with metallic lustre which dissolves in water and alcohol with a bluish red colour. Caustic soda discharges dilute acids, even CO_2 reproduce the colour. Its tinctorial power is only about one-half that of ordinary magenta.

(4) *Alkali Blue.*—Commercial alkali blue is met with as a brown powder or in lumps, which dissolve in 5 parts of water and in alcohol. Acetic acid colours blue and deposits a blue precipitate on boiling. Hydrochloric acid precipitates

the colour with decolorisation of the solution of the chemically pure colour. If carbonic acid is given off on acidification, soda is present; ammonia in excess decolorises the solution; caustic soda gives a violet red coloration, which turns to reddish brown on boiling.

(5) *Spirit Blue*, or aniline blue, is insoluble in water, but as its name implies is soluble in alcohol and ether. It is principally used in the manufacture of alkali blue. Its shade varies from blue to violet.

(6) *Phosphine* as met with in commerce is an orange yellow powder, soluble in water, from which it may be precipitated by tannic acid. Alkalies precipitate it in yellow flocks soluble in alcohol and ether. The precipitate is coloured brown red if sample impure. If concentrated hydrochloric acid be added to a solution of phosphine, a precipitate of the di-acid salt is produced which is freely soluble in water. It is soluble in alcohol and also in ether, imparting to the solution a magnificent yellow colour with intense green fluorescence. Aqueous solutions of phosphine yield in the cold, with a 1 per cent. solution of nitrate of potash, an immediate characteristic red crystalline acicular precipitate of chrysaniline nitrate.

(7) *Auramine.*—Commercial auramine is a yellow powder which yields a yellow solution with water. Alkalies give a white milky percipitate soluble in ether without fluorescence. Mineral acids gradually decolorise it in the cold. Sodium amalgam gradually decolorises the alcoholic solution, which, on the addition of water, yields a colourless precipitate, yielding a deep blue coloration with hot acetic acid. Zinc dust and acetic acid give a green colour.

(8) *Picric Acid*, although a coal-tar colour, was discovered so far back as 1799. It is met with in the form of pale lemon or straw yellow foliae, which melt at 122·5° C. It may be sublimed, but with the greatest of care, as it is highly explosive, and its alkaline compounds even more so. It may

be used by itself to colour varnishes yellow or in combination with suitable blues for green lacquers, etc. It is produced by the action of nitric acid upon carbolic acid. It is also generated when silk, wool, leather, aniline, indigo, resins, etc., are acted upon by this acid. It only dissolves in 165 times its weight of water, but 1 part in 1000 communicates a distinct greenish yellow colour and a most intense bitter taste to the water used for its solution; but it dissolves easily in alcohol, ether and benzene.

The commercial article is liable to adulteration. It should be completely soluble in benzol, and in water acidified with sulphuric acid and in 10 parts of alcohol. Any matter insoluble in alcohol would indicate mineral matter, sulphate or nitrate of soda. The solution in ammonia should give no precipitate with calcium chloride (absence of oxalic acid). Sugar may be detected by neutralising with soda and extracting with dilute alcohol. The sugar dissolves; the picrate of ammonia is insoluble.

(9) *Naphthol Yellow* is the sodium, etc., salt of binitro naphthol. It is met with in the form of needles which dissolve freely in water. Hydrochloric acid decolorises with precipitation of binitro naphthol. Ammonia has no action; caustic soda and potash give an orange red precipitate. Potassic cyanide gives a red solution, which deposits after a time red brown crystals—isopurpuric reaction. The presence of picric acid may be detected by the aqueous solution being incompletely decolorised by hydrochloric acid.

(10) *Aurantia* is met with as a crystalline yellow powder which detonates on heating. The corresponding sodium salt is very explosive. It dissolves freely in water and alcohol. Hydrochloric acid (dilute) gives a dark yellow precipitate, the solution being almost decolorised. Boiling with tin crystals gives a dark brown precipitate. Its solutions are said to produce painful blisters on the skin.

(11) *Chrysoidine.*—Commercial chrysoidine consists of deep violet crystals with metallic lustre, dissolving easily in boiling water and in alcohol. The aqueous solution coagulates into a characteristic deep blood-red gelatinous mass. This colour dyes wool orange yellow. Sulphuric acid gives a yellowish brown coloration. Tin crystals decolorise. Basic acetate of lead gives an orange precipitate. Mixed with methyl green gives a fine olive green.

(12) *Bismarck Brown.*—Commercial Bismarck brown is a blackish looking powder, which dissolves in alcohol and water with a red brown colour. It dyes wool orange brown; sulphuric acid turns the aqueous solution yellowish brown. Tin crystals and hydrochloric acid discharge the colour. Basic acetate of lead gives a brown precipitate. The aqueous solution does not gelatinise on cooling (distinction from chrysoidine). This colour is greatly used by French polishers as a mahogany stain.

(13) *Light Green S. F. Yellow Shade.*—This dye comes into commerce as a brown amber-looking powder, which dissolves in water and alcohol. The aqueous solution is completely precipitated by basic acetate of lead yielding a magnificent green lake. It is a truer green and much more permanent to light than any other aniline green, and can be safely recommended to the varnish-maker.

(14) *Malachite Green* is met with in crystalline plates of a green metallic lustre, which dissolves freely in water, alcohol and amylic alcohol (distinction from methyl green). Hydrochloric acid gives an orange coloration, the green colour being restored by dilution; tin crystals a green precipitate. Bleaching powder, ammonia and alkalies decolorises very fugitive. When heated to 100° C. it does not turn violet (distinction from methyl green).

12. *Coloured Resinates.*—These are described in another section.

SECTION III.

RAW MATERIALS (*continued*).—SOLVENTS.

Formerly, beyond the drying oils, the only other liquid solvents used to dissolve gum resins in varnish-making were methylated spirit and spirits of turpentine, but at the present day the liquids used for this purpose are much more numerous.

In fact, any volatile liquid in which the resin dissolves may be used either for the purpose of dissolving the resin in the original manufacture of the varnish or for thinning it down to any required consistency after it has been made and stored for some time and becomes thick through the evaporation of the solvent. Care must, however, be taken that the solvent used to thin down the varnish does not throw the whole of the gum-resin out of solution, and an experiment ought to be made on a very small scale before adding the solvent to the bulk of the varnish. For instance, certain varnishes made by processes known only to the maker—say, under pressure or with a different solvent to that ordinarily used—are completely spoiled by the addition of spirits of turpentine, which causes the immediate separation of the resin from the original solvent, and no amount of heating and coaxing otherwise will get the resin to re-enter into solution again. This can only be done by having recourse to the solvents and methods adopted in the original manufacture, most likely unknown to the party thinning down the varnish, and even if it were known to him very probably he has not got the requisite plant to carry out the operation even if it would pay him to do so.

The more extended use of different solvents and mixture of solvents is therefore apt to cause trouble and annoyance to the actual users of the varnish. Certain copal varnishes are made by dissolving the resin in volatile solvents, *e.g.*, with rosin spirit; under pressure, these, again, will sometimes not thin down with spirits of turpentine in the usual way, any addition of the latter, either in the hot or cold, throwing the resin out of solution.

ETHYLIC ALCOHOL (COMMON ALCOHOL).

$\left.\begin{matrix}C_2H_5\\HO\end{matrix}\right\}$ *Molecular Weight*, 46; *Boiling-Point*, 78·4° C.

This is the principal spirit obtained from fermented liquors, such as wine, beetroot, juice, fermented grain by distillation. For a good many years the distilling plant employed has been so perfected that by a single distillation an alcohol of 95 per cent. strength free from fusel oil (amylic alcohol) may be obtained. Alcohol being more volatile than water, when a mixture of the two is distilled the alcohol comes over first mixed with a certain quantity of water. When these vapours are condensed just as they are given off by the liquid subjected to distillation a spirituous liquor is collected, the strength of which depends upon how far the distillation process has been carried. The longer the duration of distillation the weaker the spirit.

But, if instead of condensing the vapours *per decensum*, we cause them to ascend into a series of receivers arranged the one above the other, or into a column containing receptacles at different heights to receive the condensed liquid, it is clear that the vapour of water will condense first and nearest to the still in which the worts, etc., are being heated, and to which the condensed liquid is constantly being returned, whilst the alcoholic vapours, on the other hand, will rise and ascend further and further away from the source of heat towards the cooler portions and where the more condensable vapour of water cannot follow them. Finally, the alcoholic vapours are themselves condensed in the cooler parts of the condenser most distant from the still, but those portions which condense first are more dilute than the latter portions which condense further away. Only this latter portion is collected for sale. The more watery portions flow back to the heated parts of the condenser, where they are again deprived of their more volatile constituents.

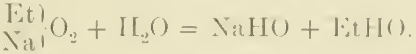
Absolute Alcohol.

Absolute alcohol is prepared by rectifying the alcohol of commerce by substances which take up water. Quick-lime is the substance most generally used. The alcohol is digested with a large quantity of quick-lime in a flask for two days. The latter is then connected with a Liebig's condenser and the alcohol is distilled off. The quick-lime does not appear to slack much. The first and last portions are rejected, because even when working with almost absolute alcohol the former contains a large proportion of water; whilst, on the other hand, owing to the high temperature, the last portion is apt to contain water extracted from the calcuim hydrate by the absolute alcohol. These two portions being collected apart the remainder is absolute potassium permanganate; does not redden it but imparts a faint brown tint.

Instead of directly distilling the alcohol through a Liebig's condenser, the flask may be attached to a vertical reflux condenser, and boiled on the water-bath for an hour, when the condenser is changed to the ordinary position and the alcohol distilled off as before; in this case the lime, some lumps of which should originally have projected above the surface of the liquid, is completely disintegrated. Care must be taken not to use too much alcohol, as the heat generated by the slaking of the lime may cause such sudden and violent ebullition as to project a mixture of alcohol and lime through the condenser.

When the alcohol originally contained more than 5 per cent. of water, a single rectification is not enough, and less quick-lime must be used, otherwise the flask may be broken by the heat developed in slaking. Better results are obtained by digestion with, and distillation over, caustic baryta, made by decomposing the nitrate.

Metallic sodium has been used to remove the last traces of alcohol. A small piece is dissolved in the alcohol, and the whole distilled at a steam heat. The sodium forms ethylate of sodium with disengagement of hydrogen, whilst the traces of water which the alcohol contains decompose the ethylate with the formation of caustic soda and alcohol.



$$\left.\begin{matrix}Et\\Na\end{matrix}\right\}O_2 + H_2O = NaHO + EtHO.$$

Sodic Ethylate and Water = Caustic and Alcohol Soda.

But, according to Mendelejeff, when either sodium or sodium amalgam are used to dehydrate alcohol, traces of sodium or sodium and mercury are found in the distillate.

Potassium carbonate has been used, but it is too weak a dehydrating agent.

A very pure absolute alcohol is obtained on the large scale by simple filtration through quick-lime. Any convenient apparatus may be used, such as an inverted two-gallon tin can, the bottom being removed and a lid tightly fitted in its place, the cylindrical part of the can being lined inside with a cylindrical perforated vessel, in the centre of which a tube is fixed. The can acts as a jacket. The inner vessel is filled with quick-lime and as much alcohol as it will hold. The central tube communicates with the interior of the jacket. The inverted neck of the can is fitted with a stop-cock for running off the dehydrated alcohol after being left in contact for fourteen days.

Properties.—Alcohol is a colourless mobile neutral liquid, possessing an agreeable spirituous odour, burning with a blue flame, and without smoke. Its density, according to Kopp, is—

C.	
0°	0·8095
15·5°	0·7939
20·0°	0·7920

Under the normal atmospheric pressure, alcohol boils at 78·4° C. It mixes in every proportion with water. The mixture takes place with disengagement of heat, and there is a contraction after cooling. The maximum of contraction takes place when 52·3 volumes of alcohol are mixed with 47·7 volumes of water. The result only measures 96·35 instead of 100. Alcohol exposed to the air attracts moisture; it also abstracts moisture from organic substances. Placed in contact with certain salts containing water of crystallisation it dehydrates them.

Alcohol is miscible in all proportions with most liquid bodies with the exception of the fatty oils, which it only dissolves in very limited proportion—castor oil and croton oil excepted, both of which it freely dissolves. In fact, next to water it is the most extensive and important solvent. Its solvent action on resins, balsams, camphor, etc., is well known.

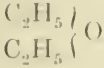
Adulterations.—Water may be detected by adding anhydrous sulphate of copper, which changes from greyish white to blue in dilute alcohol. Also by permanganate of potash, which under like circumstances turns red.

Density of Mixtures of Alcohol and Water.

Alcohol per cent.	Density.	Alcohol per cent.	Density.
90	0·83415	96	0·81245
91	·83081	97	·80829
92	·82738	98	·80390
93	·82385	99	·79926
94	·82020	100	·79433
95	·81641		

Amylic alcohol (fusel oil) may be detected by agitating 5 c.c. of the alcohol to be tested with 6 c.c. of water and 15 to 20 drops of chloroform. The chloroformic solution is decanted and evaporated leaving the fusel oil; about 0·05 per cent. may be detected in this manner.

ETHER (SULPHURIC ETHER).



$\left.\begin{matrix}C_2H_5\\C_2H_5\end{matrix}\right\}O$; *Molecular Weight*, 74; *Boiling-Point*, 34·8° C.

Ether is prepared by the action of sulphuric acid on ordinary (ethylic) alcohol. The process is conducted as follows: 9 parts concentrated sulphuric acid and 5 parts of alcohol are carefully mixed together and after cooling heated to a temperature of 90° C., and a continuous stream of alcohol is caused to flow into the mixture. The flow of alcohol is regulated so that the mixed liquid is always maintained at the same height in the etherifying vessel. The temperature gradually rises to 136° to 137° C. and remains constant as long as there is production of ether. Working in this manner, a mixture of ether and water distils over and is collected in a well-cooled receiver. At the same time a little alcohol, and, if the operation is carried too far, a little sulphurous acid also passes over. The ether thus produced generally contains an oily body, from which it may be freed by the addition of caustic potash and allowing it to stand for twenty-four hours. The ether is decanted, washed with water to remove alcohol, and repeatedly rectified over calcic chloride, and if desired exceptionally pure a small piece of sodium will remove the last traces of alcohol and water.

The tube leading the vapours from the generating vessel to the condenser should be drawn out to a point in the former, and a thermometer dipping into the boiling liquid serves to indicate the temperature.

The reaction takes place in two continuous stages. The sulphuric acid and alcohol first form sulphovinic acid and water. The sulphovinic acid reacting on the fresh supply of alcohol forms ether, and regenerates sulphuric acid, and the latter reacting on fresh alcohol reforms sulphovinic acid, and so on.

Thus the same cycle of reactions goes on indefinitely with the same quantity of alcohol. The sulphuric acid unceasingly regenerated is always the same, but in forming sulphovinic acid it reacts continuously upon fresh supplies of alcohol in such a manner that the sulphovinic acid existing at one moment is not the same as that which existed before or will exist afterwards.

Ether is a colourless light mobile liquid of a characteristic, agreeable, fragrant, ethereal odour. Its taste is first burning, then cooling. It is very volatile and inflammable; burning with a brilliant flame it does not redden litmus but becomes slightly acid by the absorption of oxygen and the formation of acetic acid from contact with the air in imperfectly stoppered bottles. When pure its specific gravity at 15·5° C. is about ·720 under the normal pressure. At − 31° C. it congeals, forming brilliant white plates.

Ether is miscible in all proportions with alcohol, carbon, disulphide chloroform, wood spirit and benzol; 36 parts pure ether dissolves 1 part of water, increasing thereby its density from ·720 to ·723 at 15·5° C.; 9 parts of water dissolve 1 part of ether. From its solution in ether the water may be completely removed by, say, carbonate of potash, provided the ether be free from alcohol and otherwise pure. When completely free from alcohol and water ether has no action on dry tannic acid, which, if either of these be present, liquesces to a thick syrupy fluid.

Ether freely dissolves essential oils, gun cotton, most of the fatty and resinous substances, alkaloids, and in general all substances rich in carbon and hydrogen. Its vapour mixes rapidly with air and forms with the oxygen contained therein a mixture which explodes most violently in proximity to an incandescent body.

Shaken with an equal bulk of water in a small graduated cylinder, ether should not lose more than one-fifth of its volume.

Blue litmus paper when immersed in both strata in the cylinder should remain unaltered; as also, when a small quantity of the ether is evaporated in a porcelain capsule until reduced to a few drops and then tested with litmus paper, a slight acid reaction would indicate acetic acid, and in crude ether possibly sulphurous or sulphuric acid. The acid reaction may also be caused by traces of ethyl sulphate. Traces of this and other ethylic or amylic ethers or alcohols are also indicated when about half an ounce of ether is evaporated from a flat porcelain capsule by causing the fluid to flow to and fro. When the ether is evaporated the surfaces of the capsule should be covered with a deposit of moisture without taste or smell and without any oily appearance.

METHYL ALCOHOL (WOOD SPIRIT) CH_3HO.

Molecular Weight, 32; *Boiling-Point*, 66·6 C.

Specification for Pure Methylic Alcohol.

(1) *Density* = 0·795 at 15° C.

(2) *Percentage of Acetone*, not more than 0·7 per cent., estimated as follows: 10 c.c. of double normal soda (80 grammes per litre) and 1 c.c. measured exactly of the alcohol to be tested are run into a 30 c.c. tube with ½ c.c. divisions, and after agitation 5 c.c. of double normal iodine (254 grammes of iodine and 350 grammes of iodide of potassium per litre) are added, and after agitation 10 c.c. of ether. The tube is now corked and again agitated, after which the volume of ether is measured and 5 c.c. drawn off and evaporated on a tarred watch glass; the residue is dried

over sulphuric acid and weighed, the weight of the iodoform, × 0·14723, gives that of the acetone which is multiplied by the relation of the total ether to that which was evaporated and brought to per cent.

(3) Ninety-five per cent. should distil over in the interval of 1° of the thermometer.

(4) It should at the most only give a bright yellow coloration when treated with double its volume of concentrated H_2SO_4.

(5) Five c.c. should give no immediate coloration with permanganate (1 gramme per litre).

(6) One c.c. of bromine in 80 parts of 50 per cent. acetic acid should only give a yellow coloration.

(7) It should remain perfectly colourless when treated with caustic soda even in excess.

Preparation.—This may be obtained in the laboratory from winter green oil, which consists of acid methyl salicylate $C_7H_4O_3HCH_3$ by distillation with potash, whereby potassium salicylate is formed and methyl alcohol distils over $C_7H_4O_3HCH_3 + KHO = C_7H_4O_2HK + CH_3HO$. The product is very pure.

Manufacture.—The crude wood vinegar obtained in the dry distillation of wood contains about 1 per cent. of wood spirit. It was in this liquor that methyl alcohol was first discovered by P. Taylor in 1812. The crude vinegar is again distilled, and the first tenth which passes over is collected apart. This is neutralised and repeatedly rectified with quick-lime, and the clear liquid, separated from the oil which floats on the surface and from the sediment, is treated with a small quantity of sulphuric acid which fixes ammonia and precipitates tarry matters, and is again distilled over quick-lime. The wood spirit of commerce is rarely pure. When mixed with water it generally turns milky, and an oily layer forms on the top consisting of different bodies insoluble in alcohol. The insoluble portion being separated, and the clear aqueous liquid distilled, methyl alcohol passes over first and is rectified over quick-lime. These processes are long and tedious, and only give imperfect results. When wood spirit is mixed with a fourth of its volume of olive oil, the latter combines with the impurities, and a comparatively pure methyl alcohol is obtained. A surer process is to distil the wood spirit with oxalic or citric acid so as to obtain a crystallisable ether (methyl oxalic ether). The latter is decomposed by distilling with water (after a previous purification by expression between folds of filter paper). The wood spirit thus obtained is rectified over quick-lime.

When pure, methyl alcohol is a colourless mobile liquid, possessing a spirituous odour. The empyreumatic odour of the wood spirit of commerce is due to impurities. Its density at 0° C. is 0·8142. It boils at 66·5° C. under the ordinary atmospheric pressure. However, its boiling-point varies between rather wide limits (60°-65°), according to the nature of the sides of the distilling vessel. Methyl alcohol is inflammable and burns with a non-luminous flame, and hence may be burnt in a spirit lamp. It mixes in all proportions with water, alcohol and ether, and dissolves certain resins and fatty and volatile oils; its deportment in this respect being similar to ordinary ethylic alcohol, although its solvent action on gum resins is often different. Thus some gums are insoluble in ordinary alcohol, and also in wood spirit, but a mixture of certain proportions of these two dissolves the gum, but it often happens that an excess of either of the alcohol precipitates the resin from solution.

Methylated Spirit.

Wood spirit to the extent of 10 per cent. is used to methylate ordinary alcohol for revenue purposes. The mixture thus obtained is known as methylated spirit. It is sold at the strength of 64 O.P., or specific gravity 0·8221. By the term 64 O.P. (over-proof) is meant that if 100 volumes of the spirit be mixed with 64 volumes of water it will produce

164 volumes of proof-spirit. Proof-spirit has a density of 0·920. On the other hand, 30 per cent. under-proof means that 100 volumes only contain 70 volumes of proof-spirit. The relation of per cent. over-proof to gravity is shown in the following table by Ure:—

Specific Gravity.	Per cent. Over-proof.	Specific Gravity.	Per cent. Over-proof.
0·8156	67·0	0·8231	63·4
·8160	66·8	·8234	63·2
·8163	66·6	·8238	63·1
·8167	66·5	·8242	62·9
·8170	66·3	·8245	62·7
·8174	66·1	·8249	62·5
·8178	65·6	·8252	62·3
·8181	65·8	·8256	62·2
·8185	65·6	·8259	62·0
·8188	65·5	·8263	61·8
·8192	65·3	·8266	61·6
·8196	65·1	·8270	61·4
·8199	65·0	·8273	61·3
·8203	64·8	·8277	61·1
·8206	64·7	·8280	60·9
·8210	64·5	·8284	60·7
·8214	64·3	·8287	60·5
·8218	64·1	·8291	60·4
·8221	64·0	·8294	60·2
·8224	63·8	·8298	60·0
·8227	63·6		

ACETONE C_3H_6O.

Molecular Weight, 116; *Boiling-Point*, 55·5° C.

Acetone was known in the middle ages. It was obtained in the sixteenth century by the distillation of sugar of lead. It was formerly known as pyroacetic spirit.

Preparation.—Acetone is prepared by distilling acetate of lime in a fire-clay retort. The vapours disengaged are condensed in a well-cooled receiver. The product is rectified with a small quantity of bichromate of potash and sulphuric acid. It is afterwards distilled on a water bath over an excess of chloride of calcium. Instead of acetate of lime dry acetate of soda may be used, or a mixture of 4 parts of acetate of lead with 1 part of quick-lime. The reaction takes place according to the following equation:—

$$2Ca\,(C_2H_3O_2)\,2 = 2CaCO_3 + {}_2C_3H_6O$$
$$80 + (96 + 12 + 128) = 316\ 200 + 116 = 316.$$

Acetone is a colourless liquid possessing an ethereal, slightly empyreumatic odour. Its density at 0° C. is 0·814. It dissolves in all proportions in water, alcohol, ether and wood spirit. When diluted with water it should give no milkiness. Acetone is very inflammable, and burns with a bright flame.

Acetone may also be prepared from sugar, starch, gum, etc., by distillation with eight times their weight of powdered quick-lime. It is, however, accompanied in this case by proprone, an oily liquid separable by water, in which it is insoluble. A saturated solution of chloride of calcium dissolves in methylic alcohol but not in acetone.

AMYLIC ALCOHOL $C_4H_9CH_2HO$.

Molecular Weight, 88; *Boiling-Point*, 132° C.

Scheele was acquainted with this body in its impure state as fusel oil. Dumas determined its composition in 1834. Cahours in 1837 pointed out its analogy with ordinary alcohol, an analogy which was confirmed by the researches of Dumas and Stas, but more especially by those of Balard.

The fusel oil, which is the last body to come over in the distillation of alcohol (whether from malt, wine, potatoes, or beetroot), consists principally of amylic alcohol. To obtain it pure the fusel oil is washed with water, and the portion which passes over between 128° and 132° C. is collected apart. The portion distilling over below 128° C. contains butylic alcohol.

Amylic alcohol is a colourless liquid which turns brownish by age: it possesses a disagreeable odour, and the vapour is most irritating to the throat and lungs. It boils at 132° C. Its density at 15° C. is 0·8184. It dissolves in alcohol and ether, carbon disulphide, essential and fatty oils, but is insoluble in water. It deviates the plane of polarisation to the left, but to a different extent according to its source. Amylic alcohol does not take fire by contact with a flame, and when dropped on paper does not leave a permanent greasy stain.[1]

CHLOROFORM $CHCl_3$.

Molecular Weight, 120; *Density*, 1·5264 *at* 0°.

This important body was discovered in 1831 by M. Soubeiran and Liebig.

Preparation.—10 parts of chloride of lime and 3 parts of slacked lime are stirred up with 60 parts of warm water. The milky fluid thus obtained is placed in a capacious retort, and ought not to fill it more than one-third at the most; 2 parts of alcohol are then added, and the whole strongly heated. Towards 80° C. a very energetic action ensues, causing a very considerable frothing up. The heat is then withdrawn. Distillation commences and continues of its own accord. As the reaction ceases it is again heated to carry on the final product, and when this has no longer the sweet taste of chloroform the operation is stopped. In the receiver are found two or three parts of a more or less liquid form in two layers. The lower dense layer is chloroform, mixed with alcohol, and coloured yellow by an excess of chlorine. The upper portion is a rather milky mixture of alcohol, water and chloroform. The chloroform is decanted, washed with water, then with a solution of carbonate of potash, and rectified over calcium chloride, and again distilled.

Chloroform is a dense, colourless, volatile, very mobile liquid of an agreeable, ethereal, aromatic odour. Its taste is first sharp, then cool and sweetish. It does not act upon litmus, is not readily inflammable, but when a wick is saturated with chloroform and ignited it burns with a greenish

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[1] Amylic alcohol is an excellent solvent for intractable resins marred only by its toxic effects. Being free from water it leaves the resin on evaporation as a highly resplendent transparent coating.

flame, giving off pungent fumes containing hydrochloric acid. It is very volatile even at ordinary temperatures, producing by rapid evaporation great cold, but leaving neither a residue nor a film of moisture, nor any unpleasant odour when wholly evaporated by the warmth of the hand by causing the chloroform to flow to and fro in a porcelain basin. It boils at 61° to 62° C. Ten parts chloroform dissolve in 7 of rectified spirit, 1 part in 1½ of ether, and 1 in 200 of water. Its specific gravity is 1·490.

It is miscible in all proportions with absolute alcohol, ether, benzol, carbon disulphide and essential and fatty oils, and is an extensive solvent for resins, acting on vulcanite, and dissolving caoutchouc, gutta-percha, paraffin, camphor, mastic, elemi, tolu, benzoin and copal—amber, sandarach and lac are only partially soluble.

ETHYLIC NITRATE $C_2H_5ONO_2$.

Molecular Weight, 91 ; *Boiling-Point*, 87·2° C.

Ethylic nitrate or nitric ether is prepared from its elements (nitric acid and alcohol) with the addition of a little urea. It is a mobile liquid of specific gravity 1·112 at 17° C. It boils at 87·2° C. It is soluble in water, mixes in all proportions with alcohol and ether. It is used in varnish-making as a solvent for Manilla copal, which it completely dissolves. But its use in this respect is not to be recommended, as it contains a large quantity of oxygen in very unstable chemical equilibrium. Varnishes therefore in which this substance has been used as a solvent are liable to explode violently when suddenly heated.

METHYLIC NITRATE CH_3NO_3; *Molecular Weight*, 77.

This is an analogous compound to ethylic nitrate. It is a colourless liquid, with a boiling-point of 66° C., and a specific gravity of 1·182.

ETHYLIC ACETATE OR ACETIC ETHER $\left\{ \begin{matrix} CH_3 \\ CO\ (C_2H_5)\ O \end{matrix} \right.$

Boiling-Point, 74° C.

This substance was discovered by Lauragnais in 1759. It is made by distilling a metallic acetate with sulphuric acid and alcohol, using either of the following formulæ :—

Potassic acetate -	3	Acetate of soda -	6	Lead acetate -	16
Alcohol, strong -	3	Alcohol - -	6	Alcohol - -	$4\frac{1}{2}$
Sulphuric acid -	2	Sulphuric acid -	15	Sulphuric acid	6

The sulphuric acid and alcohol are carefully mixed, the mixture poured upon the acetate of lead, and the whole distilled on a sand bath. The distillate is agitated with a solution of chloride calcium, to which a little milk of lime has been added. The supernatant ether is decanted and dehydrated over calcium chloride and rectified on the water bath.

Acetic ether is a colourless liquid with a very pleasant odour. It boils at 74° C. Its density at 0° is 0·9105. It dissolves in 7 parts of water and mixes in all proportions with alcohol and ether. It is separated from its aqueous solution by calcium chloride. It forms with dry chloride of calcium a combination which is split up at 100° C. with liberation of acetic ether.

Like all other ethereal salts, acetic ether is easily decomposed by potash into alcohol and acetate. Ammonia converts it into acetamide and alcohol.

AMYLIC ACETATE $\left\{ \begin{matrix} C_5H_{11} \\ C_2H_3O_2 \end{matrix} \right.$; *Molecular Weight*, 130.

Amylic acetate is obtained by distilling 2 parts of acetate of potash with a mixture of 1 part of amylic alcohol and 1 part of sulphuric acid. The product of the distillation is agitated with water, the supernatant layer is decanted and rectified, collecting that which passes over at 125° C. A process which yields a more pure product consists in heating in sealed tubes at 120° C. a mixture of amylic iodide and nitrate of silver.

Acetate of amyl has a pleasant odour, recalling that of pears. Its solution in alcohol is used in perfumery under the name of essence of pears.

It is a colourless mobile liquid, the density and boiling-point of which vary with each of its isomeric modifications as seen in the following table:—

	Density at 0° C.	Boiling-Point 0° C.
Normal - - - - -	0·896	148·4
Isoprimary - - - -	0·884	137·6
Prim. active - - - -	0·8762	144

It dissolves in alcohol and ether. Its chief use is as a solvent for pyroxyline or gun cotton.

Carbon Disulphide CS_2.

Molecular Weight, 76; *Boiling-Point*, 76·5° C.

Carbon disulphide is manufactured by heating in a suitable furnace to bright redness freshly glowed wood charcoal in large tubulated fire-clay retorts, lumps of sulphur being run in from time to time. The necks of the retorts are adapted (air-tight) to large receivers which condense and intercept any uncombined sulphur. From these receivers the carbon disulphide vapours pass through condensers. The crude product is very impure, containing sulphur, sulphuretted hydrogen and other compounds, all of which combine to impart to it an abominable smell, from which no amount of repeated distillation will free it. It can, however, be purified by shaking it up with mercury and mercuric chloride and subsequent distillation. The anhydrous sulphates of both copper and iron are also used to purify it.

Properties.—Carbon disulphide is a transparent colourless and highly volatile liquid of great refractive and dispersive power. Its taste is pungent and aromatic, and its smell when pure is similar to chloroform. Its density at 15° C. is 1·27, and it boils at 46·5° C. It burns with a blue flame yielding sulphurous and carbonic anhydride. Its vapour when mixed with air is explosive.

The technical applications of carbon disulphide are many and varied; it is an excellent solvent for oils, fats and resins, wax and paraffin, and for sulphur. In virtue of the latter property, it is much used in the manufacture of vulcanised caoutchouc, *i.e.*, india-rubber, saturated with sulphur. It is extensively employed in the extraction of oil from seeds, and of grease from wool.

But the use of carbon disulphide as a solvent for resins in varnish-making is greatly restricted by the fact that air containing a small quantity of the vapour has the same toxic effects as chloroform, producing insensibility. In large quantities it is poisonous. Moreover, there is the further risk of fire or explosion.

Carbon disulphide is not miscible with water, and when agitated with iodine or bromine water it absorbs the halogen and acquires a faint purple or brown red colour, as the case may be.

The smell of carbon disulphide should not be repulsive nor fetid. It should give no dark turbidity with a solution of acetate of lead, nor be either acid or alkaline.

Alcohol may be detected by the lower density and by the diminution in volume when the sample is shaken up with equal volumes of glycerine or water. Owing to its higher density and non-miscibility with water, it is possible to store it under water, which should always be done to avoid risk of fire or explosion.

[1] BENZOL C_6H_6.

Molecular Weight, 78; *Boiling-Point*, 80·5° C.; *Melting-Point*, 6° C.

This important substance was discovered in 1825 by

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[1] *Commercial Light Coal Tar Naphtha.*—Consists principally of benzol. When pure it has no disagreeable smell. The smell of impure samples may be removed by agitation with 5 to 10 per cent. of sulphuric acid and subsequent distillation. It may be replaced by, as a gum solvent, toluol, the next higher member toluol boiling at 111° C.; but the higher members, such as xylole, evaporate too slowly for use in varnish-making.

Faraday. Mitscherlich obtained it by heating benzoic acid with an excess of lime:—

$$\left.\begin{matrix}COHO\\C6HO\end{matrix}\right\} = CO_2 + C_6H_6.$$

Hoffmann and Mansfield demonstrated its presence in coal tar, from whence it is now extracted in large quantities. The tar is distilled, collecting apart the first portions, consisting of oils lighter than water. After being washed with dilute sulphuric acid with water, with dilute potash and again with water, they are fractionally distilled, collecting apart the products which pass between each 5°. These different substances being again submitted to fractional distillation yield principally oils passing over between 80° and 85° (benzol), 110° and 115° (toluol), 140° and 145°, 170° and 175° C.

That which passes between 80° and 85° C. is principally benzol, which crystallises on cooling this fraction to − 5° C. The crystals are collected and pressed. The melted crystals are re-frozen, and re-crystallise several times until the melting-point is situated a little above 0° C., and its boiling-point at 80° to 82° C.

According to Mitscherlich, pure benzol may be obtained by distilling 1 part of benzoic with 3 parts of slacked lime, and rectifying the oleaginous liquid which passes over, after previous agitation with potash.

Benzol is a colourless, very refractive liquid. It crystallises at 0° C. The crystals melt at 5·5° C. Its density at 0° C. is ·8991. Its boiling-point is 80·5° C. It is insoluble in water, but dissolves in alcohol and ether, wood spirit and acetone. It dissolves sulphur and phosphorus, fixed and volatile oils, wax, india-rubber, gutta-percha, several resins and some alkaloids. It burns with a bright smoky flame.

GASOLENE (PETROLEUM ETHER).

In the distillation of the crude naphtha obtained from American or Russian petroleum, or of that yielded by the distillation of Scotch bituminous shale, there is obtained a very light, highly volatile, ethereal oil, with an unpleasant but highly characteristic smell, called "gasolene," distilling below 100° C., with a density varying from ·6 to ·7. Its principal use in varnish-making is as a quick evaporating vehicle or thinner for antifouling composition. *But its use is attended with great risk*—statements by interested parties notwithstanding — and varnishes or paints into which it enters should be stored and used *with most extreme caution*, as careless handling may bring about a terrific explosion. They should never be stored in the hold of a ship, and only applied to ships in open air in dry dock far removed from a flame, as a vapour, forming an explosive mixture with air, may run towards it and thus cause an explosion.

Composition.—Gasolene is a highly complex mixture of hydrocarbides, from which the following lower members of the C_nH_2 series have been isolated, *viz.*, normal pentane C_5H_{12}, boiling at + 38° C., and isopentane C_5H_{12}, boiling at + 30° C., and normal hexane C_6H_{14}, boiling at + 71° C., an isomeric hexane as well as normal heptane, boiling at 98° C. It should be stored in drums placed in a wooden receptacle underground. When in use the drums should be immersed in cold water.

[1] PETROLEUM SPIRIT, MINERAL TURPENTINE, MINERAL NAPHTHA, SHALE NAPHTHA.

These are not so volatile, and pass over after the gasolene. Its density is ·730 to ·760, and it boils between 120° and 170° C. American mineral naphtha may be differentiated from the Russian variety by its almost incomplete solubility in acetone, imparting thereto only a slightly yellowish or bluish tint. On the other hand, Russian mineral naphtha dissolves to a slight extent in that reagent. In applying this test the mineral naphtha ought to be neutral, which may be ascertained by treating it with cupreous oxide, which, in contact with acids, forms a copper soap soluble in the

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[1] These consist of a mixture of hydrocarbides, the proportion and nature of which vary with the source. They are much more volatile and inflammable than spirits of turpentine, but unlike the latter have no tendency to oxidise to a lacky residue as they evaporate.

naphtha with a green coloration. Shale naphtha has generally a strong odour of gasolene, due perhaps to imperfect rectification.[1]

SPIRITS OF TURPENTINE.

Technology—American Practice.

Boxing the Pines.—The flow of the crude oleo-resin is stimulated by stripping the tree of its bark and collecting the exuded resin in peculiar V-shaped receptacles called boxes, cut out of the trunk of the tree. The trees are boxed during the fall and winter, the legal limit being from the 15th November to the 15th March. The lower lip of the box is horizontal, the upper arched, and the bottom of the box is about 5 inches below the lower lip and 8 to 10 inches below the upper. The capacity varies from ½ to 1 gallon. Some operators cut larger boxes than others; and, as the trees are often boxed in one to four places at a time, according to the size of the tree, many die off during the first or second year. But medium sized boxes pay best: the flow is as great, and the duration longer.

Cornering the Pines.—As soon as the boxes are cut, the crude oleo-resin commences to flow, and by the time the boxes are all cut and cornered the resin in the boxes is ready for dipping. The boxes are cornered by cutting a strip from each box with an adze.

Chipping the Pines.—As the wound made in boxing soon heals, the surface is again scraped, and the bark is chipped to the height of about 3 feet above the box by means of a steel tool called a "hack," fitted on the end with an iron ball to act as a lever and lessen the labour. The sharp edge of this tool is so turned that it cuts a "streak" from the tree of the exact

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[1] This odour is due to sulphur compounds and may be removed by filtration through dehydrated green vitriol. Pliny was acquainted with a crude method of distilling turpentine. He says: *Epice fit quod pisinnum appellant quum coquitur velleribus supra halitum ejus expansis atque ita expressis probatum maxime ex Brutea penguissima et resinosissima color oleo fulvus.* The essential oil called picinum consists of the steam or vapour of boiled pitch caught on wool fleeces spread over the cauldron and afterwards squeezed out of them. The best is made from the Calabrian as it is the richest and most resinous.

size requisite to start the flow of resin, *viz.*, $\frac{3}{4}$ of an inch wide and $\frac{1}{4}$ inch deep. The operator, standing in front of the box, commences at the top and cuts his streaks obliquely from right to left and left to right, thus giving to them a sort of **L** or **V** shape. The trees are scraped in this manner every eight or ten days, the surface and pores of the wood exposed by the previous hacking having by this time become clogged up by the solidification of the exuded resin. A very small shaving is all that is necessary to restart the flow. If the weather be warm and the streaks chipped regularly, the oleo-resin runs freely, and the boxes are dipped about once every three weeks. The resin commences to flow about the middle of March, and the flow is most copious during July and August and begins to diminish in September or October.

Dipping.—As the resin accumulates in the boxes it is removed by a " dipper," a peculiar sort of spoon or ladle which fits into the bottom of a " box ". The " dipper " is emptied into a bucket, and the bucket, when full, into a barrel, the operator leaving it to drain while he fills another bucket. The barrels when full are hauled by waggons to the still.

As a general rule, the flow of resin from the tree after having been boxed for four or five years is not sufficient to be remunerative; the resin is poor and contains but a small proportion of essential oil. On the other hand, however, the trees are sometimes scraped to such a height that they cannot be reached by the "hack," and are then scraped by an instrument with a long handle called a "puller". But a crop does not yield so much by pulling as by chipping. The higher the boxes are chipped the greater is the yield. They are sometimes, but rarely, wrought to the height of 15 feet, and ladders become necessary to hack the trees afresh. In such instances the resin, on its way to the boxes, solidifies partially on the trunk of the tree and has to be scraped off. When dead, the tree is sawn into lumber or cut up for its tar. Although the tapped wood is less appreciated and often rejected by engineers as weak and faulty, it would appear by direct experiments by the U.S.A. forestry officials that it is, if anything, superior in this respect to untapped timber.

Distillation.—The still in which the crude oleo-resin is distilled presents no peculiar features. It is of the ordinary shape, made of copper and set in brickwork over a furnace, and connected with the usual form of worm condenser.

The barrels containing the crude are rolled up to the still on skids and their contents dumped into the "kettle". When a sufficient quantity has been run in, the fire is lighted beneath the still. As the resin becomes more and more fluid, the dirt and wooden *debris* which float to the top are eliminated by skimming. The still head is then adjusted and well luted on to the mouth of the kettle and connected to the copper worm in a large tank of water. The water in the tub is renewed by pumping, or otherwise, as necessary. The heat is kept up until the resin in the still attains a uniform temperature of 212° to 316° F., and the adventitious water present in the oleo-resin is expelled.

The first runnings, commonly known as low wine, contain, in addition to water, acetic acid, formic acid, ether and wood spirit. When all the adventitious water is expelled, a thin stream of cold water is now allowed to flow into the still so that the temperature may not rise above 316° F., the boiling-point of spirits of turpentine. The distiller knows by the singing of the kettle the condition of the fluid mass within, whether it requires more or less water or if the fire has to be urged or moderated. The spirits of turpentine vapour which is expelled from the oleo-resin in the still passes over with the vapour of water and is condensed therewith in the well-cooled worm, from which the condensed liquids run into a barrel or tub. The distiller tests and examines the distillate from time to time in a proof-glass. The distillate separates in the tub into two layers, a lower layer of water, upon which the upper layer of spirits of turpentine floats. To the tub two taps are adjusted, one at the bottom, for running off the condensed water, and another on the opposite side of the tub, for running off the spirits of turpentine freed from water into another tub, from which it is dipped into casks for the market.

Yield.—Ten barrels of 220 lbs. each yield two barrels of spirits of turpentine and six of rosin.

Rosin is the residuum left in the still after the spirits of turpentine have been distilled off. It is run off from the still whilst still hot and fluid into a large trough, from which it is dipped into barrels, and the barrels coopered for shipment.

The quality of the rosin depends on the nature of the original oleo-resin. This oleo-resin as it flows from the tree during the first year is characterised by its fine white transparent colour. Towards the end of the season it shows a faint straw tinge. The product dipped in the early part of the first season is known as virgin dip, and it is this virgin dip which yields the most pale and most highly valued grades of rosin.

In the second year the oleo-resin is termed yellow dip. Each succeeding year it becomes darker in colour and poorer in essential oil, *i.e.*, spirits of turpentine. In the fourth and following years the oleo-resin is very dark, and the rosin produced therefrom is of the lowest grade—deep brown to opaque black.

Brands.—The different grades of rosin on the market are branded as follows :—

WW	Water white.
WG	Window glass.
N	Extra pale.
M	Pale.
K	Low pale.
I	Good No. 1.
H	1.
F	Good No. 2.
E	No. 2.
D	Good strain.
C	Strain.
B	Common strain.
A	Black.

An operator works, at the least, ten crops for four years, the plant for which costs 1000 dollars, and the expenses come to 24,000 dollars, or 600 dollars per crop per annum, in addition to a rental of 60 to 100 dollars per crop. A crop means 10,000 boxes, and involves, say, 5000 trees, spread over 200 acres, producing the first year:—

	Barrels.			
Dip	280	yielding	spirits of turpentine	2000 galls.
Scrape	70		rosin	260 barrels.

The yield is less, the quality worse, and consequently the profits smaller, in each succeeding year.

The annual value of pine products produced in U.S.A.	12,000,000 dolls.
Annual production of spirits of turpentine	15,000,000 galls.
of which there are used in the—	
1. Paint and varnish industry	7,000,000 galls.
2. India-rubber industry	4,500,000 galls.

German Methods of Distilling Turpentine.

In Germany crude turpentine is distilled by steam alone, as follows: It is first run into an elevated filtering vessel, the conical bottom of which is steam jacketed and the exterior fitted with a movable wire gauze strainer to free the resin from adventitions matter. Here the crude resin receives a preliminary heating so as to drive off included moisture. It may be stirred up from time to time with the stirrer (2) as occasion requires. It is run off by the pipe (3) into the steam-jacketed still, which holds, when two-thirds full, five tons of oleo-resin. In addition to the steam-jacket, which is fed by (7), direct and open steam is led in by the pipe (5), which terminates in the bottom of the still in a 4 to 8 way piece. The open ended are much to be preferred to perforated pipes, the latter being very liable to become choked. At the end of the delivery pipe from the still head, which is about 40 inches high, a steam injector (16) drives the products into the condenser (19), whilst a jet of water (17), opening just below the steam-jet, cools the distillate. From the condenser the mixture of condensed spirits of turpentine and

1. Filtering Vat.
2. Stirrer.
3. Pipe to Still.
4. Water Effluent.
5. Direct Steam Pipe.
6.
15. } Steam Feed.
7. Steam Feed to Jacket
8. Draw-off for Resin.
9.
11. } Steam Outlet.
12.
10. Outlet from Still.
13.
17. } Water Intake.
14. Outlet for First Runnings.
16. Steam Injector.
18. Water Outlet.
19. Condenser.
20. Outlet for First Runnings and Spirit.
21. Outlet for Main Distillate.

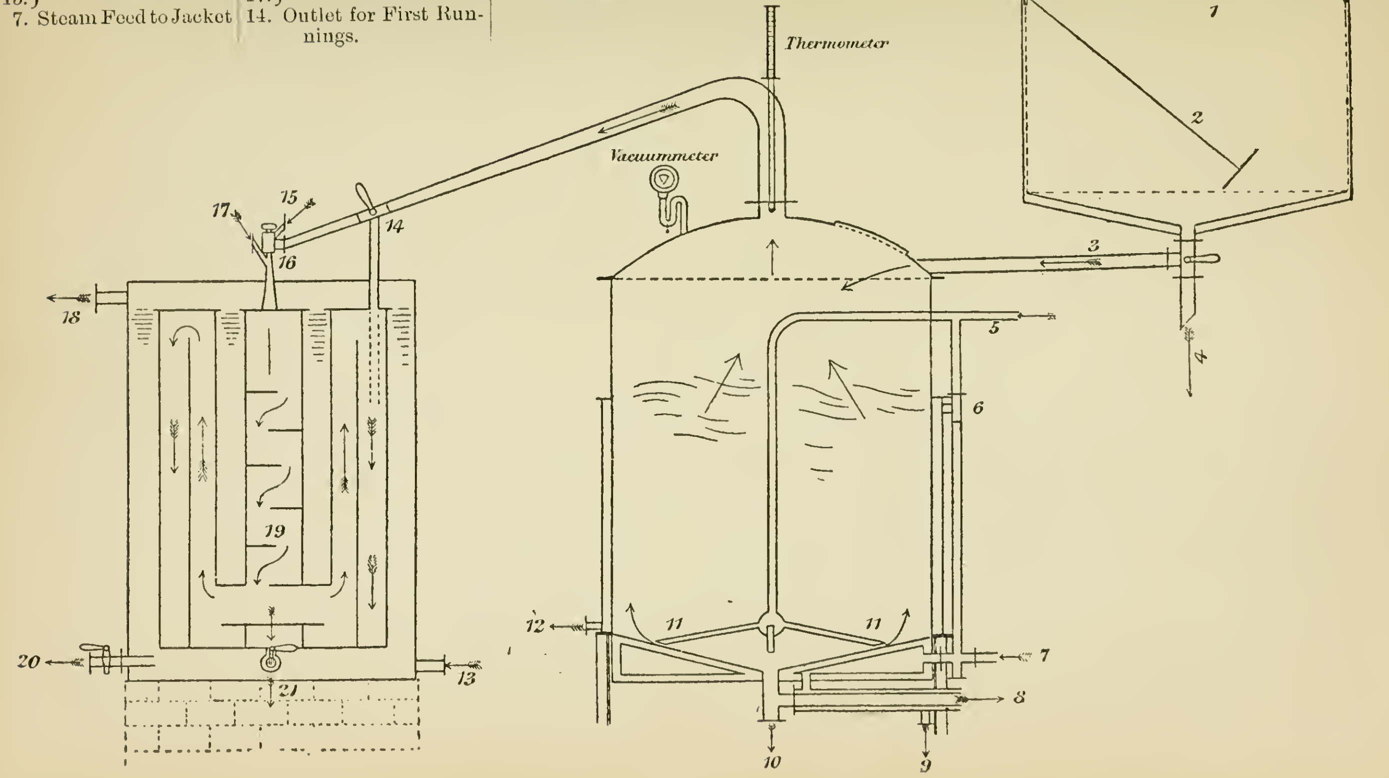

Fig. 1.

water runs into a storage tank, where the spirits and water are separated on the Florentine principle. The advantages of this method are, that, owing to the use of the steam injector, a partial vacuum is created, which greatly facilitates the filtration of the oleo-resin and the automatic feeding of the still from the preliminary heater. As the distillation is effected at a much lower temperature (initially 20° to 25° C., and finally 50° to 60° C. lower than the usual method of distillation in contact with water), the final products are therefore necessarily of a very superior grade.

Rosin Distillation—French Method.

The rosin is distilled in a cast-iron vessel 6 feet 9 inches deep, fitted with a still head to carry the vapours to a condenser. This is a very small-sized still, which will only take a charge of 1 ton 7 cwt., whereas in England we have stills which take a charge of 20 tons and upwards of American rosin. The rosin is allowed to melt before fixing on the still head, so as to avoid bumping and allow any water contained in the rosin to escape. As soon as the melted rosin becomes quiescent, the still head is fixed in its place, and carefully luted with cement and clay.

Over in France two methods of distillation are in vogue: (*a*) the quick process, lasting four hours, and yielding strong-smelling, heavy, thick, viscous oils with a density of 0·980 to 0·990; (*b*) the slow distillation process, which lasts for twelve hours, and which gives lighter and more limpid oils, of density 0·950 to 0·960. The comparative results obtained in the two processes are illustrated in the accompanying table:—

Quick Distillation—1350 parts Rosin yielded :	Slow Distillation—1325 parts Rosin and 13 parts Lime yielded :—
30 parts rosin spirit.	30 parts rosin spirit.
975 ,, ,, oil, blonde.	250 ,, ,, oil, blonde.
100 ,, ,, ,, blue.	100 ,, ,, ,, white.
221 ,, pitch and loss.	160 ,, ,, ,, blonde.
	140 ,, ,, ,, blue.
	80 ,, ,, ,, green.
	278 ,, pitch and loss.

Lime yields paler oils and facilitates distillation, the distillate coming over in a more even and regular manner. The oil is then clarified by subsidence in long vertical tanks, after which it is decanted into shallow sheet-iron tanks and exposed to the action of the sun and air to decolorise it. The tanks are covered with glass frames to keep out dust and dirt. However practicable this method may be in France, it would be impracticable in this country, where we work upon much larger quantities ; and, again, the sun's rays are not so powerful here as there. Another method is to heat the oil to 40° C. and agitate it with a solution of caustic soda. Water is then added, and the temperature maintained for some time at 40° C. The alkaline solution is run off, the oil washed with water, and again exposed to the action of the sun.

They make their oil for lubricating purposes and for printers' ink in the latter manner.

Siccative Rosin Oil.

When required for paint purposes they make a siccative rosin oil as follows :—

1. Agitate the oil with 2 per cent. sulphuric acid in a lead-lined tank.
2. Wash with boiling water in a current of steam.
3. Macerate the oil for two hours over pulverised quick-lime.
4. Distil the oil over same quick-lime.
5. Wash with boiling water in a current of steam.
6. Boil for two hours with 8 per cent. of dry white lead.

The oil becomes siccative. It is left to settle and then exposed to sunlight to bleach it.

Rosin Distillation—German Practice.

According to Bolleg, the most suitable stills for rosin distilling are undoubtedly large cylindrical, cast-iron vessels, holding at least five tons when three-parts full, *i.e.*, 74 by 94 inches, without counting the lid. They are made with a rounded bottom, and are either cast in a single piece or, better still, composed of two parts—the cylindrical portion, strengthened below, and advantageously made of boiler plate, and the still stronger bottom piece. This form is advisable on account of the large heating surface it affords, and the quieter and more regular distillation thereby rendered possible, as well as the greater strength and durability of the still itself. Economically speaking, the division of the vessel into two parts is a great advantage, since it enables the casting to be more carefully performed, and also permits of the bottom being renewed when worn out or damaged; and there is no danger of leakage, provided the flanged joint be properly packed with asbestos and red lead, etc.

The cast-iron domed lid (12 inches high) is attached by screwing, and is fitted with a manhole at the back, another at the side, for emptying, and a safety-valve in the centre, in addition to a flanged sleeve, 10 inches high and about 14 inches wide, for the attachment of the still-neck. This latter is preferably made of copper, and should—in order to obtain a thin distillate, as far as possible free from resin—be at least 40 to 60 inches high, and is fitted with baffles to cause the vapours to take a spiral course. The descending limb gradually tapers until, at the condenser, its internal diameter is only 2½ to 3 inches. The condenser is an important part of the plant, a simple coil, however well cooled, being insufficient when products other than mere waggon-grease oils are in view. In fact, a double condenser, similar in construction or principle to that used in the distillation of turpentine should be employed, since it is evident that a superior distillate for refining purposes will be obtained if the first-run acid-water, resin-spirit, and intermediate oil—as well as the strong-smelling spirit and light oil distillates and gases formed during the entire course of the operation—be led through a different condenser than the main distillate (the light or "blonde" oil). A condenser of large capacity for the chief distillate, and a smaller one for the bye-products, are therefore desirable, to which should be added a third coil, specially reserved for the "green oil" coming over at the end of the distillation.

A process of simple fractional distillation will generally suffice, but for large installations it is frequently desirable to work with a partial vacuum—6 to 10, or more rarely 20 to 35, inches of mercury—*i.e.*, with continuous exhaustion of the vapours, a means ensuring a more perfect fractionation of the strong-smelling spirit and light oils. In such cases a double vacuum-receiver with exhaust is required, and this applies specially in cases where drying oils are to be produced, being also advisable for sundry purposes in rectification. In such event the sufficient decomposition of the distillate is ensured by the addition of from 3 to 5 per cent. (in special cases 10 per cent.) of fresh, powdered lime, and complete distillation to dryness.

As fuel coal is used, being supplemented, in the later stages of distillation, with the fixed gases given off in the process, the hearth should be of large size, and not at too great a distance from the open arch of firebrick which serves to protect the bottom of the still from the direct impact of the flame. Lump coal, intermixed with nuts, is used, the proportion of the latter being increased as distillation pro-

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Fig. 2.

1. Still Neck with Baffles.
2. Gas Pipe, leading to.
3. Fire.
4. Rosin Still.
5. Steam Feed Pipe.
6. Still Neck.
7. Fractionising Dephlegmator.
8. Outlet for First Runnings, and Light Distillate.
9. Water Outlet.
10. Condenser.
11. Outlet for Gas.
12. Outlet for Light Distillates.
13. Water Intake.
14. Outlet for Main Distillate.
15. Air Valve.
16. Injector.
17. Pale Rosin Oil Tank.
18. Blue Oil Tank.
19. Light Distillate Tank.

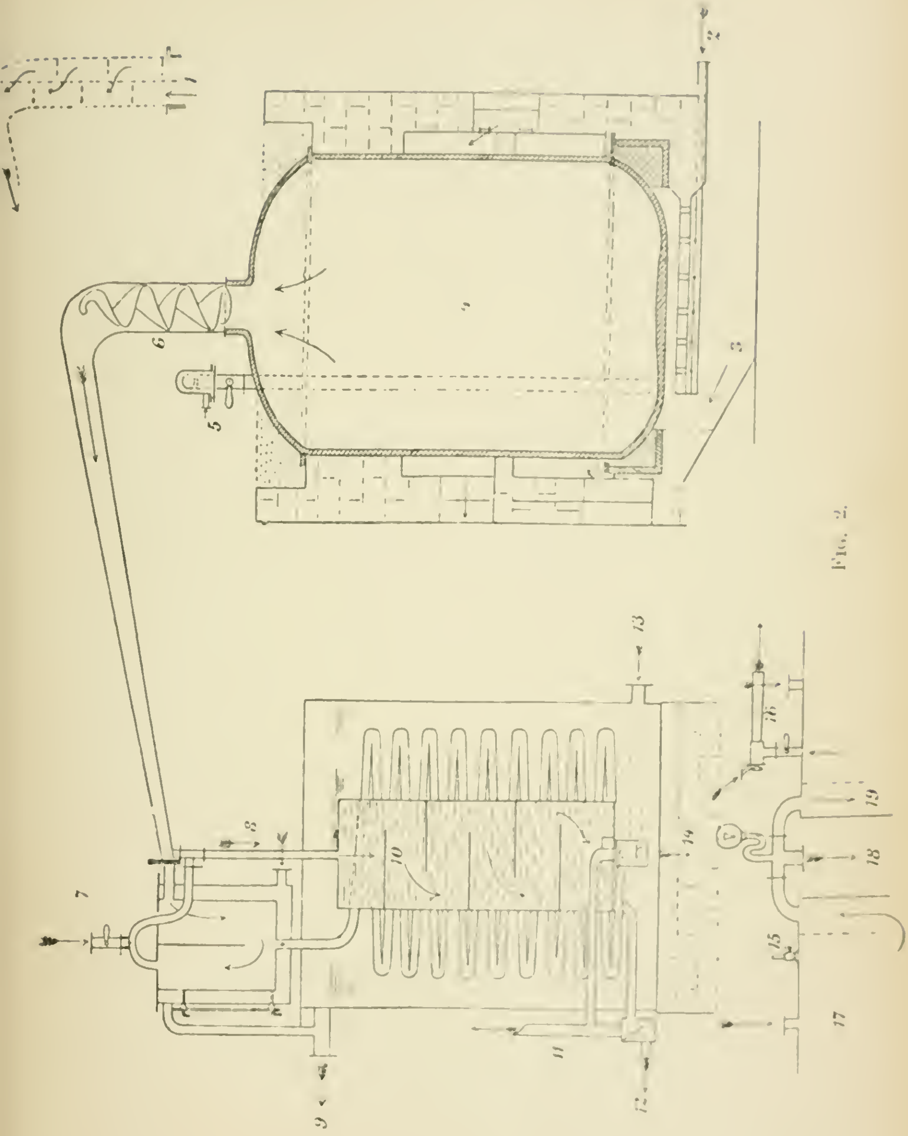

Fig. 2.

gresses. The employment of gas will save about one-third of the coal bill, and, moreover, enables a considerable quantity of pinolin and similar light essences to be recovered, so that this method of combined firing is a profitable one. Side flues are 6 inches wide and 16 to 18 inches high—one in a forward and one a reversed direction on each side—should be provided for heating the walls of the still, and fitted with dampers for shutting off the fire at will.

Except in French distilleries, American "good strained" rosin is the chief raw material treated. The barrels are rolled or hauled up from the store to the manhole of the still, where they are broken and the resin shovelled into the vessel, the staves and heads of the casks being subsequently steamed to recover the still adherent particles of resin. The fire is meanwhile kindled, but is not made too fierce at first, otherwise there is danger of the melting resin frothing up and boiling over, especially when it contains much water. Until all is melted and frothing has ceased, the still must be left open, but, as this may delay the distillation for several hours, recourse is had to paraffin oil, of which some 2½ to 5 cwt. are added per charge (5 tons) of rosin, the effect of this being to remove all danger of frothing over, and, consequently, to allow the still to be closed at once. Moreover, this paraffin oil ensures better and lighter quality products, freer from resin, and more suitable for all usual purposes, in addition to increasing the yield of "blonde" oil by about 5 per cent.

The products obtained from 5 tons of rosin distilled in such a plant will average: 6 to 8 per cent. of resin spirit (pinolin), including acid-water; 50 to 55 per cent. of pale rosin oil (blonde oil) for refining; 20 to 15 per cent. of blue oil for the same purposes or for waggon-grease making; 6 to 7 per cent. of green oil for working up into waggon-grease and carbolineum; *i.e.*, a total of 82 to 85 per cent. of distillate, leaving 15 to 18 per cent. of residue, of which

5 to 7 per cent. is utilisable gas and the remainder almost worthless.

Since it is more economical in point of time, and also as regards the quality of the products, to carry on the distillation of the final fractions (green oil) in a separate vessel, the work is arranged as follows:—

The still is filled, say, in an afternoon, and the rosin will be thoroughly melted by 7 P.M., whereupon the paraffin oil is added, and the still closed and left all night with a banked-up fire. At 4 or 5 A.M. next day the fire is made up, so that by half-past six at the latest the still begins to run, and the operation will progress all day, the final runnings of blue oil being over by 8 P.M., the paraffin oil enabling the distillation to be forced, and the high still-neck with its internal baffles ensuring the proper decomposition of the distillates. If vacuum distillation is performed the operation will go on much more rapidly, and will be completed a couple of hours earlier. The liquid residue can then be removed by an injector into the separate still employed solely for distilling the green oil, and the main still be re-charged the following morning with ready-melted rosin (where turpentine is distilled the residue from the retorts can be run into the still after well ventilating the latter for about a quarter of an hour to prevent risk of explosion) and worked all day, the operation concluding on this occasion at 9 or 10 P.M. This arrangement dispenses with the inconvenience of working all night, and enables the stills to be charged four times a week, besides effecting an improvement in quantity and quality of product and a saving in labour, repairs, etc. The stills are cleaned out once a fortnight, except where lime is used, in which case, as the distillation has to be continued to dryness, the stills must be cleaned out every time.

The residue from eight stills is united and distilled in smaller cast-iron vessels over direct fire, and either to complete dryness or else to within a few per cent. of green oil, the residue in such latter event being a more or less solid pitch suitable for working up into iron lacquer, shoemakers' pitch, and similar purposes. To prevent incrustations, the stills should be cleaned out after every second distillation.

Refining Rosin Oil.

The apparatus consists of a washing tank 120 × 60 × 60 and a bleaching tank of similar size. Five tons of blonde oil are placed in the former to allow the acetic-acid water to settle, about four parts of hot water are added, and the whole boiled by direct steam. When freed from acid water the oil is again boiled by direct steam, and treated with 4 to 5½ per cent. of soda lye of 36° to 40° B. sprinkled over the surface, the heating being continued until a sample shows the oil to be free from resin and acid, whereupon the steam must be instantly shut off or the product will assume a dark colour and turbidity due to dissolved rosin soaps; the steam coil should then be at once lifted out of the liquid.

Immediately after the steam is shut off, the steam coil, with its attached feed pipes, is lifted up out of the oil by means of the stuffing-box arrangement in the head of the vessel, and is cleared of liquid by a momentary blast of steam. This simple but valuable improvement was recently introduced by Bolleg, and its importance will be appreciated by those who work with this class of apparatus, the cleansing of the coil after alkali washing, and before the after-washing is begun, entirely obviating an inconvenience hitherto prevalent in the refining of resin oil, whether the washing was performed in similar vessels or in those fitted with agitators, *viz.*, that by the presence of small quantities of resin soaps adhering to the stirrers or the coil, the oil was always, to some extent, contaminated darkened in colour, and frequently rendered persistently turbid during the subsequent washing with water. Since the only feasible method of avoiding this inconvenience was the tedious plan of having a separate washing tank for the second operation, the new appliance saves the loss of time in transferring from one vessel to another, and the cost of a second tank, besides yielding a much cleaner, paler, and always clear oil.

After leaving the mixture of oil and lye at rest for about an hour to enable the latter to subside, the lye is drawn off into a vessel until pure oil begins to run out of the tap. The steam coil is then lowered again and direct steam passed, gradually and carefully, into the oil, warm (50° to 60° C.) water, to the extent of about one-third the volume of oil present, being at the same time run in from the lye and water tank. Here again care is necessary not to boil the mixture too violently or too long, a temperature of 105° C. being the maximum and the operation continuing only until a test sample shows that the water (like the lye in the former washing) settles down quickly and leaves the oil clear. Under careful and accurate manipulation, the water will subside completely within the hour, and in such event—provided, as should be the case, the oil is clear and the washings do not appear too dirty and soapy—no further treatment will be required, and the oil can be drawn off through the upper tap into the bleaching (oxidising) apparatus.

In order that this last-named apparatus may be caused to yield the best results, and furnish a pale, odourless oil, free from resins and acids, the following procedure, lately originated, must be adopted: The oil, occupying a depth of 30 to 40 inches in the vessel, and previously warmed by indirect steam to a temperature of about 60° C., is treated for two to three hours with a current of air at 60° to 80° C., forced in by means of a blower, through a finely-perforated coil. At the same time the light oil vapours and gases evolved at the surface should be drawn off by a ventilator and discharged through a wide pipe into the condenser—an operation which is indispensable to this process, but which is often erroneously omitted. By means of increasing the supply of steam and air, the temperature of the oil is raised to 115° C. in the course of the next hour and is there maintained for 15 to (maximum) 30 minutes to remove the fluorescence (bloom) of the oil, which thus loses its bluish cast ; at the same time the evolved vapours acquire a peculiar pungent odour recalling that of camphor. The exhaustion of these vapours is not restricted to the time the oil remains in the bleaching vessel, but must be continued in the storage and cooling tanks until the temperature of the oil has receded to about 30° C. This is found to be indispensable for the production of fine, clear and inodorous oils. The use of open bleaching pans has proved both troublesome and actually dangerous ; on the other hand, an addition of 10 to 15 per cent. of brine to the oil—the water lost by evaporation being replaced by hot water either every half-hour or twice during the final hour—has been found to be highly commendable, since it prevents the renewed formation of oxidation and empyreumatic resins, and absorbs the substances which would go to form these resins, its colour being thereby changed to brown, though it settles down without difficulty. The new-finished oil is actually free from resin and acids, so that, unlike oils bleached by other methods it no longer reacts with caustic soda. This is the sole method available for the production of resin oils drying without cracking or tackiness, and suitable for varnish and lacquer-making.

The same method as for crude blonde oil is employed in working up rectified or double distilled blonde oil for special purposes, *e.g.*, lubricating oil, the vapours being continuously drawn off and salt water added, the only differences being that $1\frac{1}{2}$ to 2 per cent. less caustic soda lye (*i.e.* only 3 to $3\frac{1}{2}$ per cent.) is needed, and that the temperature of the oil at the close of the bleaching process is not allowed to rise above 105° C. For refining blue oil, the same method is pursued, 3 to 4 per cent. of lye being used; but to obtain a clear, transparent product, not too deep red in colour, it is necessary that the oil should be treated, or at least washed, while fresh from the still, and worked up immediately after rectifying. Under these circumstances a fine orange-yellow to pale red oil, suitable for many purposes, is produced. The practice of employing sulphuric acid to make the oil water-white is condemned, a better result being attainable by the addition of quick-lime, or caustic soda, and animal or wood charcoal, to the oil during the rectification *in vacuo*, time and expense being also saved.

The oils prepared by the foregoing method are, on account of their freedom from acids and resins, and their exceedingly mild aroma, far more suitable for their various uses than those obtained in the ordinary manner, and can consequently be utilised to better advantage. The various grades of rectified oil can be employed, according to quality, for making varnish, lacquer paint, printers' ink, lubricating oil, preparations for boring and planing tools, polishes, wood-work, leather work, as preservatives, for making carbo-lineum, artificial train oil and degras, in the linoleum, insulating material, soap and perfumery industries, and for medicinal purposes.

PROPERTIES OF ROSIN OIL.

According to Renard, rosin oil consists of :—

	Per cent
Diterebenthyle $C_{10}H_{15}$ - - - - - -	80
Diterebenthylene $C_{10}H_{14}$ - - - - - -	10
Didecene $C_{20}H_{36}$ - - - -	10
	100

Diterebenthyle $C_{10}H_{15}$ is a colourless oil, boiling at 333° to 336° C., and of density 0·9688 at 18° C., rotating the planes of polarised light 0·590 to the right (dextro-rotatory). Its index of refraction is 1·53. In a thin layer it absorbs 10 per cent. of oxygen from the air. Concentrated sulphuric acid converts it into a sulphuric derivative. It is attacked by chromic acid, nitric acid and bromine, like the terpene hydrocarbons.

Diterebenthylene $C_{10}H_{14}$ is a thick colourless oil, slightly fluorescent, boiling at 345° to 350° C. Its density is 0·9821 at 120° C. It is dextro-rotatory (+ 4°). It does not change on exposure to the air, and neither sulphuric or hydrochloric acid have any action upon it.

Didecene $C_{20}H_{36}$ is a colourless non-fluorescent oil, boiling at 330° to 335° C., and possessing a density of 0·9362 at + 12° C. It is levo-rotatory (− 2°). It does not change on exposure to the air, and neither sulphuric nor nitric acid have any action upon it.

Rosin oil is not saponifiable. Its density varies from 0·973 to 0·981. Sulphuric acid—one drop spotted on ten of oil—gives a brownish red nucleus, quickly passing to black.

Reactions.—Bichloride of tin produces a blood-red coloration, quickly passing to purple. Nitric acid attacks rosin oil in the hot, with production of nitrous vapours. The product when thrown into water changes to a yellow waxy mass. Rosin oil is soluble in alcohol and glacial acetic acid. It is dextro-rotatory (+ 30°) and rarely levo-rotatory (− 8°24). Mineral oil is without action on polarised light.

Although the density always affords sufficient data to differentiate between mineral oil and rosin oil, we can confirm our inference by the elaidin test: in the presence of rosin oil a deep red coloration is produced, whilst mineral oils remain unchanged. Glacial acetic acid dissolves rosin oil, but mineral oil is insoluble in that reagent.

The iodine number rarely goes above 14 in mineral oils, whilst it may go as high as 43 to 48 in rosin oils. With shale oil, however, the iodine number may rise to 21.

Storch-Liebermann Test.—In his work on quinovic acid, Liebermann has shown that a solution of that acid in glacial acetic acid, when treated with a little concentrated sulphuric acid, gives a beautiful red coloration, which is likewise obtained with pimaric and sylvic acids, these acids being present in rosin oil. Storch, *Dingl. Poly. Jn.*, t. 267, p. 28, proposes the Liebermann reaction as a test for rosin oil in oils and fats.

If we add to a few drops of rosin oil a cubic centimetre of glacial acetic acid and then one drop of concentrated sulphuric, we obtain a red violet coloration, which passes rapidly to brown.

TERPENES.

Chemical Constitution.—Spirits of turpentine, from whatever source, is an essential oil which consists of a mixture of isomeric hydrocarbides, which all agree in having the same centesimal composition, *viz.*, that represented by the formula $C_{10}H_{16}$. Some of these hydrocarbides, which are known as terpenes, are natural products, and enter into the composition, wholly or partially, of numerous essential oils. Many terpenes are, however, artificial products, which may or may not exist in nature.

Amongst the latter, mention may be made of camphene, which is a solid body. But more often the terpenes are liquid substances lighter than water, which rotate the plane of polarisation, and the bulk of which exist in both dextro and levo-rotatory modifications, boil between 150 and 200 C., and under the action of heat and reagents become altered in density, boiling-point, rotatory power, and even in chemical composition. Amongst the latter class of phenomena, mention may be made of the great affinity of the terpenes for halogenhydride, especially hydrochloric acid, resulting in the formation of a well-defined class of compounds known —but erroneously so—as artificial camphors.

The whole of the members of the class of trees known as *Coniferæ* yield *turpentine* in greater or less abundance, which, by distillation with water, yields *spirits* of turpentine, and leaves behind in the still a residue of common rosin. But the spirits of turpentine from one species of cone-bearing trees differ in both physical and chemical properties from that yielded by another species.

Berthelot investigated the properties of the spirits of turpentine yielded by the *Coniferæ* of different countries, and applied the following terms to their essential constituent :—

Country.	Principal Constituent.	Conifer.
France.	Terebenthene.	*Pinus maritima.*
America.	Australene.	*Pinus australis.*
Russia.	Sylvestrene.	*Pinus sylvestris.*

Terebenthene is, according to Berthelot, obtained from French spirits of turpentine by neutralising it with carbonate of soda and then distilling *in vacuo.* It is a colourless mobile liquid, boiling at 161° C., with a density equal to 0·864 at 16° C. It is levo-rotatory [$a = -42^\circ{\cdot}3$]. When heated to 250° C. in a sealed tube, it is converted into a mixture of hydrocarbides—the first an isomer isoterebenthene $C_{10}H_{16}$, boiling at 177° C. ; and the second, a polymer isoterebenthene $C_{20}H_{32}$, a viscous liquid.

Armstrong believes American spirits of turpentine consists of two terpenes, one levo-rotatory, and identical with that which predominates in French spirits of turpentine—levo-terebenthene; and the other, the chief ingredient, dextro-terebenthene, which is also found in a state of great purity in *Pinus khasyana*, a tree indigenous to British Burmah.

Notwithstanding the classical investigations of Berthelot, together with those of Tilden, Armstrong, Wallach and others, the lines of demarcation and differentiation between the terpenes are far from being well-defined and distinct.

The following classification may, however, be taken to represent the latest results of scientific investigation under which the terpenes may be separated into three main groups :—

(A) Those which, provided they be not in the interval converted into isomers, can combine with only one molecule of halogenhydride, ex. 9, HClHBr,HI, to form monohydrochlorides $C_{10}H_{16}HCl$: *pinene, camphene, fenchene.*

1. Pinene is the chief constituent of American spirits of turpentine, oil of juniper,[1] eucalyptus oil, etc. It is a colourless mobile liquid with a characteristic odour, and a density, according to Wallach, of 0·860, boiling between 159° and 160° C. When dry hydrochloric acid gas is passed through it, combination ensues, with liberation of much heat and the formation of a crystalline substance $C_{10}H_{16}HCl$ pinene monohydrochloride, possessing an odour very similar to natural camphor, and hence, but erroneously, known as artificial camphor. Although very similar in appearance, it may be easily distinguished from the latter by the abundant fumes of hydrochloric acid gas which it gives off on ignition. A white solid body termed *camphene* is said to be obtained by heating pinene monohydrochloride under pressure with caustic alkali, but it is very difficult to obtain this terpene free from halogenhydride.[2]

By exposing pinene to sunlight in contact with water, a crystalline compound, termed by Armstrong sobrerol $C_{10}H_{18}O_2$, is produced. Under the influence of alcohol and nitric acid, or when left for a long time in contact with water, pinene is converted into a crystalline hydrate, ter-

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[1] Obtained by distilling the crushed bays of juniper ; density 0·84 to 0·88; boiling-point 155° to 161° C.

[2] See also "Pinene Monohydrochloride," p. 134.

pene hydrate $C_{10}H_{16}3H_2O$, insoluble in spirits of turpentine, sparingly soluble in carbon disulphide, chloroform, ether, slightly soluble in cold but rather more so in hot water.

Pinene exists in two isomeric modifications, which only differ from each other by the one being dextro-rotatory and the other levo-rotatory. The physical differences of the commercial varieties of spirits of turpentine depend upon which of these predominates. An inactive pinene may be obtained by heating pinene nitroso-chloride with aniline, NOCl being eliminated. A similar inactive substance may be obtained by mixing equal quantities of dextro and levo-pinene. Pinene oxidises in contact with air, and the rotatory action of its oxidation products are the same as that of the original pinene.

2. Camphene is a solid body produced artificially by heating pinene monohydrochloride with dry soap. It is said to be oxidised to camphor by chromic acid mixture (see p. 129).

3. Fenchene, an artificial product of no importance to the painter.

(*B*) Those which combine with two molecules of halogen-hydride but not with nitrous acid, dipentene, sylvestrene, or terpinolene to form dihydrochlorides $C_{10}H_{16}2HCl$.

1. Limonene—the dextro-rotatory variety—is present in oil of orange rind, and together with pinene in oil of citron. Levo-limonene is present with levo-pinene in oil of fir cones.

2. Dipentene, cinene, inactive limonene, boiling-point 175° to 176° C., is present in oil of camphor and in Russian and Swedish oil of turpentine, in cineol, in oleum cinae, and may be prepared artificially by heating pinene, camphene, sylvestrene, or limonene 250° to 270° C. for several hours, as well as by the abstraction of 2HCl from its dihydrochloride, and in several other ways. It has a pleasant essence of lemon smell, and is altogether more stable than

pinene. It readily forms a dihydrochloride with hydrochloric acid and a crystalline tetrabromide with bromine of melting-point 125° C.

3. Sylvestrene, specific gravity, ·846 at 20° C., boiling-point 175° C., is the dextro-rotatory (+ 19·0°) and principal constituent of Russian and Swedish spirits of turpentine, and is one of the most stable of the terpenes. It gives a magnificent blue colour reaction with acetic anhydride and concentrated sulphuric acid. Unlike that of pinene, its monohydrochloride is liquid. Its dihydrochloride $C_{10}H_{16}2HCl$ is isomeric with dipentene dihydrochloride. Under the influence of air and sunlight, it oxidises more rapidly than pinene. Neither its levo-rotatory isomer nor any inactive varieties have been discovered.

4. Terpinolene, an artificial product, of only theoretical importance.

(C) Those which form nitrosites, terpinene and phellandrene.

1. Terpinene, an artificial product, produced like terpinene by isomeration of pinene and limonene, yields a nitrosite.

2. Phellandrene, dextro-phellandrene, occurs in water dropwort.

Phellandrene aquaticum and levo-phellandrene in eucalyptus oil yield a nitrosite.

	M. Pt.	B. Pt.	Bromides M. Pt.	Hydrochlorides M. Pt.	Nitritis M. Pt
1. Pinene - -	Liq.	159°-160°	Br_2 170	+ HCl 125	—
2. Camphene -	50°	160°-161°	—	,, decomp.	—
3. Fenchene -	Liq.	158°-160°	Br_2 liq.	,, liq.	—
4. ± Limonene -	Liq.	175	Br_4 101	+ $_2$HCl 50	—
4*a*. Dipentene -	,,		,, 125		—
5. Sylvestrene -	,,	175°-176°	,, 135	,, 72	—
6. Terpinolene -	,,	185°-190°	,, 116	,, 50	—
7. Terpinene -	,,	180	—	—	155
8. Phellandrene -	,,	about 170 !	—	—	102

Constitution of Commercial Varieties of Spirits of Turpentine.

1. American consists essentially of dextro-pinene. The specific gravity is 0·864 to 0·868. Dextro-rotatory, varying greatly from + 8° to + 16°, that of pure dextro-pinene being 21·5. Its affinity for atmospheric oxygen is greater than that of French turpentine. The air oxidation products have a variable rotation, and may be separated into two isomers, the one dextro and the other levo-rotatory, which by being mixed in equal quantities produce an inactive variety.

The spirits of turpentine used for American home consumption would appear to be grossly adulterated with petroleum products by the retailer or middlemen. That exported to this country would, however, appear to be of uniformly good quality.[1]

2. French spirits of turpentine. Gravity same as American. It is levo-rotatory, different samples giving generally − 30°. Its boiling-point is more constant, and its behaviour is altogether more uniform than the American spirit.

3. Russian spirits of turpentine—the oxidation product of which forms the basis of the disinfectant known under the trade name of "Sanitas"—is very similar to American spirits of turpentine. Like the latter it is dextro-rotatory, and varies from + 15° to + 23°, the deviation of pure sylvestrene being + 19°. Its density also varies from 0·864 to 0·870. It begins to boil about 155° C., the bulk of the distillate passing over between 172° and 176° C., leaving a residue which does not distil below 180° C. It has an unpleasant odour, simultaneously recalling that of Stockholm tar[2] and rosin spirit. Owing to this rank smell neither Russian-Polish nor German spirits of turpentine are in favour with painters, upon whom their toxic effect is greater than that of the

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[1] Spirits of turpentine dissolves in 10 to 12 parts of 90 per cent. alcohol—incomplete solution indicates benzene.

[2] Obtained by distilling the "blocks" and roots of pine trees.

American spirit, producing in some individuals headache and great lassitude. Hence its use is not general, either as a solvent for varnishes or as a paint vehicle.

Adulteration is indicated by alteration (1) of behaviour to polarised light; (2) of density and lowering of flash-point (96° F.), and a greasy residue on evaporation.

Kingzett, the patentee of "Sanitas," has given the name of camphoric peroxide $C_{10}H_{14}O_4$ to the air oxidation product of Russian turpentine. This oxidised product, when heated with water, is said to yield camphoric acid $C_{10}H_{16}O_4$ and hydrogen peroxide.

GENERAL PROPERTIES OF SPIRITS OF TURPENTINE.

Reactions.

Action of Air.—Recently distilled spirits dries quickly and free from tackiness; old spirits dries slowly, leaving a coat which remains tacky for a long time.

When spirits of turpentine is exposed to the air in an imperfectly closed vessel it gradually absorbs oxygen. Assuming a deeper and deeper tint of yellow, it becomes more viscous in proportion, and finally resinifies.

During this slow oxidation process ozone is generated and dissolved by the oil which then possesses oxidising properties. It should therefore be stored in air-tight vessels plated with tin or tea lead to prevent discoloration. Formic and acetic acid are also said to be produced during the oxidation process.[1] This oxidation goes on much more energetically when spirits of turpentine is heated in contact with air along with litharge.

Concentrated nitric acid acts so energetically on spirits of turpentine that the mixture may take fire. But when it is subjected to a long boiling with the dilute acid, terephthalic acid and other bodies are produced.

When treated with a mixture of alcohol and nitric acid,

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[1] The amount of oxygen absorbed is an index of purity; it varies with the variety. Good spirits is neutral to test paper.

spirits of turpentine assimilates the elements of water, and is converted into terpene hydrate.

Terebene.—When spirits of turpentine is mixed with $\frac{1}{20}$th of its weight of concentrated sulphuric acid, and the whole well stirred, it is converted into an isomeric terebene and a polymeric colophene or diterebene, which may be separated from each other by fractional distillation.

Terebene $C_{10}H_{16}$ is a colourless liquid with a thyme-like odour, of density ·864 at 8° C. It boils at 156° C., and is optically inactive. It combines with hydrochloric acid to form a liquid monohydrochloride. This substance—used in medicine as an embrocation—must not be confounded with the "trade" liquid drier "terebine," with which it has but little or no analogy.

Colophene $C_{20}H_{32}$ is a colourless liquid by transmitted, but which fluoresces strongly with reflected, light.

With the exception of the liquid and solid monohydrochlorides, which are good solvents for rubber, etc., the compounds which the terpenes form with reagents are of little practical value for varnish-makers. A solution of gum resins in the liquid monohydrochloride, or of the solid in an alcoholic or spirits of turpentine solution of resin, is a specific against the ravages of white ants.

Solid Pinene Monohydrochloride.

The solid body consists of perfectly colourless crystals, with a smell analogous to camphor; it melts at 115° C., and boils with decomposition at 165° C. The tension of its vapour at the ordinary temperature is so great that it sublimes like camphor in the form of small brilliant crystals in the bottles in which it is stocked. It is insoluble in water, and gyrates on the surface of that liquid like ordinary camphor.

This body—known under the name of artificial camphor—was discovered in 1803 by Kindt. It is prepared by passing a current of dry hydrochloric acid gas through spirits of turpentine kept at a regulated temperature. The liquid darkens, and in course of time deposits crystals having the formula $C_{10}H_{16}HCl$. There is simultaneously produced a liquid monohydrochloride said to be of the same composition. The solid is separated from the liquid by filtration and pressure in the cold.

LIQUID PINENE MONOHYDROCHLORIDE.

This substance, when well prepared, is a colourless water white mobile liquid of specific gravity ·960 at 15° C., possessing solvent properties analogous to those of spirits of turpentine, but it is a far more energetic solvent for rubber. It is levo-rotatory.

OIL OF SPIKE.

This essential oil is obtained by distilling the spike lavender (*Lavandula spica*), a species with larger leaves than that of our common garden lavender. This plant grows very abundantly in France (*Midi*). But Violette states that it is rarely found pure. All that comes to market has a strong smell of spirits of turpentine, along with which may be detected a slight trace of the balsamic vapour from which it takes its name. Moreover, he states very appropriately that as far as the varnish-maker is concerned, oil of spike presents no advantage over spirits of turpentine, and that the practical man will experience no difficulty in replacing the former by the latter—a wise proceeding.

LAVENDER OIL.

This oil is extracted from (*Lavandula vera*). It is yellow, smells of lavender, and has a bitter taste. The specific gravity of the commercial oil is 0·898 at 20° C., but only 0·877 when newly rectified. This oil resinifies very easily, and in this condition has enough consistency to prevent porcelain enamel colours mixed with it from running under the brush. Moreover, when spread over the surface of an object, it prevents too rapid drying.

Uses.—Mordants, *i.e.*, fixative for gold leaf, as a vehicle for porcelain enamels, and facilitate solution of copal.

CAJEPUT OIL.

Source.—Cajeput oil is obtained in the East Indies by distilling with water the leaves of the *Malaleuca leucodendron.*

Colour.—It is of a greenish colour, due to the presence of a small quantity of resinous matter. Sometimes this colour is imparted artificially through the medium of a copper salt to oils which have become bleached by natural oxidation and thus lost their greenish cast.

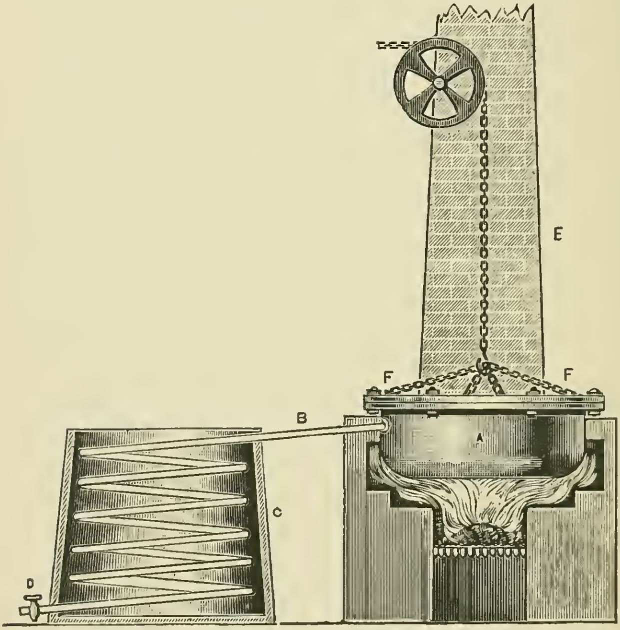

FIG 3.

Taste.—It has a burning taste, with a strong smell of camphor, turpentine and savin.

Composition and Properties.—It may be separated by fractional distillation into two portions—two-thirds of the liquid having a density of 0·897 passes over between 175°

and 178° C., and the remaining third having a density of 0·920 between 178° and 250° C.

Uses.—Its principal use in varnish-making is said to be as a solvent for Angola copal. It is also used to preserve entomological specimens and in medicine.

CAOUTCHOUCINE.

This solvent is a product of the dry distillation of india-rubber in specially constructed plant as shown in Fig. 3. The still is made of iron and built into brickwork with a furnace underneath. A connecting tube B leads to the worm in the condenser C. The still has a closely fitting iron cover which may be raised or lowered at will by the overhanging chain and pulley. A hole in the cover gives passage to a thermometer by which the temperature is regulated. The process is started by raising the still head and introducing a sufficiency of rubber cut into small pieces either by itself or along with half of its weight of crude distillate from a previous charge. The still head is then lowered and secured with nuts and bolts as shown and the fire lighted. As the thermometer approaches 600° F. a dark liquid comes over which condenses in the worm and runs off the stop cock D into a receiver. When the thermometer rises above 600° F. the fire is withdrawn. The crude distillate may be separated by fractionation into three liquids of different boiling-points and specific gravities thus:—

0·67 boiling at 90° F.
0·69 boiling at 104° F.
0·92 boiling at 599° F.

Caoutchoucine is not only an excellent solvent for rubber but also for gum resins, even copal being said to be easily soluble therein.

BORNEO CAMPHOR (BORNEOL $C_{10}H_{18}O_2$).

Origin.—This camphor, first described by M. Pelouze, is obtained from the *Dryballonops aromatica*, a tree which grows on the islands of Sumatra and Borneo. The tree is cut up into cross pieces, which are then split up with axes so as to expose the camphor which is found in crystals between the woody fibres. As much as 12 to 22 lbs. are extracted from a single tree.

Borneol is also met with in small quantity in essence of valerian, where it is formed, according to Gerhardt, by the hydration of bornene previously present therein.

Preparation.—By heating ordinary camphor from eight to ten hours with an alcoholic solution of potash from 180° to 200° C., Berthelot resolved laurel camphor into camphate of potash and borneol.

$$\underset{\text{Camphor.}}{2C_{10}H_{16}O} + \underset{\substack{\text{Caustic}\\\text{Potash.}}}{2KHO} = \underset{\substack{\text{Potassic}\\\text{Camphate.}}}{C_{10}H_{15}KO_2} + \underset{\text{Borneol.}}{C_{10}H_{18}O_2}.$$

The borneol thus obtained exhibits all the properties of natural borneol. Its rotatory power is however greater. Borneol is also formed in small quantity when amber is distilled with a dilute solution of caustic potash. It differs from natural borneol by its more feeble rotatory power.

Properties.—Borneol is found as small, colourless, transparent, very friable crystals; its smell recalls both that of ordinary camphor and peppermint. Its taste is ardent. It melts at 198° and boils at 212° C. (about 220° C. according to Berthelot). It deviates the plane of polarisation to the right. It is insoluble in water, but is very soluble in alcohol and ether.

Reaction:—Heated with nitric acid, it is converted into ordinary camphor.

$$C_{10}H_{18}O_2 + O_2 = C_{10}H_{16}O + H_2O_2.$$

When borneol is distilled with anhydrous phosphoric acid, a hydrocarbon passes over isomeric with ordinary pinene, *viz.*, bornene $C_{10}H_{16}$.

When heated with concentrated hydrochloric acid, borneol is converted into a solid monohydrochloride isomeric with pinene monohydrochloride.

Berthelot has described combinations of borneol with stearic and butyric acids.

All these combinations tend to show that borneol is an alcohol or pseudo-monotomic alcohol.

There exists in the oil of garance a residue obtained by the distillation of garance alcohol, a borneol, identical in its chemical properties with borneo camphor, but which deviates the plane of polarisation to the left.

The essential oils of cajeput and coriander contain an oxidised principle isomeric with borneo camphor.

LAUREL CAMPHOR $C_{10}H_{16}$.

Origin.—To the concrete volatile oil which exists abundantly in almost all parts of plants of the laurel tribe, but especially in the camphor laurel (*Laurus camphora*), the Arabs, who introduced it into Europe in the fifth century, gave the name of Kaphur and Kamphur—hence the English term camphor. The camphor laurel grows principally in Japan. Extensive groves or forests of this tree are owned by the Government, principally in the provinces of Shi Kokui and Kuishui, overlooking the south-eastern coast. It is also cultivated in Kisin.

The camphor laurel is a handsome (ever) green tree with small elliptical vivid green leaves, and berries like black currants. The trunk usually grows to the height of 20 feet, and then spreads out into branches. The trees live to a great age, over 100 years; trees of this age being those that are selected to be felled for the extraction of camphor, as at that age they are richest in that product. Some trees are said to attain a diameter of 20 feet.

Extraction.—The extraction process is crude in the extreme, being carried out by peasants, who are said to only make a precarious living at the work.

The trunk, branches, roots, etc., of the felled trees are broken into chips. The chips are run into a wooden tub, which stands on and fits closely to the top of an iron pan filled with water, which is kept boiling by a fire underneath. The bottom of the tub is perforated so that the steam may pass through the chips. A steam-tight cover is fitted to the top of the tub. The steam rising from the water in the pan passes through the tub, and in its passage extracts the camphor and the camphor oil from the chips, and carries them in the state of vapour through a bamboo pipe fixed to the tight-fitting lid of the tub to a condenser consisting of a wooden trough surrounded by another containing cold water to facilitate condensation. This trough is divided by vertical partitions into compartments communicating with each other at alternate ends, so that the vapours travel successively through each compartment in the trough from one end to the other.

All the camphor and camphor oil are condensed in these compartments, together with a portion of the steam. The uncondensed steam escapes into the air through a pipe fitted to the condenser.

A continual stream of water flows from a wooden pipe into a wooden trough placed over the condenser. From this upper trough the water flows into the third or lowermost one, thus keeping the condenser cool during the whole course of the distillation, which lasts about twenty hours.

Fresh water is then run in through the top of the tub into the pan for the next distillation, and being thus heated in its passage through the hot chips, time and fuel are saved in the next heating of the pan. The tub containing the chips is then emptied. The latter, after drying, are used as fuel. The tub is then recharged, well closed, and the distillation proceeded with as before.

As the distillation proceeds a semi-solid distillate of camphor and camphor oil collects in the compartments of the trough, floating on the condensed water. This is allowed to accumulate until several charges have been distilled, being usually removed at intervals of from five to ten days.

The relative proportions of solid camphor to liquid camphor oil varies with the temperature of the surrounding atmosphere. In summer only 2 per cent. per diem of solid camphor is obtained from the wood, whilst 3 per cent. is obtained in winter. In summer 18·04 litres of liquid oil are obtained from the semi-solid distillate produced during a ten days' distillation, whilst only 5 to 7 litres are obtained in winter.

Formerly the crude oil containing a large per cent. of camphor was considered useless, but it is now redistilled from iron stills connected with a brass worm condenser.

The distillate is collected in suitable vessels, cooled and filtered or pressed to separate the solid camphor, and the filtrate still containing camphor is mixed with a fresh quantity of oil and again distilled and the distillate cooled and pressed as before.

Working in this way 20 to 25 per cent. of solid camphor is obtained from the quantity of crude oil distilled, the latter losing half of its bulk during the process.

Properties.—Camphor may be easily distinguished from other substances by its whiteness and ice-like semi-transparency, by its warm aromatic taste and its sharp penetrating rosemary-like odour which persistently adheres to the hands and spreads widely through the surrounding atmosphere. It has the density of 1 at 0° and ·992 at 10°. Its vapour tension at the ordinary temperature is sufficient to cause it to sublime in hexagonal plates on the sides of the stoppered bottles in which it is kept. It melts at 175° C., and boils at 204° C. without decomposition. It has in fact so great a tendency to volatilise that it gradually evaporates into the air when exposed and disappears altogether, leaving not a single trace of its existence. It is very combustible, and suddenly bursts into flame when brought in contact with a lighted taper, and burns with a white, brilliant, very smoky flame, acrid and strong smelling. It even burns when placed on water on which it floats.

Gyratory Motion.—Water only dissolves $\frac{1}{1000}$ part of its weight, nevertheless it acquires its taste and smell. A fragment of camphor thrown upon water assumes a gyratory motion, which stops as soon as a drop of oil is thrown on the surface of the liquid. A cylinder of camphor of four to five millimetres in diameter, one part of which dips into the water whilst the other part remains in the air, communicates a to and fro movement to the water, and after the lapse of a few days is cut a little above the line of flotation. This remarkable occurrence is due to the simultaneous evaporation of the camphor and the water, which is most active at the surface of the liquid.

Solvents.—Alcohol is the best solvent for camphor; 100 parts of this vehicle dissolve 120 parts of camphor in the cold. It is precipitated from its alcoholic solution in the form of finely divided flakes. This is the best method of obtaining it in a fine state of division, because owing to its elastic nature it is very difficult to grind in the ordinary way with a pestle and mortar, at least without having been sprinkled with alcohol. Dissolved in weak spirits of wine it forms camphorated brandy, which is so often employed as an embrocation to disperse coagulated blood due to cuts or blows.

It dissolves freely in ether, fatty oils, acetic acid, etc. When it is desired to suspend it in water in a divided state it is triturated with egg yoke. Its solution in nitric acid was formerly erroneously known as oil of camphor.

Camphoric Acid.—If camphor be distilled with eight times its weight of this acid it is converted by the oxygen of the latter into a white, slightly bitter acid, which crystallises in feather-shaped crystals, freely soluble in water. This acid, discovered in 1785 by Kosegarten, is known as camphoric acid. Treated with sulphurous acid gas, camphor yields the liquid disinfectant known as *thiocamf.* During the process the camphor liquefies and absorbs a large quantity of sulphurous acid. By spontaneous evaporation this solution gives off the combined vapour of sulphurous acid and camphor.

Adulterations.—Pinene monohydrochloride, detected by lower boiling-point and evolution of hydrochloric acid fumes.

Uses.—Camphor is used as an ingredient of fireworks, not only on account of its great inflammability but also because of the whiteness of its flame.

Its smell is supposed to be fatal to small animals, especially insects. This explains the use of camphor in cases containing natural history specimens. It prevents moths and other insects from laying their eggs thereon. For the same reason small satchels containing camphor are placed in drawers in which hats and woollen articles of dress are stowed away. Camphor is added to varnishes to increase their lustre, elasticity, and to facilitate the solution of refractory resins in the manufacture of spirit varnishes.

CAMPHOR OIL.

The crude oil after repeated distillation in the manner indicated under camphor yields refined camphor oil. The latter is a colourless liquid which like solid camphor burns with a bright smoky flame. It has a specific gravity of 8·95, that of the crude oil being ·959. It is oxidised into ordinary camphor by the action of air, oxygen, or nitric acid. It is a good solvent for various resins, such as mastic, elemi, gamboge, dragon's blood, rosin, asphaltum, sulphur.

When 8 parts of mastic or such-like resin is dissolved in 10 parts of camphor oil and the solution thinned with 3·3 parts of spirits of turpentine, a transparent paper varnish is obtained which dries with a bright gloss.

An elastic waterproof varnish is obtained by dissolving linoleate of magnesia or other insoluble linseed oil soap in camphor oil with the addition of a solution of a resin dissolved in the same solvent.

But these varnishes soon lose their lustre, which is in large measure due to the camphor dissolved in the camphor oil. Consequently, however brilliant they may be at first, they rapidly become dull as the camphor, etc., evaporates.

The chief application of camphor and camphor oil is in the manufacture of celluloid and as insecticides. The refined oil might find a useful application in the perfumery of toilet soaps.

Amber and Copal Oils.

The deep brown oils produced along with succinic acid by the dry distillation of amber or copal have a density between 0·8 and 0·9 at 15° C. They are miscible with all the usual varnish solvents employed in the manufacture of oil varnishes, and may be very advantageously used to dissolve the softer copals without previous fusion. All that is necessary is to heat the finely-ground copal with the oil with constant agitation. But the harder copals do not yield a clear solution when treated in this manner unless they have been previously fused.

Moreover, these oils have a very disagreeable smell, which still further restricts their use in this direction.

Amber oil may, however, according to Jolles, be bleached as follows:—

The 5 to 6 per cent. of amber oil yielded by the dry distillation of amber is freed from water, and the 2 per cent. of succinic acid which accompanies it by deposition. The oil floats to the top whilst the succinic acid forms an intermediate layer between the oil and the water. The oil is then syphoned off. It is a strong smelling fluorescent viscous liquid. Its ultimate analysis proves it to be a compound of carbon, hydrogen, oxygen and sulphur, but it is free from nitrogen. When heated with lead salts it yields a precipitate of black-lead sulphide.

Neither dilute nor concentrated hydrochloric acids act to any extent on amber oil, no more do dilute sulphuric or nitric acids even on heating. Fuming nitric acid decomposes amber oil with great energy, with evolution of nitrous fumes and formation of much succinic acid, with the simultaneous formation of a reddish yellow balsamic resin, of a musk-like odour, often sold as artificial musk.

Alkalies appear to have no action on either the colour or the smell of the oil. Reducing agents, such as sulphur, zinc chloride, sodium thiosulphate, which have generally a marked action on vegetable oils, do not in any way affect amber oil, neither do oxidising agents such as chlorine have any action either upon the smell or the colour of the oil; and although both permanganate and bichromate mixtures are reduced, the colour of the oil is not affected.

Better results are obtained by rectifying the oil. When amber oil is distilled, water comes over first, then a yellow, followed by a light green oil, and finally a dark green viscous oil. As soon as the water has distilled over, the thermometer rises very quickly to 150° C., and keeps on rising until the distillation finishes at 360° C., only remaining stationary for brief periods at 230° and 255° C. The pitchy residue amounts to about 10 to 15 per cent.

Practically identical products are obtained and in the same order by distillation *in vacuo*. The following results were obtained by fractional distillation :—

Temperature.	Description of Oil.	Percentage of Total Distillate.
130-180° C.	Light Yellow	33
180-300° C.	Light Green	15
300-360° C.	Dark Green	15

The yellow oil still retains its offensive odour, but this is removed when the crude amber oil is distilled in a current of steam. When steam is passed through amber oil undergoing distillation with half its volume of water, the steam carries over and dissipates the offensive vapour. The yield of rectified inodorous yellow oil is 38 per cent. of the crude. Distillation from a 10 per cent. alkaline solution has no effect upon the yellow oil distillate, but removes the offensive smell from the residue in the still, which is separated from the emulsion formed in the process by salting out, after which its odour is pleasant.

The yellow oil produced by distillation in a current of steam, unlike the crude oil, may be bleached by reducing agents, such as sulphurous acid, or by oxidising agents, like permanganate or bichromate in sulphuric acid solution. Eight parts of permanganate bleach 100 parts of yellow oil. The hot permanganate solution is added a little at a time, and with constant stirring to the oil previously mixed with dilute sulphuric acid. The whole is then allowed to cool, and the oil which floats to the top is filtered, after which it is mixed with a little salt or plaster of Paris, allowed to stand in contact therewith for twenty-four hours, and again filtered. From 7 to 9 per cent. of the distilled oil is lost in the bleaching process.

RESIN SPIRIT.

The crude spirit (p. 121) is treated with direct steam and 3 to 3½ per cent. soda lye of 36° to 40° B. at 80° to 100° C. After removing the lye, washing with tepid water, mixing with 2 per cent. of sulphuric acid of 66° B. at 15° to 25° C., and allowing the acid to settle out (in 3 to 4 hours), 2 per cent. soda is added to neutralise, and the spirit is finally rectified like spirits of turpentine *in vacuo* by direct and indirect steam, over ½ per cent. of caustic soda and 3 to 5 per cent. of lime water. The spirit approaches true spirits of turpentine more closely by admixture, either before or during rectification, with either the spirit obtained by distilling the stumps and roots of trees, wood tar oil or turpentine resin, and a better sale is obtained for such a product.

SECTION IV.

RAW MATERIALS (*continued*). CLEANSING, GARBLING AND CRUSHING OF RESINS.

CLEANSING AND GARBLING OF RESINS.

THE cleansing and preparation of resins—copals in particular—is a point of great importance, looking to the quality of the varnish, into the composition of which the resins will subsequently enter.

This preparation generally includes (1) a sorting out of the lumps or pieces according to their size; (2) washing; (3) a sorting out according to quality; and (4) crushing.

Copals: 1st Garbling.—It is customary to separate these into five kinds, using sieves of previously determined dimensions. The meshes of the sieves in general are one square centimetre in section, then four meshes per square centimetre, and finally twenty-four meshes per square centimetre. They are thus obtained:—

1. The large pieces sorted out by hand.
2. The pieces having more than 10 millimetres in diameter.
3. The pieces having more than 4 millimetres in diameter.
4. The pieces having more than 0·4 millimetres in diameter.
5. Dust.

Washing.—Generally only the sorts 1, 2 and 3 are washed, because too great a loss would occur if it were attempted to wash the kind No. 4.

Each batch washed consists of pieces of gum resin of the same size, because different sized pieces are unequally attacked by the cleansing agent. Finally, and more generally, only the hard copals are washed, particularly Angola, Benguela, Sierra Leone and Congo copals, and sometimes Manilla.

The wash water consists of a weak caustic or carbonated alkaline lye, which partially attacks the crust with which the pieces are coated, and enables it to be removed by the simple attrition or rubbing of the pieces against each other. The process is conducted in a wooden vat half-filled with water, and to which has been added for every 100 parts of water 5 parts of a solution of either caustic or carbonated soda lye of 5 per cent. strength. The resins are allowed to stand in contact with this lye for an hour, after which they are passed through a washer fitted with brushes which revolve in the same solution and free the lumps of the crust encoating them.

It is plain that the duration of the process will depend on the size of the lumps and the kind of copal treated. There is no advantage in prolonging the action of the alkaline solution so as to remove all the persistent specks. It is better to remove them by hand, either with a brush or with a sharp-pointed knife.

The process is finished by several washings in pure water and the pieces laid out to drain and dry upon cloths, taking care that they do not touch each other. They are afterwards completely dried either in the air or in a warm room.

2nd Garbling.—After washing, the pieces are sorted out according to quality. The larger pieces are broken up into medium sized ones with a hammer, and they are then sorted out according to their colour. The smaller kinds are sorted according to their outside colour. Besides colour, purity must be taken into account, and those pieces which contain extraneous matter, such as vegetable *débris*, should be put upon one side. The latter are very apt to char when the copal is being "run," and thus by darkening the colour these vegetable remains diminish the value of the varnish.

Hard copals are sorted out in this way into two or three qualities, and four, five, and even more qualities in the case of soft copals.

CRUSHING.

The lumps of which each lot is composed are now crushed or broken up, and this should be done methodically, because the size of the fragments is of great importance when the copal comes to be heated. The size varies with the hardness, and it is customary to break up the pieces into the following sizes, *viz.* :—

½ c.c. in bulk for East African (Zanzibar) copal.
¾ c.c. in bulk for West African (Congo) copal.
1½ c.c. in bulk for soft copals (Manilla).

The crushing or grinding should be done in such a manner as to produce as little dust as possible, because the dust darkens when heated. Even when but slightly heated, the dust agglutinates together and afterwards requires a higher temperature to dissolve it. Now, when it is heated more strongly, the particles of dust in contact with the sides of the vessel carbonise and colour the whole mass.

For this reason breaking up should not be done with the hammer, because it pulverises, nor with an ordinary leverage cracker, which produces powder and numerous splinters.

The most simple method consists in using a cracker analogous to that used for breaking loaf-sugar into lumps—one arm is fixed to the table and the other, provided with a long handle, is free ; great leverage is thus obtained, and the lumps have not far to fall on to the table.

Machines have been made which will break 2½ to 3 tons a day, yielding a percentage of dust varying from 8 to 10 per cent. according to the kind of copal. They also grade the copal into different sizes. If the copals are not to be fused but simply treated by a solvent, they are not crushed but pulverised straight away. The powder produced in crushing is also reserved for treatment in the cold by solvents.

From the appearance of the large pieces, their colour and their origin, the manufacturer knows fairly well what he may expect from any given lot of copal in regard to its behaviour with different solvents. But in the case of small pieces—often the case with soft copals—they cannot be broken up for examination as to colour, purity, etc. In that case a fair average sample is taken and melted at as low a temperature as possible, and upon the mass thus obtained the necessary trials are made so as to ascertain how the bulk will behave.

Dammar is not washed; it is freed from dust and smaller fragments by sifting, and the lumps graded into two sorts according to whether light or darker coloured. They are then pulverised, or crushed into pieces 1½ cubic centimetres in bulk, according to the treatment to which they are to be subjected.

Mastic and Sandarach.—These are simply passed through the riddle to separate the tears according to size. They are graded according to purity, which is easily ascertained by placing them on a sheet of white paper.

Shellac.—It is employed as it is delivered. The pieces which have stuck together are detached so as to facilitate solution or fusion, which is still further facilitated by coarsely crushing the shellac.

But in order to obtain bright alcoholic solutions, it is necessary to treat the shellac chemically, so as to remove the 6 per cent. of wax which it contains, because the latter is insoluble in alcohol. Andés has indicated two processes:—

1. The first process due to Gräges is laborious and costly. Good shellac is dissolved in 3 or 4 parts of 92° alcohol by heating on the water bath; distilled water is added, generally 1 part of water for 3 of alcohol. The wax separates as a cheesy, which is separated by filtration through cloth. This wax may be again stirred with alcohol of 67 per cent. strength, which is added to the first alcoholic

liquor. The liquid is finally filtered through paper, and the alcohol separated by distillation. A wax-free resin is thus obtained, which yields clear solutions with alcohol.

2. A more simple process consists in treating 15 parts of shellac with a solution of 2 per cent. of carbonate of soda in 100 parts of water—the shellac dissolves whilst the wax remains unaffected. The liquid is filtered through cloth, and the shellac precipitated by dilute sulphuric acid. The resin is carefully washed to remove all trace of acid, and a gum shellac is thus obtained which is rolled into sticks or tresses, and from which clear alcoholic solutions are obtained.

SECTION V.

THE EFFECT OF HEAT UPON RESINS.—DIFFERENT FORMS OF PLANT FOR FUSING RESINS BY NAKED FIRE, SUPERHEATED STEAM AND SUPERHEATED AIR.

The Influence of Heat upon the Solubility of Hard Resins.

The harder the resin used the harder and brighter is the resultant varnish, but under ordinary manufacturing conditions the hard resins, amber and copal, are insoluble in the usual solvents, such as alcohol, spirits of turpentine and drying oils.

The study of processes whereby hard resins may be dissolved without losing any of their properties, colour, etc., is of the utmost importance in varnish-making.

Soehnée Brothers have succeeded in completely dissolving these resins in 90 per cent. alcohol by grinding them under water and then leaving them in a state of impalpable powder for sometimes more than a year. But this process, however applicable for the manufacture of special varnishes, would scarcely meet the wants of a factory working continuously, even taking into account the improvements which have been effected upon it by practical men.

It has been attempted to facilitate the solution of copal in alcohol by the addition of certain softer resins, but the process does not appear to have yielded the wished for results; besides, the presence of these soft resins would materially alter for the worse the quality of the resultant varnish.

It has also been proposed to treat copal with a mixture of 1 part of bisulphide of carbon, 1 part of spirits of turpentine, 1 part of benzol.

The resin, 1 part, is left in contact with the solvent, ½ part, for several days in a closed vessel, and the liquid decanted. Complete solution is however rarely obtained.

The best results are obtained by previously heating the resin. Violette was the first to investigate the matter. He obtained resins which dissolved completely in the usual solvents. The explanation of this transformation was given by M. Riban, who stated that a body is more soluble the less is its degree of polymerisation. The study of the bodies yielded by the dry distillation of resinous substances led him to apply the preceding theory to the transformation of insoluble resinous substances into soluble ones.

On the other hand, the changes effected in copal under the influence of heat were systematically studied by Schwarz, of Gratz, and his experiments, conducted with much care, are very interesting.

Schwarz took a very fine specimen of raw copal, colourless, transparent and limpid, and dried for a long time over sulphuric acid, and placed it in ether, where it swelled. By a series of experiments he found that 66 per cent. of the raw copal swelled and gelatinised without dissolving in ether. This copal, which he called *swollen copal*, and which on drying became converted into a horny mass, had always the same composition, swelling in solvents without dissolving. He then heated this swollen copal to fusion, and found that it now dissolved in the usual solvents. He called it *soluble copal*.

On the other hand, raw copal was heated in the same way. He thus obtained a further product, *viz.*, *pyrocopal*, which dissolves in solvents, but the addition of alcohol to a solution, say, in chloroform, precipitates a gelatinous product which he called swollen pyrocopal, whilst the product remaining in solution, in spite of the addition of alcohol, he termed soluble pyrocopal.

Now, the analysis of these different substances yielded the following results :—

	Raw Copal.		Swollen Copal.		Soluble Copal.	
	Found.	Calculated for $C_{19}H_{30}O_2$.	Found.	Calculated for $C_{48}H_{76}O_4$.	Found.	Calculated for $C_{36}H_{56}O_4$.
Carbon - -	78·72	78·62	79·95	80·44	78·00	78·26
Hydrogen -	10·24	10·34	10·87	10·75	10·30	10·40
Oxygen - -	11·04	11·04	9·18	8·81	11·70	11·16

	Pyrocopal.		Swollen Pyrocopal.		Soluble Pyrocopal.	
	Found.	Calculated for $C_{19}H_{28}O_2$.	Found.	Calculated for $C_{48}H_{94}O_3$.	Found.	Calculated for $C_{36}H_{54}O_3$.
Carbon - -	83·63	83·82	83·01	83·23	81·02	80·89
Hydrogen -	10·36	10·29	10·52	10·69	10·37	10·01
Oxygen - -	6·01	5·83	6·47	6·08	8·61	9·10

Without attaching too much importance to the formulæ, it will be observed that the percentage of carbon is increased in every case by the action of heat. The change in composition arises at first from a loss in the elements of water, and finally, at a higher temperature, from a disengagement of hydrogen, carbonic oxide, carbonic acid, and at last the volatilisation either of essential oils or the products of their decomposition. It is to the product which has been subjected to these changes that Riban's theory applies.

If the heat applied be limited to the simple fusion of the hard resins, they do not dissolve in spirits of turpentine, neither in the hot nor in the cold. If the temperature be increased the quantity of soluble resin increases, but in order to effect complete solution the heat applied must be of such intensity and duration as to cause a partial decomposition of the resin, by which it loses about 25 per cent. of its weight.

The greater the loss by distillation the more easily are the hard resins dissolved, but, on the other hand, the darker in colour is the resultant varnish, and the more unprofitable is the process from the manufacturing point of view.

Moreover, it must not be forgotten that the resins alter more and more in proportion as the distilled product increases; they become soft and pitchy, and do not yield solid, durable varnishes, but varnishes which are dry, soft and tacky.

The best process consists in working at as low a temperature as possible, knowing that hard copal melts at 315° to 340° C., and distils about 360° C. Amber melts at 287° C. and distils at 320° C.; medium hard copal melts at 180° C. and distils at 230° C.

According to Leppert, it is necessary to cause the following resins to lose by heat the under-mentioned weights before they become soluble :—

	Per cent.
Manilla, hard dust - - -	11·7 to 13·3
Do. lumps - - - -	14·6 ,, 15·8
Do. - - - - -	12·3 ,, 16·5
Angola, red, crude - - -	21·6 ,, 16·6
Do. washed - - -	16·6
Sierra Leone - - - -	17·2
Zanzibar - - - - -	32·0
Amber - - - - - -	30·0

In conducting his experiments Violette employed a small glass retort to melt the copal, and heated it in a bath of molten tin at about 360° C., collecting the distilled products. His results are given in the following table :—

Loss in weight by distillation. Per cent.	Quantity of Oil resulting from the distillation.	Solubility of the heated Copal in Spirits of Turpentine.
3·0	3·0	Insoluble.
9·0	8·5	,,
10·5	10·2	,,
16·0	15·7	,,
20·0	19·0	Slightly soluble.
22·0	21·3	More easily soluble.
25·0	24·5	Very easily soluble.
28·0	27·1	,,
30·0	29·0	,,
32·0	31·0	,,

The experiments to determine the solubility of the product were made by keeping the resin and the solvent at 100° C.

If under ordinary conditions it is necessary to cause copal to lose 25 per cent. of its weight, Violette has, however, made the following interesting observation, which perhaps has not in actual practice received the attention it deserves. He has shown that copal, which has only lost about 10 per cent. of its weight, and even less still, and which, as we have seen, is insoluble in ordinary spirits of turpentine, dissolves, on the other hand, very easily and completely in spirits of turpentine which has been simply thickened by long exposure to light and air. It would appear to be easy by means of apparatus identical with those employed for the oxidation of drying oils to obtain such a product at a small cost, and the manufacture of copal varnishes would thus obtain lighter coloured products, and at a less cost in consequence of the smaller loss by distillation.

Fusion or Running Copal, etc., on the larger scale.—Hard resins are decomposed by heating them either in open vessels or in plant provided with a condenser for the collection of the volatile products. The latter arrangement has the great drawback that the varnish-maker cannot follow the operation except by observing the quantity of condensed liquid. Now, the melting may be incomplete and the distilled liquid may come principally from the resin in contact with the most heated parts. Working with open vessels, on the other hand, the quantity is in general smaller, and thus no difficulty is experienced in ascertaining when the whole is melted, but in the latter process this point can only be accurately ascertained by weighing the receiver, which is but rarely done; the varnish-maker trusting more often to his experience.

In any case, preliminary laboratory experiments ought to be previously made upon the well-graded lots of resin which are to be converted into varnish. In the case of every new kind of resin a fair average sample should be taken of the bulk and the amount of loss which it undergoes before a completely soluble product obtained is noted.

FUSION IN OPEN VESSELS OVER A NAKED FIRE.

Bartky's Method.

According to Bartky, a cylindrical pot or pan may be used, having a diameter almost equal to its height, made of cast-iron, carefully enamelled and furnished with a tight-fitting lid. This pan is placed on a fire, and is kept in position by a circular flange cast on to it about one-third of its height starting from the bottom.

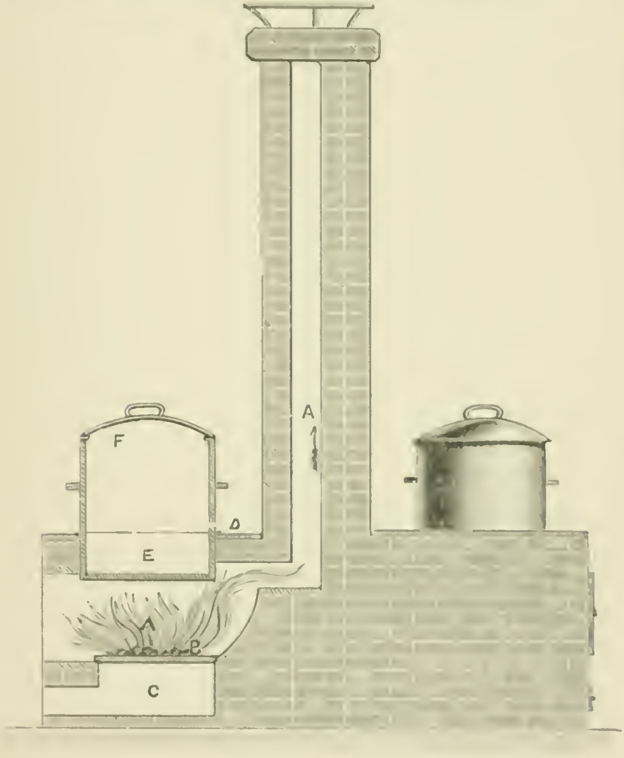

FIG. 4.

Generally about five pounds of copal, broken into pieces about the size of peas and freed from dust, are fused at a time. The pieces easily distil, whereas the fine copal is more refractory and yields dark varnishes. If the quantity of dust be considerable it is better to heat it separately.

During the first five minutes the fire is carefully regulated at a moderate temperature, during the next ten it is more brisk, and it is afterwards maintained as much as possible at a temperature such that the melted copal does not froth up before half an hour has elapsed. The fire is now allowed to die down slightly, so that all the pieces of copal may be well melted; if this be not done certain pieces simply softened will be carried up by the froth of the melted pieces. As occasion may require the crust of the softened copal is broken by an iron rod, avoiding, however, any agitation of the mass beneath.

When the whole mass is melted the heat is increased in such a manner that the copal rises up two-thirds or three-fourths the height of the pan, then by lowering the fire the copal is allowed to fall down again to the bottom of the pot. Afterwards a stronger heat still is applied, avoiding, however, any overheat, but still sufficient that after two or three minutes the copal oil distilled begins to condense on the lid, which has been carefully adjusted on the pan, which is changed several times to prevent the copal oil from falling back into the pan. Finally, when abundant vapours begin to come off the lid is taken away and a hood made to descend and surround the neck of the pan so as to carry the vapours outside, so that they neither spread through the workshop nor fall back into the pan.

The pan is withdrawn from the fire when it is thought that a sufficient quantity has been distilled, and the whole is run into an iron basin which is covered, and the whole allowed to cool.

According to Schwarz, who worked upon Zanzibar copal of first-class quality, the most suitable process consists in melting about 12 lbs. at a time in a cylindrical copper vessel shaped like a large bottle with a flat bottom and capable of being closed by means of a tight-fitting lid. The mouth is fitted with a circular flange in the form of a gutter with a spout for running off the melted copal.

This vessel, being charged and fitted with its lid, is placed on a simple brick furnace heated with wood charcoal, which has been carefully lighted throughout so that fusion may soon start with disengagement of abundant vapours. At this moment a movable flue is placed over the neck of the vessel, which conducts the vapours to the outside. After fifteen to twenty minutes, and two or three agitations with an iron rod, the melted mass becomes clear and free from bubbles. It is then withdrawn from the fire, the cover is removed, and the vessel and contents allowed to cool a little, often at a gentle heat. The mass is then run into an iron basin; at any rate, as will be seen afterwards, it is not used immediately for varnish-making.

Furnaces.—The gum melting pots are placed upon either fixed or movable furnaces.

The latter consist simply of a grate placed inside a sheet-iron covering supported on a tripod, with a pipe to carry away the smoke. They have the advantage of being placed anywhere, but they have the inconvenience of allowing great part of the heat to pass away by the flue, and thus cause an extravagant use of fuel. They are used, however, in small factories where the work is done in the open air. Coke or wood charcoal is the fuel generally used.

On the large scale, with continuous working, the furnaces are fixed and made of brick, and are arranged in a special building. Such furnaces preserve the same heat for a long time, and either coal or coke can be used as fuel.

They, however, are inconvenient for the fusion of certain copals which froth much, especially Sierra Leone, which is profitable to work in spite of this deficiency on account of its relatively low price and good quality.

In such cases the pan is placed in a basculed truck, by which it can be easily removed from the fire if the gum threatens to prime over.

Gum Pots.—The form given to these is very variable; when they bulge out they can be placed directly over the opening in the furnace, but it is preferable to furnish them with an iron ring, which enables them to rest solidly on the plate of the furnace, and which, measuring at least two inches in width, has the further advantage of cutting off all communication with the fire, and consequently diminishing the risk of fire.

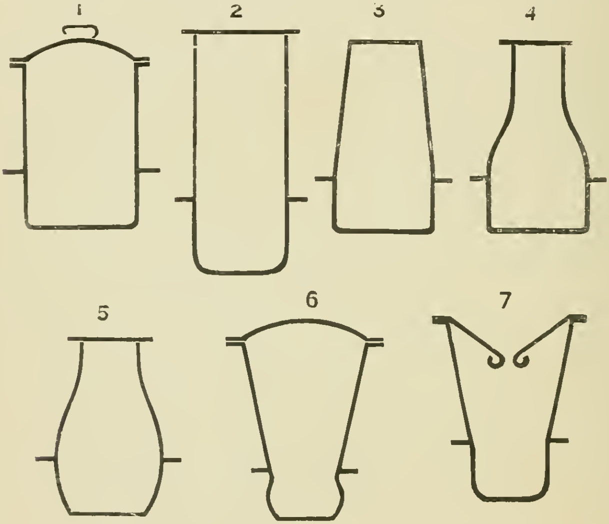

Fig. 5.

Cylindrical pots are met with a diameter equal to about $\frac{10}{13}$ of the height, made of cast-iron, carefully enamelled and furnished with tight-fitting lids. But the weight is too great, so these pots are too heavy to handle.

They are therefore made of iron plate or copper. As the heat-conducting power of these materials is very great, the extent of the part of the pot which is directly heated is restricted, whilst at the same time the height is increased so as to prevent the copal suddenly rising and priming over.

Copper can only be used for varnishes the colour of which will not be affected by verdigris, which forms and colours the varnish, but copper has the advantage of being capable of being used in thin sheets, which impart lightness; further, they last a long time, and as copper is a good conductor of heat resins melt in such vessels very rapidly.

For the manufacture of pale bright varnishes, pots made of wrought iron and covered with a hard and resistant white enamel should be used.

If the wrought iron has the disadvantage of burning rapidly, and especially over a coal fire, on the other hand, it of itself imparts no colour to the varnish.

Conical pots have also been used principally with the view of preventing, as far as possible, the interior temperature from lowering. To prevent the copal as it rises from plugging the restricted part of the pot, *bottle-shaped* pots have been designed. But this form has the disadvantage of allowing the disengaged vapours to condense too suddenly and fall back into the pot.

The *expanded* form obviates this inconvenience and prevents the copal from rising so rapidly and tumultuously, but each time the lid is opened either for the purpose of stirring the mass or to allow the vapours to escape the interior temperature is lowered too soon and too suddenly.

The same shaped pot, but provided with a lid in the form of an *inverted* cone, has lately come into vogue. The summit of the cone is sufficiently open to allow the passage of a stirring rod and the stirring of the resin. This shaped lid can—if need be—be used as a funnel in factories where varnish is made on the large and continuous scale. It may be objected that the distilled liquids condense on the surface of the lid and return to the centre of the pot, but as the lid is only on the pot during the time the distillation is not brisk, it is easy to adjust around the edge of the orifice an annular gutter which retains the products first distilled and which condense readily, thus hindering them from falling back into the pot.

We have incidentally spoken of the material which is used in the manufacture of gum melting pots.

Enamelled iron pots have the disadvantage of cracking easily and conducting heat badly; further, the enamel is rapidly attacked and is soon partially eaten away. They may, moreover, be overheated. For these reasons only small quantities of copal can be fused in vessels constructed of this material, and even these often become darkened, and the value of the varnish depreciated.

With *copper* pots, thanks to the conductivity for heat of that metal, larger quantities can be fused at a time and much more quickly without fearing any discoloration from the charring of the resin on contact with the hot sides. But, on the other hand, there is formed, as we have said, a coloration due to the formation of copper salts. There is observed a dull red cloudiness in copal fused in a copper pot, and this dulness is due to the presence of copper compounds formed by the action of the products of distillation on the neck and the lid of the pot. These green-coloured compounds fall back into the melted resin, and in consequence of a reducing action yield the ruby-red colour of copper.

Silvered pots.—In order to do away with this inconvenience, which diminishes appreciably the value of the varnish, Schwarz silvered the inside of the copper pots. He took a pot which had been in use, cleaned it well with acid, and silvered it with a mixture of nitrate of silver, cyanide of potassium, and carbonate of lime. He found that under such conditions the melted copal was not coloured, only becoming very slightly dull red. With copper lined with silver the result was still better.

Nickeled or galvanised copper might be used. Galvanised iron has been employed with very good results.

Wrought iron pots are rivetted and coated with an enamel. Copper pots are made of one piece, at least as far as the bottom is concerned, which is then solidly adjusted and rivetted to the top part; the latter is often made of sheet copper a little thinner than the lower part.

The lower part of the pan should have no sharp angles, but should be slightly rounded, rather egg-shaped, so that it can be easily cleaned, without, however, affecting its stability when it stands on the ground.

Many varnish-makers only clean their pots but very rarely, which is done with a little spirits of turpentine. By cleaning the pot after each operation there is no risk of leaving charred residues, or residues which may be easily charred, which would colour the gums afterwards fused in it. It would, therefore, appear to be advantageous to carefully clean the pots, and if it be feared that the copper thus cleansed would colour the gum, the inside may be given a coat of oil varnish, which will diminish this risk.

Depth.—The depth of the pots should not be too great, it being necessary to reach the bottom with the hand when cleaning them. For easy working the dimensions generally are 2 feet to 2 feet 6 inches in height, with a capacity of from 4 to 5 gallons, and a weight of from 22 to 26 lbs.

Size.—In fact the size of the pot is proportional to the amount of gum to be fused in one operation. Now, practice teaches that small quantities are more quickly fused, and with less fear of a dark-coloured product. With large quantities the portions in contact with the side of the vessel become fluid before the others, and quickly turn brown and then char, and thus colour the whole mass. Small quantities, on the other hand, melt quickly and almost at the same time. It is advisable not to exceed at one operation $2\frac{1}{2}$ lbs. of hard copals or ambers, and 5 lbs. of soft copals.

CONDENSING PLANT.

Andres and Tingry used a condensing plant which on account of its simplicity should be of service to manufacturers. It consists (Fig. 6) of a sheet-iron cylinder, well rivetted together, which terminates in the form of a cone, and which rests on a sheet-iron cylinder acting as a furnace. In the annular central space a second cylinder of copper of less diameter is placed, the lower part of which, likewise conical, is pierced with small holes. Armatures maintain this interior cylinder in such a position as to leave a free space of from $\frac{2}{5}$ to $\frac{3}{5}$ of an inch between the copper and the sheet

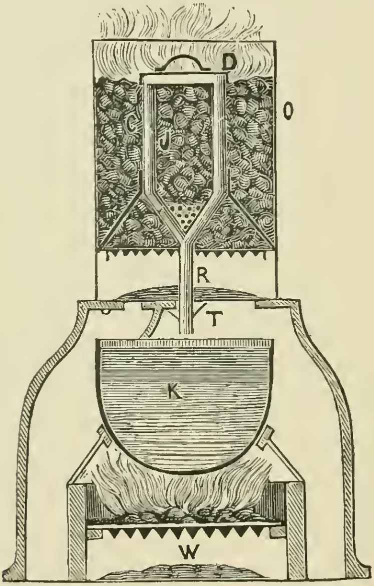

Fig. 6.

iron. The copal is placed in this copper cylinder. After having shut the mouth of the cylinder with a well-luted lid, and brought the temperature to the melting-point of copal, the latter melts and runs away in drops, whilst at the same time a portion of the products of distillation condense in the conical part. The melted copal and the condensed products are received in a suitable vessel, which in the case of oil varnish may already contain the necessary quantity of linseed oil at a suitable temperature. When one operation is finished it is not customary to clean the copper cylinder, because the slight coating of resin which remains on the metal prevents it from being attacked by succeeding fusions, but, on the other hand, the resin in contact with this coating becomes coloured, as it is easily charred.

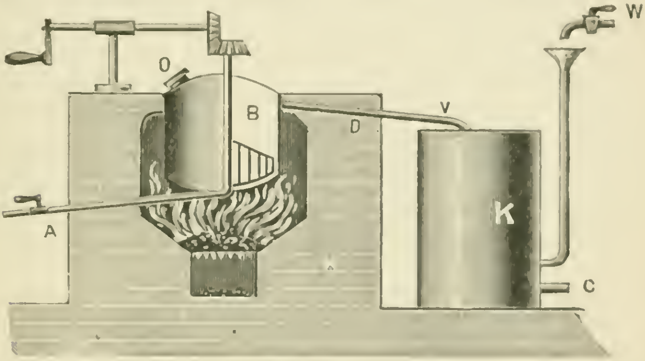

Fig. 7.—Distilling Plant and Condenser for Fusing Copal.

But by this process the distillation of copal cannot be conducted with the requisite precision, and it is preferable on the large scale to use a proper distilling plant fitted with an internal agitator, and communicating with a condenser (Fig. 7). The gum resin may be heated over the naked fire, or, what is better, by means of a bath of molten lead or a sand bath. The still is suitably connected at the bottom with a wide pipe, so as to enable it to be emptied. This pipe should be so arranged, on the one hand, as to be sufficiently warm to prevent the copal from solidifying in it, and, on the other, so that the receiver may be completely emptied after each operation.

Generally 25 lbs. are operated on at a time in a copper still, silvered inside and built in masonry up to the capitulum. When it is not thought necessary to silver the inside, it is advisable to cover it with a coat of oil varnish. The coloration of the varnish with copper compounds is thus avoided, and this coating ought to be renewed each time that the alembic is cleaned.

A disengagement tube conducts into a chimney the fumes which escape condensation, but these are but very weak if the operation be well managed, and it is precisely by the quantity of products condensed that it is ascertained how far the distillation has been pushed. The condensed liquid, which on an average has a density of ·800 for copal and of ·900 for amber, is received in a vessel graduated according to the following data for 100 lbs. of copal :—

10° of distilled products = 8 lbs. of water.
11° ,, ,, = 8·8 ,,
12° ,, ,, = 9·6 ,,
13° ,, ,, = 10·4 ,,

That is to say, that each one-hundredth part corresponds to ·8 lb. of copal, and to ·9 lb. of amber.

Treatment with Steam.

Andres gives some instructions on the use of steam for heating in closed vessels as proposed by Bartky.

The apparatus consists of a copper receiver placed inside a wrought-iron boiler, leaving an annular space of about 10 inches through which superheated steam circulates.

The cylinder and the boiler are provided with safety valves and manholes, and a mechanical agitator for stirring the resins, which are afterwards run off through a tube at the bottom. The condensed steam is run off through a tap.

Bartky has principally wrought with the mixtures used in varnish-making, and he claims to have obtained good results.

Violette, who conceived the idea of heating resins under a pressure of twenty atmospheres, found that the results varied with the resins treated.

SUPERHEATED AIR.

M. Holtzwich, of Dresden, has proposed the use of superheated air circulating in closed vessels. The gaseous products which escape are passed through a condenser, and their physical appearance, as well as their quantity, indicate the moment when the operation should be stopped. The hot air as it leaves the apparatus might find an application in the oxidation of linseed oil for use in the manufacture of oil varnishes.

SECTION VI.

SPIRIT VARNISHES: MANUFACTURE, PLANT, HOT AND COLD PROCESSES, FILTRATION, BLEACHING, STORAGE, PROPERTIES AND USES, ANALYSIS.

Spirit varnishes are made by dissolving one or more resins, etc., in one, or more than one, volatile solvent. On evaporation of the solvent or solvents, with or without the aid of heat, a coating of varnish is obtained consisting practically of the unchanged resin or mixture of resins which have not been acted upon chemically in any appreciable way. When an article is coated with an alcoholic spirit varnish, the nature of the coat will vary according to the resins or mixture of resins, etc., from which it was manufactured. Hard resins like amber, copal and shellac yield a hard, lustrous, but rather brittle, coating; on the contrary, sandarach, mastic, elemi and Venice turpentine yield a supple, elastic coating. The quality of a varnish, therefore, depends principally on the resins employed. The kind of resin, or resins, and the proportions thereof also vary with the kind of varnish it is desired to make and the purpose for which it is to be used.

More or less volatile liquids are used as solvents—such as methylated spirit, spirits of turpentine, ether, etc. The harder the resin and the more volatile the solvent the quicker does the varnish dry; but the coating does not wear so well, being easily affected by alternations of temperature. If the coat be too thin the varnish will "pit"—*i.e.*, become dotted with numerous small holes; on the other hand, if too thick, the resin, being very brittle, will shell off very easily. The quantity of solvent, or solvents, required for any particular spirit varnish will depend not only on the thickness, etc., of the coating desired, but also on the nature of the resin, or resins, and their degree of solubility in the solvent, or mixture of solvents, as well as on the greater or less rapidity with which the solvent, or the different volatile ingredients of the mixture of solvents, volatilise. Speaking generally, the consistency of a varnish should harmonise with that of the solvent. The more volatile the solvent the thinner should be the varnish, the necessary thickness being imparted by applying several thin coats.

Mixing separate Solutions of different Resins to obtain a desired Varnish.—When the manufacturer has a large trade in a great variety of varnishes of very different compositions, in each of which several resins are associated together, no good purpose is served in multiplying operations by preparing each of those varnishes singly. It is far better and more economical to make separate solutions of each of the resins and mix them, when required, in the desired proportions, according to the particular varnish it is desired to send out. In order to save calculations, and thus be in a position to make the necessary mixtures promptly, it is advisable, as far as practicable, to make all the resin solutions of the same strength—that is, the amount of resin and solvent should be the same in each case.

Thinning down concentrated Varnishes.—Some manufacturers prepare several thick varnishes, which they thin down to the requisite consistency and blend the one with the other according to the nature of the "orders" they are making up. In actual practice, about 2½ parts of solvent are used for every part of resin, *i.e.*, about 2 to 3 lbs. to the gallon; but when the varnish has afterwards to be bleached or filtered, etc., a larger proportion of solvent may be used, and the excess afterwards recovered by distillation.

The solvents in most general use are methylated spirits and spirits of turpentine, more familiarly known as "turps".

Alcoholic strength.—When alcohol is the solvent or vehicle employed, the quicker the varnish is required to dry the stronger must be the alcohol. In all cases it should contain 90 per cent. of anhydrous, or absolute, alcohol O.P.; but generally, when it is desired to thin the varnish, alcohol of 85 O.P., or even 80 per cent. O.P., strength answers very well. But it is first advisable to experiment with a small quantity of the varnish to see how far the alcohol can be reduced before starting to thin down the varnish, for if too weak alcohol be used the water which it contains will throw the resin out of solution, rendering the varnish turbid, and giving more trouble and annoyance in again clarifying it than would compensate for any profit incidental to the process. It is also necessary to note whether the varnish becomes cloudy on lowering the temperature. Moreover, too dilute alcohol imparts a tendency to "bloom," etc., to the resultant varnish.[1]

Spirits of turpentine always leaves a residue on evaporation. It partially resinifies owing to the absorption of oxygen, and this resin, or balsam, is embodied in the resultant coat, which consequently does not dry so quickly as a methylated spirit varnish. But, on the other hand, spirits of turpentine does not evaporate so quickly as methylated spirit: consequently the coating is formed more slowly, and therefore in a more uniform and free manner, thus diminishing pitting and cracking. Recently distilled spirits of turpentine does not oxidise or resinify to any great extent, but when it has been stored for some time with free excess of air it oxidises very appreciably, and the thick gluey balsam left on evaporation seriously injures the quality of the varnish. The use of spirits of turpentine as a solvent for varnishes for indoor work is restricted by its injurious action on the nervous system of the painters and decorators who inhale its vapours in too great a quantity.

Now-a-days the varnish-maker has at his disposal cheaper

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[1] See table on page 100.

solvents, such as benzol, solvent naphtha, gasoline, petroleum ether. It is cheaper in some cases, and where practicable, to make an original thick solution of resin in these solvents and then thin down with methylated spirits or spirits of turpentine. A saving of time is thus effected, as very often the resins dissolve more freely in these solvents than in either spirits of turpentine or alcohol.

It has also been found advantageous in France to replace spirits of wine by wood spirit on account of the difference in price, or by amylic alcohol (rectified fusel oil), which volatilises less rapidly.

In some cases a complex mixture of the most varied solvents is used, of which the following recommended for dissolving copal is typical:—

1 part carbon disulphide.
1 ,, spirits of turpentine.
1 ,, benzene.
1 ,, wood spirit.

Putting to one side for the moment a matter of extreme importance—*viz.*, the choice of resins—we must bear in mind that the solvent used to dissolve the resin exerts a very appreciable influence on the quality of the resultant varnish. If, for example, we use a very volatile solvent the varnish will have a tendency to pit or pinhole; if, on the contrary, we use a solvent which evaporates slowly, we may be compelled to invoke the aid of heat to cause it to evaporate more rapidly or to drive off the last traces; but then the coating may be liable to come off. Moreover, varnishes containing shellac are turbid, owing to the pressure of a waxy constituent natural to the gum, which is insoluble in alcohol. But as this rather improves the varnish than otherwise, no attempt is made to remove it.

Dissolving the Resins—Cold Process.

This operation is simple, but is not, however, always carried out in the most rational manner. It is found that different samples of the same resin do not always dissolve equally readily in the same solvent. But pieces of the same degree of hardness, colour and brilliancy all dissolve with the same rapidity and to the same extent. It is therefore advisable to sort out the pieces as received from the merchant according to colour, transparency, etc., so that solution may go on more regularly and more homogeneous varnish obtained. In the case of copal no assortment is made; the small particles of dust rejected as unsuitable for oil varnishes are used. The different pieces are then reduced to the size of a lentil, or ground to a fine powder, for the finer the powder the easier does the resin dissolve; but if the powder be submitted too quickly to the action of the solvent, the particles will agglutinate together and take longer to dissolve. Further, by the solution of the resin in the solvent there is formed a liquid of greater density than the original solvent. This is especially the case with the liquid in contact with or in proximity to the resin. This liquid ought, therefore, to be renewed by agitation, so as to bring fresh portions of the solvent in contact with the resin and keep the solution always of the same density throughout the entire mass. This prevents the powder from agglutinating and prevents solution from only going on at the bottom of the vat.

When working with small quantities the necessary agitation can be produced by manual labour—*viz.*, by using a barrel as the dissolving vessel—driving the bung in tightly and rolling it about the floor until complete solution is effected; or, if cramped for space, a clean empty barrel may be stood on end, the other end knocked out, the resin and solvent added, and the whole stirred from time to time with a large wooden spatula. On the other hand, when working with larger quantities, it is better to use a mechanical agitator; but with open vessels a loss of solvent by evaporation will be unavoidable. It is therefore preferable to use closed mixers in which the resin and solvent are continually stirred.

A very simple form of mixer consists of a truncated tub-shaped wooden vessel, closed by a lid through which there passes an agitator fitted with arms. But in working with this apparatus there is one great disadvantage, caused by it being customary to leave the substance to dissolve of its own accord in contact with the solvent for several hours, so as to obtain more rapid solution when the agitator is put in motion. Now, in working with some resins—shellac in particular—a thick, stiff, swollen mass is obtained, consisting of the undissolved gum embedded in a viscous saturated solution of the solvent, which, on starting the agitator, either breaks the blades or the shaft which carries them.

In preference either a cask laid horizontally, supported by and turning on two pivots placed in the axis of length, or a cylindrical receiver, supported by and turning on two opposite pivots on its cross axis, is used. The opening by which the materials are introduced is closed by a lid, which presses against an india-rubber washer ; a wooden cross-piece, through which two bolts pass, is screwed up by the nuts so as to press strongly against the washer, and thus hermetically seal the apparatus. This arrangement is much better than the old method of closing by means of a screw stopper, as we thus avoid the encrustation in the thread of the screw, and on emptying the apparatus the mouth can easily be freed from any varnish which would solidify there.

A cylinder (A, Fig. 8) may also be used, the two pivots (E E′) of which, placed in the axis of length, are connected by two vertical rods (D D′) with a horizontal shaft (C) bent disymmetrically at its point of attachment; as the shaft revolves each end of the cylinder occupies alternately a higher position, and a more complete commingling of the contents is effected.

It takes about six to seven hours to make forty gallons of spirit varnish, whether agitated by hand, or by a crank, or by means of a mechanical agitator.

Whatever precautions may be taken, the varnish is always impure and must therefore be clarified. This is effected by allowing the varnish to rest quietly in large bottles or metallic reservoirs in a fairly warm place, where there is no vibration. The clear liquid is decanted.

Filtration.—But when very brilliant varnishes, as clear as crystal, are required, they must be filtered at the outset by passing them through a linen filter placed in a wooden funnel fixed above the reservoir. When time is an object, filtration is hastened by using narrow linen bags as filters. These are securely tied to the mouth of the funnel and hang down into the reservoir itself. These bags, measuring about 12 inches broad by 30 inches deep, are enclosed in another envelope of coarse sacking, 8 inches wide by 30 inches deep. Working thus, the interior bag cannot swell completely with the pressure of the liquid, and folds are thereby formed which facilitate filtration in the beginning. Further, a greater number of these filters can be suspended at the exit of a special form of funnel.

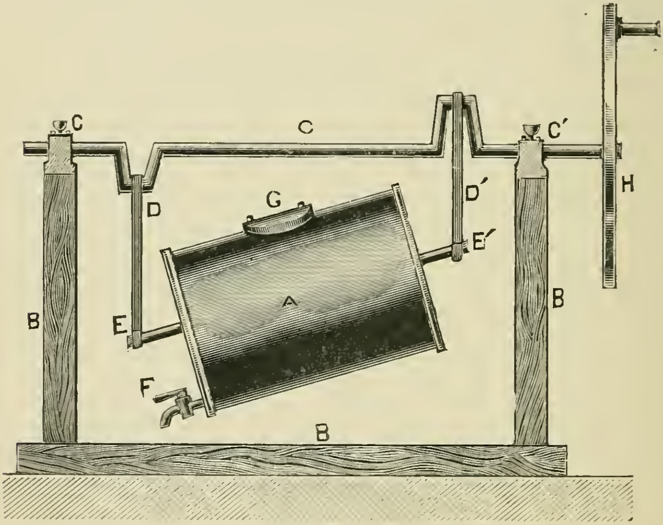

Fig. 8.—Resin Solution Apparatus.

The operation is followed by the indications of a glass gauge on the side of the reservoir. The temperature of filtration should be kept at about 20°.

Loss of solvent occurs in filling the funnel, which loss is accentuated by its being closed by a loosely-fitting lid. Consequently the liquid filling the funnel thickens, filtration becomes slow, and the quantity of thick solution retained by the impurities causes considerable loss.

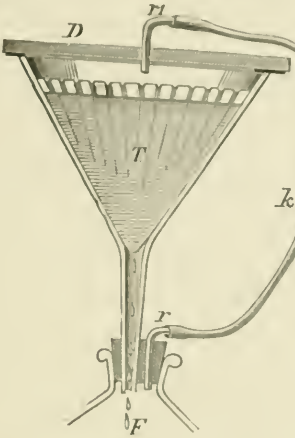

Fig. 9.—Varnish Filter.

F glass receiver closed by two-holed cork, *r* glass, *k* rubber, r^1 glass tubing connecting *F* with *T*, *D* wooden cover with india-rubber ring fitting tight on to ground rim of funnel.

Any loss due to imperfect closing may be done away with by using the following arrangement. The funnel (Fig. 9 *T*) is closed by a tight-fitting lid *D*, which is connected with the lid of the reservoir by an india-rubber tube, the air displaced by the filtered varnish passes from the reservoir *r* through the india-rubber tube into the funnel, and thus occupies the space vacated by the varnish by which it was displaced. Filtration can thus go on notwithstanding the fact that the apparatus is hermetically sealed. This arrangement is very suitable when working on the small scale, and with small apparatus where the funnel fits tightly into the receiver.

Simultaneous Solution and Filtration.—It has been attempted to dispense with agitation in making varnish, by adopting an arrangement which both dissolves the resin and simultaneously filters the resultant varnish. Agitation prevents the finely-powdered resin from agglutinating at the bottom of the vessel, and replaces the resin saturated liquid in contact with the gum resin by fresh quantities of the solvent; or rather, it prevents this saturated solution from forming by continually renewing the solvent in contact with the resin. A somewhat similar occurrence takes place when we sweeten tea with sugar. If we do not stir the tea until we have emptied the cup to the bottom, we might almost as well not sweeten it at all, for the great bulk of the sugar remains either undissolved or in the form of a saturated solution, which prevents further solution taking place without agitation.

Agglutination may, however, be prevented by isolating the particles of resin by admixture with an inert body—thus accomplishing one of the objects of agitation. The second object of agitation can also be effected by arrangement based on the fact that the saturated solution at the bottom is denser than that at the top. By suspending the substance to be dissolved as near the surface of the solvent as may be, the solution formed by the contact of the resin and the solvent, on account of its greater density, sinks to the bottom as soon as formed, whilst its place is taken by a less saturated portion of the solvent, which in turn falls to the bottom; in fact, by taking advantage of the phenomena of diffusion of liquids we are able to dispense with agitation altogether, and as the mixture of resin and inert substance has to be suspended in a bag near the surface of the liquid filtration is effected at the same time.

Equal parts of resin and inert substance are used; the latter may be either fine-washed and ignited silver sand or ground glass freed from too fine particles by sifting. This mixture is first placed in a double envelope of filter paper and then tightly packed in coarse muslin, and the whole finally placed in a linen bag which dips into the solvent. Working in this manner, all impurities and insoluble matter are retained in the bag; moreover, the liquid retained by the impurities and the linen, etc., not being thickened by evaporation, the loss is thus diminished.

On the large scale linen or muslin-lined wire baskets of a size and shape to suit the reservoir are used. Several folds of filter paper are placed on the top of the muslin. The mixture of resin and inert substance is then placed in the basket and covered with filter paper and linen. The baskets are best made of galvanised iron wire. Copper wire should be rigidly excluded.

The spirit varnishes prepared by the methods described are erroneously termed *colourless varnishes*, although they have often a very pronounced colour. Even when the palest resins are used, they are of a more or less deep yellow tint. This tint may be removed by special treatment. Animal charcoal or bone-black in the form of coarse sand is the most energetic decoloriser. The phosphate is removed from the bone-black by treatment with acid, preferably hydrochloric acid, and after well washing and drying the residual carbon is fit for use. Rather coarse charcoal should be used, because although finer charcoal has a more powerful bleaching action, yet the pores of the filter are soon choked up, and filtration almost ceases, thus causing an unnecessary loss of solvent.

The best arrangement consists in running the varnish to be decolorised into a reservoir, with a hole in the bottom, closed by a cork, through which a tube passes which leads the varnish into a receptacle containing the animal charcoal. The extreme point of this receptacle passes through the tight-fitting lid of the funnel, fitted into the cork of the neck of the vessel destined to receive the filtered varnish; a lateral tube carries the air displaced in the receiving vessel up into the space vacated by the filtered varnish in the reservoir containing the varnish to be decolorised. There is thus no loss of solvent during filtration.

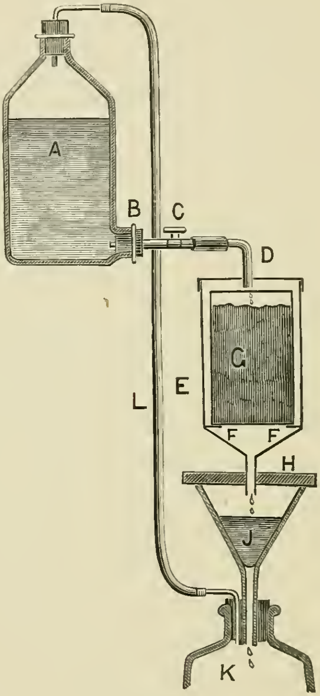

Fig. 10.

Coloured Spirit Varnishes, Spirit Varnish Stains.

In the manufacture of coloured varnishes the colouring matter is added to the finished varnish. If the colouring principle be soluble it is added in the state of solution, preferably in the same solvent as used for the resin. These coloured varnishes are sometimes called transparent varnishes, owing to the surface of the object remaining visible after application. They ought to be very fluid, rich in colour, and dry rapidly to an elactic lustrous coating.

These varnishes are generally made by dissolving liver-shellac, or sandarach, or mixtures of the two in methylated spirit for dark, and bleached shellac for pale, colours. They are generally made of greater body than usual, because the colouring solution thins them down slightly unless an aniline dye be used, when only a very insignificant quantity imparts the desired tint.

If an earth or mineral colour—insoluble in the solvent used for the resin—be used to colour the varnish, the two are intimately mixed in a paint mill. The colour ought to be in an impalpable powder, and bright, lustrous varnishes are preferred to those made from copal and shellac. The grinding is done with a part of the varnish only; it is afterwards thinned down with the remainder.

The colours used are very numerous, *viz.*, lamp-black, ultra-marine blue, chrome green, vermilionette, etc., etc. Zinc oxide should not be used: it forms zinc resinates, and thus decomposes the methylated spirit varnishes. For the same reason red lead should not be used, as it solidifies in a few days.

Various substances are used to impart some property or other: thus spirit varnishes are made more adherent and elastic by the addition of 1 per cent. of boracic acid, especially when metallic objects are to be varnished, but not more than 1 per cent. should be used, otherwise it has an opposite effect.

Hot Process.—To shorten the time occupied in the process the solution of the resin in the solvent is effected with the aid of heat.

If the solvent used be not too volatile, *e.g.*, alcohol, the operation may be conducted in a glass flask, the lower half of which dips into a water bath, being kept in position by the usual rings. The flask should be frequently shaken to hasten solution and economise solvent. In the case of intractable resins, such as copal and some kinds of Manilla, the naked fire must be resorted to, but great care must be taken to prevent the mixture taking fire.

Varnishes made by the aid of heat are more brilliant, but they are darker, and do not bind so well as those made in the cold.

To prevent loss of solvent, especially when working with very volatile solvents, Andres uses a cylindrical digester with a diameter about equal to its height, enamelled inside, resting upon a tripod placed in a water bath which narrows at the top so as to catch hold of the digester; the mouth of the digester is provided with a flange, to which the lid is fastened by bolts and nuts after the previous insertion of an india-rubber or leather washer. A shaft carrying a mechanical agitator passes through the lid, and a tube carries the evolved vapours to a condenser placed above the digester in such a

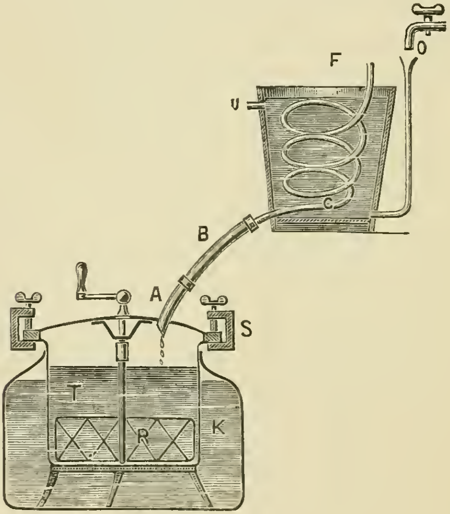

FIG. 11.

manner that the condensed solvent falls back into the digester.

With spirits of turpentine, coal-tar naphtha, or petroleum naphtha, the water in the bath is heated to boiling; in the case of chloroform, bisulphide of carbon and wood-spirit, the heat should not exceed 50° C.; whilst in the case of ether and petroleum ether and gasoline 40° C. should not be exceeded, care being at the same time taken to add a little ice to the condenser water to ensure complete condensation.

This plant can also be used to thicken spirit varnish by collecting the condensed solvent apart instead of returning it to the digester.

When working on the large scale, and also when using very volatile solvents, regular extraction plant may be employed, similar to that used for fat extraction by solvents on the large scale, consisting of three enormous canisters placed one above the other. The solvent is heated in the first canister; the evolved vapours led to the top canister, where it is condensed; the condensed liquid running down into the middle canister, in which the resin to be dissolved is placed; finally, the solution runs baçk into the first canister, where the solvent again distilled off re-ascends to the top canister, to be again condensed and fall into the middle one, and so on until complete solution is effected. The advantage of this plant is that large quantities of very intractable resins may be dissolved with a minimum of solvent. Its compactness also enhances its value.

PROPERTIES OF VARNISHES YIELDED BY DIFFERENT RESINS.

Whatever influence the solvent used may have on the quality of the resultant varnish, it will be readily conceded that it is the unchanged resin alone which—after the evaporation of the solvent—constitutes the coat, and imparts to the varnish its distinguishing properties—corresponding with, or are identical to, those possessed by the resin previous to solution.

Hard resins yield bright but brittle varnishes; soft resins yield varnishes which produce less lustrous but more elastic coats, due to the essential oil contained in the resin, but the elasticity diminishes as the essential oil disappears. Finally, by dissolving several resins together, we can more or less do away with the objectionable features of some, whilst modifying the properties of others, thus securing a varnish adapted in every way for the object in view—an object most likely which could not be attained by the use of any one single resin. Again, it is sometimes possible by a judicious combination of cheaper resins to produce a varnish similar too, if not identical with, that produced by a more costly resin.

It behoves us, therefore, to study the character of varnish which each individual resin produces, and see whether or not we can in any way improve upon it by the addition of other resins. But, first of all, we must know the relative hardness of each resin, *i.e.*, the position it occupies in the following scale of hardness, which starts with the hardest resin, amber, and closes with asphaltum, each resin being softer than the one above and harder than the one below it.

SCALE OF HARDNESS OF RESINS.

Amber.		
Copal.	Hard.	Zanzibar.
		Mozambique.
		Madagascar.
	Medium.	Sierra Leone.
		Angola, Red.
		Benguela.
		Bastard Angola.
		Pebble Copal.
		Akra.
		Benin.
		Loango.
		Gaboon.
		Congo.
		Sierra Leone.
		Angola, White.

Copal (*continued*).	Soft.	Manilla. Borneo. Singapore. South American (Brazil, Guiana). Cochin China.
Dammar.		
Shellac.		
Mastic.		
Sandarach.		
Rosin.		
Elemi.		
Turpentine.		
Burgundy Pitch.		
Asphaltum.		

The quality of the resultant varnish depends to a great extent upon a judicious selection and admixture of these different resins.

Having regard only to the predominant resin, on the characteristics of which depends the suitability of the varnish for special purposes, we can classify spirit varnishes as follows :—

Varnish.	Specially adapted for
Amber - - -	Metals, photography, bookbinding.
Copal (fused) -	Bookbinding.
Copal (soft) - -	Interior decoration.
Dammar - -	Interior decoration, and objects not much handled.
Shellac - - -	Articles subject to wear and tear, toys, leather, cardboard, blackboards, French polish.
Do. bleached	Colourless varnishes.
Mastic - - -	Toys and turned articles, pictures, cardboard, golden lacquer.
Sandarach - -	Pounce cabinet work, water-colours, visiting cards, varnish to be used to thin down colours, golden lacquer, white spirit varnish, wood carving, metal plate work, photographic negatives.
Rosin, etc. - -	Poor quality picture varnish, etc.
Asphaltum - -	Varnish to be used as a vehicle for colours, metals.
Caoutchouc - Gutta-percha - Celluloid - -	Flexible articles, electrical insulation, photographic negatives, waterproof varnishes.

AMBER VARNISHES.

Owing to the high price of the hardest and finest copals attempts have often been made to substitute amber for them. But although amber yields varnishes of great hardness and durability, it has the great disadvantage of darkening on heating, especially towards the end of the operation. Hence this operation has been conducted in several successive stages, at the end of each of which the soluble portions are extracted with spirits of turpentine, the product of the first stages being lighter in colour than of those later on. But this process would only be applicable on the small scale. Schrader and Dunicker have, however, devised an apparatus which in one single operation accomplishes the object in view, and in which over half a ton of amber can be treated in one operation. The amber is melted in a cylindrical boiler, fixed above a furnace built of masonry, and inclined in such a manner that the products as they melt run away by a discharge tube connected with the bottom of the boiler, whilst the vapours escape through a condenser connected with the top, so as to avoid all oxidation steam at a pressure of 1 or 2 atmospheres; or other inert gas, such as carbonic acid or nitrogen, is injected into the boiler to displace the air; then after a sufficient interval,

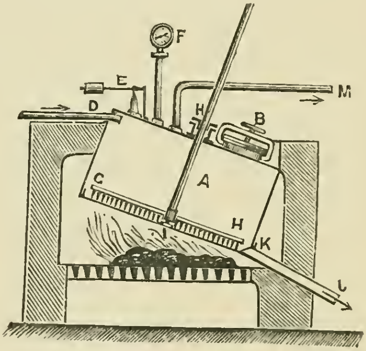

FIG. 12.

indicated by experience, the melted products are run off, thus avoiding the high temperature necessary to fuse the whole mass. The melted products run directly into closed receivers, fitted with a disengagement tube for the escape of air and vapour.

Three grades of fused amber are obtained in this manner, *viz.*, 30 per cent. of very bright and lustrous resin, 60 per cent. good resin, very hard, 10 per cent. dark-coloured resin. Whatever method is employed the action of heat on hard resins renders them easily soluble in the different solvents, and the resultant varnish is so much the better in quality the more care and skill has been displayed in the operation and in the choice of resin submitted thereto.

AMBER SPIRIT VARNISH.

As we have seen, amber is insoluble in all varnish solvents, and that it is necessary to subject it to a previous fusion so as to render it soluble. But the amber, which has been subjected to this process and lost about 20 per cent. of its weight, has also become softer than natural amber: during the process the resulting varnish presents the disadvantage of being rather tacky. Fused amber, or pyro-succin, is never used in varnish-making alone, except in the case of photographic varnish, where its transparency and resistance recommend it. The usual solvent in this case is chloroform. Sometimes 22 parts of fused amber are dissolved in 14 parts of spirits of turpentine: but this varnish dries much more slowly. In many cases the fused amber is associated with a resin which will impart elasticity to the varnish. For example, 10 to 15 parts of fused amber are dissolved in 15 to 20 parts of spirits of turpentine, according to the quality of the amber, and 1 part of Venice turpentine added.

In the case of ordinary spirits, varnish sandarach is added, but the varnish is more brittle, or mastic, elemi or Venetian turpentine, which give elasticity. The following formula is in very general use:—

Fused amber	4 parts
Sandarach	4 to 6 parts
Mastic	1 part
Venetian turpentine	1 part
Alcohol, 90°	12 to 24 parts.

According to whether a dark or lighter-coloured varnish is required. Often a little camphor is added, ½ part for example, so as to facilitate solution.

Copal Spirit Varnish.

By Solution in a Single Solvent.

By Solution in Ether.—Attempts have been made to obtain a quick drying varnish by dissolving copal in ether, but, according to Violette, and, contrary to the assertion of Tingry, copal does not dissolve directly in ether without being previously heated so as to drive off one-fourth or one-fifth of its weight.

The fused or pyro-copal thus obtained is soluble in ether, but it is not so hard as the original copal, and may even be tacky like fused copal.

Process.—500 parts of finely pulverised pyro-copal are run into a flask, and 1000 parts of sulphuric ether gradually added, shaking after each addition; the resin rapidly dissolves, and after complete solution the whole is allowed to stand for some time, and the clear liquid is filtered through linen or paper, taking care to prevent loss by evaporation in the manner previously indicated.

Before using this varnish, which possesses a beautiful, brilliant lustre, it is advisable to prime the surface to which it has to be applied with an essential oil, such as spirits of turpentine, oil of spike, or rosemary oil, so that the varnish does not thicken too rapidly and the coating pit from the escape of enclosed ether.

Solution in Alcohol.—Hard copals should be heated to drive off 25 per cent. of their weight. They then become soluble in alcohol. Although hard copal is insoluble in alcohol without this treatment, the medium and soft copals do not require it, as they dissolve very readily in their raw state, yielding colourless varnishes which are not tacky. The softer the resin the easier does it dissolve. Manilla and Borneo copals especially yield very bright solutions, which quickly clarify, and leave a very brilliant coating. Benguela, Angola and Sierra Leone copals yield darker and duller varnishes, because they take more alcohol to effect solution, and consequently the coat is thinner. Even however in the case of Borneo and Manilla copal different samples do not all dissolve equally well, but sometimes form a viscous mass, which strings strongly and only dissolves but very slowly and partially, always leaving an insoluble residue. These kinds should not be used until after they have been finely pulverised and exposed to the air for a very long time, twelve months in some cases. Treated in this manner, they dissolve much more easily and leave a far less bulky residue. Venice turpentine or elemi may be added to give elasticity.

The recipes generally given vary according to the copal used and the elasticity desired in the varnish within the following limits:—

Copal	10 parts
Alcohol, 95 per cent.	21 to 18 parts
Elemi or Venice turpentine	5 to 3 parts

Very often medium hard copals, difficultly soluble in alcohol, are heated before solution, but not to such an extent as to convert them into pyro-copal: this treatment afterwards facilitates the solution of the resin in methylated spirit.

Two parts of copal are melted at a very gentle heat and when the resin is completely fluid 1 part of Venice turpentine is added, which is well mixed, and when the mass is perfectly homogeneous it is poured on a glass plate and allowed to cool. This mixture is pulverised and dissolved by the aid of heat on the water bath in 4 parts of 95 per cent. alcohol.

Three parts of copal are cautiously melted, and whilst in a state of fusion poured in as fine a stream as possible into a large quantity of water.

After cooling, the water is decanted, and the well-dried copal is ground and mixed with 3 parts of sandarach and 3 parts of mastic, both finely pulverised and dissolved in the usual manner in 88 per cent. alcohol to which 2 parts of Venice turpentine have been added.

Solution in Spirits of Turpentine.—Only pyro-copal can be used, which is dissolved in 1½ parts of spirits of turpentine. But this gives a deep brown-coloured varnish. Very often camphor or oil of rosemary is added to assist solution, but this has the effect of softening the varnish and causing it to dry "tacky" and more slowly.

A very durable varnish is made by dissolving 3 parts of pyro-copal and 2 parts of amber in 20 parts of spirits of turpentine. One part of Venice turpentine is then added to the solution gently heated on the water bath.

Attempts have been made to dissolve copal in spirits of turpentine without previous fusion, the processes being based on the facts (1) that copal becomes more soluble after prolonged exposure to the air, and (2) that exposure to air and light during the process facilitates solution in spirits of turpentine. Tingry showed that when a small quantity of copal is added to this solvent and the mixture exposed to air and sunlight, the solution dissolves copal very readily without the aid of heat.

Process.—Spirits of turpentine is rectified so as to produce 90 per cent. of rectified spirits, of which 480 parts are run into a bottle and 10 parts of finely ground copal added, and a loosely-fitting cork inserted into the mouth of the bottle, and the whole abandoned to air and light. Little by little the copal dissolves, and when complete solution is effected, the latter may be used to dissolve a larger quantity of copal. But some kinds of copals do not dissolve in this way.

Solution in Acetone.—The greater number of copals dissolve partially in acetone (3 parts of acetone for 1 of copal), and the resultant varnish dries hard and brilliant. If the acetone be distilled off the residual dry copal dissolves much more easily and in a smaller quantity of acetone.

Solution in Mixed Solvents.—In making copal varnish it is preferable to work with mixed solvents, for each of the distinct resins constituting copal requires a separate solvent. By only using a single solvent it is true that a certain proportion of the copal dissolves, but in most cases there is left a swollen residue which absolutely resists solution.

Ether Alcohol and Spirits of Turpentine.—This varnish, due to Bottger, is perfectly limpid; it may be thinned down with ether, and the addition of a little Venice turpentine prevents it from drying too quickly. Finally, it may be made very quickly: 20 parts of finely ground copal are mixed with 5 parts of camphor and added to 60 parts of ether. It is left to stand for twenty-four hours, after which the whole is run into a mixture of 2 parts of spirits of turpentine and 20 parts of 98 per cent. alcohol. After stirring the solution assumes the form of a clear, thick, homogeneous liquid. On standing for several days it separates into two layers, the lower richer in copal, and the upper poorer therein and absolutely limpid. The top layer, which forms an excellent varnish, may be drawn off, and the bottom layer again treated with camphorated ether which still contains undissolved swollen copal.

According to Heeren, only West Indian copal with a smooth surface, bright colour, often colourless, in irregular fragments with a rounded surface and conchoidal fracture, should be used, whilst East Indian copal, in small pieces with a wrinkled surface and yellow colour, should be rejected. The latter do not dissolve but form a gelatinous mass.

The best proportions for the mixed solvent are :—

Alcohol, 98 per cent., by weight - - -	6 parts
Sulphuric ether, by weight - - - -	10 ,,
Spirits of Turpentine, by weight - - -	40 ,,

Sixty parts of copal dissolved in this mixture give a varnish of the consistency of linseed oil—the gum does not swell or gelatinise. Solution may be facilitated by the aid of a very gentle heat.

The largest, brightest and palest pieces should be chosen, but as some of these may dissolve badly it is best to try each lump separately by dropping it into a test tube containing a small quantity of the solvent; it ought to dissolve in a few minutes without gelatinising. When the requisite quantity of copal which has stood this test has been obtained, the resin is dissolved and filtered if need be. A quick drying, limpid, almost colourless varnish is thus obtained, but which, like all turpentine varnishes, is still tacky for some time.

Alcohol and Spirits of Turpentine.—As Sierra Leone copal consists of a mixture of two resins, the one soluble in spirits of turpentine, the other in alcohol, a mixture of these forms the most rational solvent for this resin.

The operation is performed, according to Andés, by treating in an enamelled iron vessel 2 parts of finely pulverised Sierra Leone copal and 1 part of 95 per cent. alcohol, stirring the mass until the resin soluble in alcohol is dissolved and the insoluble resin separates as a greyish-white gelatinous mass. The alcoholic solution is decanted, and constitutes by itself an excellent spirit varnish. One and a half parts of rectified spirits of turpentine are now added to the residual resin and the whole stirred until completely dissolved. By mixing the two solutions an uncommonly good varnish is produced.

Andés has completely dissolved in this same solvent—

obtained by mixing equal proportions of alcohol and spirits of turpentine—Manilla, Borneo, Sierra Leone and Benguela copals; on the other hand, Zanzibar and Angola swelled without dissolving even by the aid of heat.

Alcohol and Oil of Lavender.—Four parts of copal are finely pulverised with 1 part of camphor, 8 parts of oil of lavender are added, and after twelve hours' contact 15 parts of 98 per cent. alcohol.

Spirits of Turpentine and Oil of Lavender.—Five parts of finely pulverised copal are dissolved in a mixture of 6 parts of oil of lavender and 20 parts of spirits of turpentine.

Alcohol and Acetone.—Three parts of pulverised copal are added to 6 parts of acetone, and after twenty-four hours 10 parts of 96 per cent. alcohol are added; the solution is aided by heating gently on the water bath, and when complete half a part of Venice turpentine is added. A very tenacious, elastic varnish is thus produced.

More Complex Mixtures.—Finally, according to the object in view, much more complex mixtures may be used as solvents. We have already indicated the use of a mixture of spirits of turpentine, wood-spirit, benzol and carbon disulphide.

Similarly, Valenta used a mixture of 300 parts of ether, 200 of acetone, and 10 of chloroform, and thus prepared an excellent photographic varnish (negatives), and giving a hard, durable coat, by dissolving in this mixture 30 parts of Angola copal and 30 parts of yellow amber *débris*. A part of these resins dissolve either in the cold or after heating in an apparatus connected with a reflux condenser, and there is thus obtained a faintly yellow, limpid liquid which gives a brilliant and transparent coating.

Whilst recognising the great advantage of these mixed solvents, it must be borne in mind that they do not act similarly with all varieties of copals. The published formulæ scarcely ever mention the variety operated on. It is therefore always necessary to make a preliminary trial on the sample of copal to be treated.

The following further recipes for copal spirit varnishes may be useful :—

1.	Manilla copal	160 lbs.
	Venice turpentine	40 ,,
	Methylated spirit	36 gals.
2.	Manilla copal	160 lbs.
	Burgundy pitch	50 ,,
	Methylated spirit	36 gals.
3.	Manilla copal	50 lbs.
	American rosin	30 ,,
	Burgundy pitch	30 ,,
	Methylated spirit	20 gals.
4.	Manilla copal	50 lbs.
	Australian sandarach	50 ,,
	Venice turpentine	30 ,,
	Methylated spirit	30 gals.
5.	Manilla copal	50 lbs.
	Australian sandarach	50 ,,
	American rosin	30 ,,
	Venetian turpentine	30 ,,
	Methylated spirit	31 gals.
6.	Borneo copal	140 lbs.
	Venice turpentine	60 ,,
	Methylated spirit	40 gals.
7.	Borneo copal	140 lbs.
	Burgundy pitch	70 ,,
	Methylated spirit	40 gals.
8.	Borneo copal	110 lbs.
	Elemi	40 ,,
	Methylated spirit	33 gals.
9.	Angola copal	100 lbs.
	Burgundy pitch	40 ,,
	Methylated spirit	22 gals.
10.	Benguela copal	100 lbs.
	Burgundy pitch	50 ,,
	Methylated spirit	25 gals.
11.	Sierra Leone copal	100 lbs.
	Venice turpentine	30 ,,
	Methylated spirit	12 gals.

Dammar Varnish.

Dammar varnishes are inferior to copal varnishes made from good sorts of copal. For certain purposes, however, they are preferred on account of their lustre ; moreover, they are generally brighter and paler than amber or copal varnishes, but their great fault is want of durability.

Solution in Ether.—Although dammar is only partially soluble in ether, the ethereal solution is used as a varnish, particularly as a picture varnish, on account of its lustre and transparency. It is necessary to work with dilute solutions, otherwise the rapid evaporation of the ether causes the formation of a superficial layer, which rapidly hardens to a pellicle, which hinders the varnish underneath from drying, thus doing away with any benefit derived from the use of a quick drying varnish.

Spirits of Turpentine.—This is the best solvent for dammar. It dissolves therein completely in the cold, but it is preferable to work with the aid of heat, so as to drive off moisture which is present in large proportion, and which would otherwise yield a cloudy varnish; the heat should not however be so great as to darken the varnish. Venice turpentine is added to impart greater body and elasticity.

Process.—The Venice turpentine is melted in an enamelled, round-bottomed, cast-iron pan, 25 ins. deep by 18 ins. wide, and heated over a suitable furnace built into brickwork. The roughly-crushed and well-sifted dammar is then added, rejecting the powder which passes through the sieve, because it gives a deeper coloured varnish. The mass is well stirred and covered with a tight-fitting lid. The melting of the dammar takes from an hour to an hour and a half according to the quantity used. Towards the end the boiling mass rises in the pan; the process has therefore to be watched carefully, and stirred with a wooden agitator. When the mass is completely fluid the pan is removed from the fire, and after cooling a little the spirits of turpentine is added, at first a little at a time, whilst stirring slowly; when the mass is homogeneous the remainder is added. The resultant varnish is filtered through coarse linen and allowed to stand for several weeks to completely clarify. Sometimes the dammar is heated by itself, and to the melted mass deprived of its moisture the solid turpentine is added, and the operation continued as before. When it is not desired to add any solid turpentine it is best to add a little spirits of turpentine to the dammar at the beginning to prevent the resin from sticking to the sides of the pan.[1]

The proportions generally used are:—

Dammar	20 parts
Spirits of turpentine	21-24 ,,

---

[1] Far better results are obtained by working with a steam jacketed pan.

If solid turpentine be used the proportions for 20 parts of dammar vary between—

Spirits of turpentine - - - - - -	22 to 32 parts
Solid turpentine - - - - - - -	4 to 20 ,,

According to the body and elasticity desired in the varnish; the quantity of spirits of turpentine increasing proportionately with the solid turpentine.

Each part of dammar requires 1 part of spirits of turpentine, whilst each part of solid turpentine requires ½ part of spirits; these proportions give a good bodied varnish. With cheap varnishes the amount of solid turpentine is increased, but it must be borne in mind that if more than 2 parts of solid turpentine to 1 of spirits beyond that which is required for the solution of the dammar be used the varnish will be deficient in body and dry very difficultly.

After standing these varnishes are bright, but they are not absolutely transparent. To remedy this, there is added for every 20 parts of finished varnish 1½ parts of absolute alcohol, and the whole well stirred; the solution soon becomes clear and transparent, but slightly darker in colour. This observation seems to support the opinion of Miller, who attributed the fact that dammar varnish often becomes dull to an imperfect solution of the resin. For the same reason Poppinghausen recommends the use of benzol in the place of spirits of turpentine. He says that solution is perfect and that a better and quicker drying varnish results.

Photographic Varnish.—According to Valenta, a good photographic varnish (negative) is obtained in the cold, or even for proofs upon paper if suitably diluted, by dissolving dammar resin, 8 parts, in a mixture of benzol, 90 parts, and absolute alcohol, 10 parts, but it is necessary to gelatinise the resin in alcohol before adding the benzol.

According to Lamb and Boyd, an excellent varnish is obtained by adding amylic alcohol to spirits of turpentine or to benzol in a proportion varying from 20 to 80 per cent. of the total solvent according to the rapidity of drying desired. For quick drying varnishes 20 per cent. of amylic alcohol is enough; the proportions recommended are:—

Dammar	20 parts
Benzoin	10 ,,
Solvent	120 to 240 parts

According as the varnish is to be applied on metallic surfaces, on wood, or on paper.

SHELLAC VARNISHES.

Shellac varnishes have a wide application either on wood or on metal, or for covering flexible objects such as leather, paper, etc.

The solvent employed is almost always methylated spirit, to which spirits of turpentine, benzol, or petroleum ether is added, so as to dissolve the wax of the shellac and thus produce a limpid varnish, but in this case strong alcohol is used, 96 to 98 per cent. (Sp. g. 0·81245 to 0·8390.)

A very clear varnish may be obtained by stirring the alcoholic solution with a quantity of white lead equal to that of the shellac used. After this treatment, which ought to last at least two days, the varnish is allowed to stand and then decanted. Either bleached or coloured shellac is used, according to the quality and depth of colour required in the varnish. With bleached shellac, it must be borne in mind that it is apt to contain chlorine addition or substitution products which must be guarded against. Further, under the influence of energetic oxidising agents, many kinds of shellac become very difficultly soluble in alcohol. This may be remedied by covering the broken-up shellac with ether, and leaving it in contact therewith for twelve hours; the solution is then effected more readily.

Alcohol.—Shellac dissolves in cold alcohol. By using 5 to 12 parts of 90 (Sp. g. 0·83415) to 96 per cent. (Sp. g.

0·81245) alcohol to each part of shellac a turbid varnish is obtained, which is generally used in that condition, especially in the case of furniture varnishes. The varnish becomes more elastic on the addition of 1 to 3 per cent. of alcohol.

When applied to metals the adherence of the varnish is increased by the addition of not more than 0·3 to 0·5 per cent. of boracic acid.

Graeger's Process.—As before stated, when a limpid solution is desired, a little benzene or petroleum ether is added. The following process, due to Graeger, may be substituted, but it has the disadvantage of eliminating the wax from the shellac, and consequently it is more brittle. One part of shellac is dissolved on the water bath in 4 parts of alcohol of 92 per cent., and to the solution distilled water is added a little at a time until a cheesy mass is deposited and the liquid becomes perfectly clear: this happens when the amount of distilled water equals about $\frac{1}{3}$ of the alcohol used. The whole is filtered through filter paper, which is washed with 67 per cent. alcohol. The filtered liquid is distilled, and the residue, dried on the water bath until of constant weight, is dissolved in 96 per cent. alcohol.

When it is desired to produce an elastic varnish, such as is used for leather, metal foil and plate work, etc., Venice turpentine, mastic, Burgundy pitch, and elemi are added to the varnish. Generally the alcoholic solution of shellac is allowed to stand for several days, often even filtered, before the addition of these substances.

Filtration through paper is a laborious operation requiring frequent changes of filters, owing to the pores being rapidly choked by the wax. Filtration is effected much more easily by using double the quantity of alcohol to dissolve the shellac, and recovering the excess after filtration by distillation.

When common varnishes are made, the duration of the process may be shortened by adopting the following method.

The necessary quantity of Venice turpentine is heated in a pan, and when melted, and after it has begun to emit vapours, the shellac is added in two or three portions, without stirring, otherwise the shellac would aggregate together into lumps difficult to dissolve; moreover, no further addition of shellac should be added until the mixture becomes fluid and homogeneous after the addition of the previous portion. When all the shellac has been added and the mass become perfectly fluid the pan is withdrawn from the fire, allowed to cool for a few minutes, and the alcohol added.

Numerous recipes are given for elastic varnishes, apparently of very different composition, but on examination they can all be resolved into the following unique formula. Dissolve 1 part of shellac in 5 parts of alcohol of 96 per cent. strength, and add to this solution, according to the qualities and elasticity desired in the varnish, either mastic or Venice turpentine, Burgundy pitch, sandarach, benzoin or camphor, or a mixture of these different products in such proportions that the quantity added is comprised between $\frac{1}{10}$ of a part and 1 part.

This is illustrated in the following recipes for varnishes to be applied to very different purposes:—

			LEATHER VARNISH	
	Varnish for Bottle Capsules. Parts.	Bookbinders' Varnish. Parts.	Ordinary. Parts.	Nubian Blacking. Parts.
Shellac - - -	1	1	1	1
Alcohol, 96 per cent. -	5	5	5	5
Substances to produce elasticity - -	$\frac{1}{10}$ Venice Turpentine.	$\frac{6}{10}$ Mastic, Sandarach, Camphor.	1 Venice Turpentine, Mastic, Sandarach.	1 Camphor.

The required colouring matter previously dissolved in a small quantity of alcohol is then added. Thus for gold lacquers, dragon's blood, gamboge, saffron and annatto are added in alcoholic solution. Moreover, the varnish may be thinned down with alcohol if too concentrated for the object in view.

Iridescent bronze varnishes, which are so much employed now-a-days to produce iridescence on millinery ornaments, sprays, feathers, birds' wings, etc., are coloured by fuchsine (magenta crystals). The iridescence is first produced by coating the object with a magenta-tinted varnish, and when it is well dried it is placed on filter paper placed over bleaching powder, thinned down to a thin paste with water. Under the influence of the escaping chlorine, in a few hours the colouring matter is oxidised, thus producing the desired iridescence.

Finally, for certain particular varnishes, leather varnishes for instance, complicated recipes have been given into which various substances enter. According to one recipe, 80 parts of shellac and 3 parts of wax are dissolved in 15 parts of alcohol and 2 parts of castor oil, the whole is heated to the consistency of syrup and applied to the leather with a brush moistened with alcohol.

Similarly, Sitner recommends as a brilliant varnish for leather to digest 2 parts of shellac with 10 parts of 95 per cent. alcohol in a closed vessel in a warm place for two or three days, stirring the liquid daily. On the other hand, ¼ part of dry Marseilles soap is dissolved in 4 parts of hot alcohol and ½ part of glycerine added, the whole is well stirred, and this solution is added to the shellac solution. Finally, to impart a beautiful black brilliant lustre, ½ part of myrosine dissolved in 1¼ parts of alcohol is added, and the whole allowed to stand in a warm place for a fortnight.

But these empirical, or rule of thumb, receipts are not, strictly speaking, spirit varnishes.

Blackboard Varnish.—Shellac varnish is employed to "slate" blackboards. 25 parts of shellac and 7 parts of sandarach are dissolved in 25 parts of alcohol; on the other hand, 3 parts of gutta-percha are dissolved in 14 parts of spirits of turpentine; after cooling, the two solutions are mixed and incorporated with 50 parts of finely pulverised emery, and 12 parts of very fine bone-black. The board is coated whilst in a vertical position and the alcohol inflamed, and another coat applied as soon as possible. This operation is repeated five or six times, and a remarkably fine grain is obtained at the finish, with a beautiful, flat, lustreless surface.

The following process is also employed: 3½ parts of shellac are dissolved in 20 parts of alcohol, and 5 parts of emery added to the solution, and then 2 parts of very fine bone-black, and the whole ground for a very long time. The varnish is applied to the board, the alcohol inflamed, and the operation repeated.

If it be desired to dispense with setting fire to the alcohol, the operation may be conducted as follows: 50 parts of sandarach, 20 parts of pyro-copal, 100 parts of shellac, 3 parts of Venice turpentine are dissolved in 400 parts of 96 per cent. alcohol and 40 parts ether; to this solution there is added a mixture of 15 parts of lamp-black, 5 parts of ultramarine blue, and 100 parts of emery. It is not necessary to inflame the spirit, and if there be a few shining spots, these can be removed by rubbing with amber, so as to obtain a lustreless surface.

Shellac Water Varnishes. — Before finally disposing of shellac varnishes, it is necessary to describe the varnishes known as water varnishes and ammonia varnishes. The water varnishes are obtained by dissolving shellac in a solution of borax. One part of borax is dissolved in 20 parts of hot water, it is preferable to take distilled water; to this solution there is added, little by little, 3 parts of white shellac, taking care each time not to add any more until the previous lot is dissolved. The whole is allowed to stand, and the wax eliminated from the shellac is separated by filtration.

The solution may either be applied to *leather* already stained black, or the solution itself may be stained with aniline black before application to the leather ; in either case a beautiful coating is obtained.

The ammonia varnish is used by *hatters* to replace the alcohol solution. Three parts of orange shellac, 1 part of sal ammoniac, 6 to 8 parts of water, are shaken in a flask. After twelve hours' contact the whole is heated until completely dissolved.

The ammonia varnish, with the addition of alcohol, constitutes the crystal varnish used for *photographic* negatives. Alcohol would give an opaque coating, whilst ammonia gives a brilliant one. According to Valenta, the best method is to dissolve ammonia gas in absolute alcohol ; to 100 grains of this solution 8 parts of shellac are added, and on heating on the water bath a yellow liquid is obtained. The proportion of shellac may be increased, so as to have a better bodied varnish. If aqueous ammonia be used, the coating does not dry so well.

The following additional recipes for varnishes, in which shellac is the chief ingredient, will show their extensive and varied use :—

Shellac Spirit Varnishes.

1. Brown Polish.

Oxidised turps	50-80 lbs.
Garnet shellac	80 ,,
Methylated spirit	45 gals.

2. Pale Polish.

Orange shellac	90 lbs.
Methylated spirit	45 gals.

3. Darker Polish.

Venice turpentine	50 lbs.
Garnet shellac	100 ,,
Methylated spirit	45 gals.

4. White Polish.

Bleached shellac	100 lbs.
Methylated spirit	40 gals.

5. Floor Varnish.

Sandarach	50 lbs.
Orange shellac	80 ,,
Methylated spirit	45 gals.

6. Bookbinders' Varnish—Pale.

Venice turpentine	40 lbs.
Orange shellac	100 ,,
Methylated spirit	40 gals.

7. Bookbinders' Varnish—Dark.

Burgundy pitch	20 lbs.
Garnet shellac	100 ,,
Methylated spirits	40 gals.

7a Bookbinders' Varnish.

Shellac	82½ lbs.
Spirits of turpentine	3 gals.
Methylated spirit	80 ,,

8. Bookbinders' Varnish—White.

Venice turpentine - -	50 lbs.
Bleached shellac - -	100 ,,
Methylated spirit -	12 gals.

9. White Polish.

	I.	II.	III.
Bleached shellac	40	50	60 lbs.
Methylated spirit	27	27	27 gals.

10. Pale Polish.

	I.	II.	III.
Bleached shellac	40	50	60 lbs.
Orange shellac -	10	10	10 ,,
Methylated spirit	27	27	27 gals.

11. Pale Brown Polish.

	I.	II.	III.
Orange shellac -	40	50	60 lbs.
Methylated spirit	28	28	28 gals.

12. Brown Polish.

	I.	II.	III.
Orange shellac -	40	50	60 lbs.
Garnet shellac	20	20	20 ,,
Methylated spirit	30	30	30 gals.

13. Dark Brown Polish.

	I.	II.	III.
Garnet shellac	40	50	60 lbs.
Methylated spirit	25	25	25 gals.

Mixed Shellac Spirit Varnishes.

1.	Shellac, orange - -	40 lbs.
	Venice turpentine - -	30 ,,
	Manilla copal -	50 ,,
	Methylated spirit - -	34 gals.
2.	Shellac, orange - -	20 lbs.
	Burgundy pitch - -	20 ,,
	Manilla copal - -	50 ,,
	Methylated spirit - -	23 gals.
3.	Garnet shellac - -	40 lbs.
	Burgundy pitch - -	30 ,,
	American rosin - -	50 ,,
	Methylated spirit - -	27 gals.
4.	Garnet shellac -	30 lbs.
	Burgundy pitch - -	30 ,,
	American rosin - -	70 ,,
	Methylated spirit -	27 gals.
5.	Bleached shellac	40 lbs.
	Venice turpentine -	20 ,,
	Sandarach - -	40 ,,
	Methylated spirit	23 gals.
6.	Bleached shellac -	20 lbs.
	Venice turpentine -	20 ,,
	Window glass rosin -	60 ,,
	Methylated spirit	20 gals.
7.	Orange shellac - -	40 lbs.
	Venice turpentine	150 ,,
	Sandarach - -	40 ,,
	Methylated spirit - -	12 gals.

Spirit Stiffening for Hats (Ure's).

Orange shellac - -	7 lbs.
Sandarach - - - - - -	2 ,,
Mastic - - - -	4 ozs.
Amber rosin - -	½ lb.
Copal spirit varnish - - - -	1 pint
Methylated spirit or wood naphtha -	1 gal.

The shellac, sandarach, mastic and rosin are dissolved in the spirit and the solution of copal is added last.

ALKALI STIFFENING (URE'S).

Shellac	7 lbs.
Amber rosin	1 ,,
Gum thus	4 ozs.
Mastic	4 ,,
Borax	6 ,,
Copal spirit varnish	½ pint

The borax is first dissolved in a little warm water, say a gallon, and the solution run into a copper pan (heated by steam) together with the resins, and boiled until of proper consistency. If it sets when run on an inclined cold slab it requires more water. When the resins seem dissolved a half pint of wood naphtha is added and the copal varnish. The solution is then run through a sieve when it is perfectly clear and ready for use. Both the foregoing formulæ would appear to yield too stiff a solution for practical use.

MASTIC VARNISH.

This varnish is highly esteemed for pictures in oil, water-colour paintings, cardboard, paper, etc., but the high price of mastic makes its use expensive.

(1) *Solution in Alcohol.*—It yields a quick drying varnish. Water-colour varnishes and paper varnishes are made as follows :—

	Water-Colour.	Paper.
Mastic	5	4
Alcohol	14	14
Venetian Turpentine	2	2½

A colourless varnish for *bookbinders* is made by dissolving 3 parts of mastic in 20 parts of absolute alcohol and adding 1 part of bleached shellac.

As alcohol only dissolves 90 per cent. of mastic, the residue is employed in making mastic varnishes with spirits of turpentine as the solvent. But very often complete solution is effected in alcohol by the addition of a little spirits of turpentine.

(2) *Solution in Spirits of Turpentine.*—This varnish is highly esteemed for pictures. One part of mastic is dissolved in 1½

parts of spirits of turpentine either on the cold or on a water bath. Finally, acetone has been recommended as a solvent, mastic being very soluble in the cold in that reagent.

SANDARACH SPIRIT VARNISH.

Alcohol is the customary solvent for sandarach, but occasionally a mixture of alcohol and spirits of turpentine are used. Sandarach varnish varies from straw yellow to deep yellow according to the colour of the resin Venice turpentine is generally added to produce elasticity. When applied in very thin coats it gives a very beautiful lustre.

Sandarach varnish is used for labels, placards, papier-mâche work, and in general for turned or carved articles, for leather, wood and metals.

The general formula is:—

Sandarach - - - - - - -	5 parts
Venice turpentine - - - -	3 to 5 parts
Alcohol, 95 per cent. - - - - -	15 to 24 parts

However, in the case of varnishes expected to give a thin coating, the amount of alcohol may be as high as 45 parts. This is the case with the varnishes used to fix drawings and water-colours.

When the varnish is to be applied to wood, the 5 parts of Venice turpentine in the above formula may be replaced by 2 parts of elemi or mastic, or by adding to the varnish 5 parts of a solution consisting of a mixture of equal parts of resin and shellac dissolved in 95 per cent. alcohol.

When the varnish is to be applied to metals it is advisable to replace the mastic either by a mixture of mastic shellac and benzoin or by a mixture of mastic and Venice turpentine; in this case instead of alcohol a mixture is employed of equal parts of alcohol and spirits of turpentine.

A flexible varnish may also be prepared by melting 4 parts of sandarach and 2 parts of resin, and dissolving in 8 parts of spirits of turpentine. A solution of india-rubber in coal-tar naphtha is then added.

Again, a varnish for photographic negatives is made by simply dissolving 18 parts of sandarach in 100 parts of alcohol. To prevent dulness in drying, 1 part of spirits of lavender is added to give brilliancy and transparency.

Sandarach Varnish.

1. Paper Varnish.

Australian sandarach	50 lbs.
Venice turpentine	30 ,,
Methylated spirit	18 gals.

2. Paper Varnish, Second Quality.

Australian sandarach	50 lbs.
Burgundy pitch	30 ,,
Methylated spirit	18 gals.

3. Sculptors' Varnish.

Australian sandarach	40 lbs.
Venice turpentine	30 ,,
Methylated spirit	20 gals.

4. Pale Bookbinders' Varnish.

Australian sandarach	50 lbs.
Venice turpentine	35 ,,
Methylated spirit	21 gals.

5. Varnish for Black and White Drawings.

Australian sandarach	20 lbs.
Venice turpentine	20 ,,
Methylated spirit	22 gals.

6. Water-Colour Varnish.

Sandarach	40 lbs.
Venetian turpentine	40 ,,
Methylated spirit	17 gals.

7. Varnish for Silver.

Sandarach	40 lbs.
Venice turpentine	80 ,,
Methylated spirit	23 ,,

Mixed Sandarach Varnishes.

1.	Sandarach	60 lbs.
	Mastic	60 ,,
	Venice turpentine	30 ,,
	Methylated spirit	29 gals.

2.	Sandarach	30 lbs.
	Rosin	20 ,,
	Burgundy pitch	10 ,,
	Methylated spirit	12 gals.

3.	Sandarach	30 lbs.
	Mastic	20 ,,
	Rosin	20 ,,
	Venice turpentine	10 ,,
	Methylated spirit	13 gals.

4.	Sandarach	30 lbs.
	Rosin	20 ,,
	Elemi	10 ,,
	Methylated spirit	12 gals.

Rosin Varnish.

Rosin varnishes are cheap but not durable. They are improved by the addition of a little Venice turpentine, sandarach or mastic, which imparts a greater tenacity, thus causing the varnish to adhere better to the object to which it is applied, but then the varnish is not by any means cheap.

A varnish for toys and common articles is made by simply dissolving French or American rosin in methylated spirits. The process is similar to that given for shellac varnish. The colour varies with that of the resin used. Generally Venetian turpentine is added.

The general formula is:—

Rosin	20 parts
Alcohol, 95 per cent.	12·7 to 14·5 parts

There may be added:—

Venice turpentine	3·6 parts

If spirits of turpentine be the solvent used, as rosin is very soluble therein, the operation may be done in the cold, but heat accelerates solution.

If with a view of improving the varnish, Venice turpentine, sandarach or mastic be added to the resin, they are all cautiously melted together, and when the mixture is homogeneous the spirits of turpentine is added after withdrawing the vessel from the fire.

The usual formula is:—

Rosin	20 parts
Spirits of turpentine	12 ,,
Venice turpentine Sandarach or mastic	6 to 40 ,,

But it is then necessary to increase the proportion of spirits of turpentine, allowing 3 to 6 parts of spirits for each part of substance other than resin.

Sometimes resin varnish made with turpentine if kept in a rather warm place turns turbid. It may be clarified by reheating and adding 5 per cent. of spirits of turpentine.

If it is desired to obtain a varnish which will dry more slowly than the alcoholic varnish, the resin to which Venetian turpentine has been added is sometimes dissolved in a mixture consisting of 3 parts of 95 per cent. alcohol and 1 part of spirits of turpentine.

Finally, low-class varnishes are made by dissolving resin in coal-tar or petroleum naphtha.

Asphaltum Varnishes.

This varnish is prepared from ether, bitumen of Judæa, the natural asphaltum, or from coal tar or other pitches. When natural asphaltum is used, it is advisable to melt it previously, especially when used in conjunction with amber or copal. The solvents used are spirits of turpentine and coal-tar naphtha. A copper vessel is used.

The general formula is:—

Coal tar or asphaltum - - - - -	- 1 part
Coal tar naphtha - - - - -	- 1 to $1\frac{1}{10}$ parts

The varnish thus obtained is brilliant but brittle, especially when wanted for leather. Elasticity may be imparted by adding a little elemi and copaiba balsam. It has also been recommended to add a small quantity of a solution of india-rubber in coal-tar naphtha. Finally, by adding castor oil a slower drying varnish is obtained, but more flexible and adhering better to the glass when tested.

If spirits of turpentine be used to dissolve the asphaltum, etc., the following is the general formula:—

Coal tar or asphaltum - - - - -	- 2 parts
Spirits of turpentine - - - - -	- $3\frac{1}{2}$,,

It is advisable before dissolving to add spirits of turpentine in the proportion of 15 per cent. of the bitumen used to the vessel in which the latter is melted so as to hasten fusion.

Cheaper varnishes are made by dissolving 2 parts of coal tar and 1 part of Stockholm tar in 4 parts of coal-tar naphtha. Asphaltum may be replaced by resin or deep-coloured fused amber, without however going beyond half of the quantity of bitumen usually employed.

India-Rubber Varnishes.

These varnishes are generally employed as waterproof varnishes, *i.e.*, to render the object to which they are applied impermeable to water, but they are more often used in the form of oil varnishes by the addition of linseed oil and ordinary oil varnish.

If india-rubber varnishes have the advantage of great elasticity, they have on the other hand the great disadvantage of drying slowly.

The principal solvents are benzol and spirits of turpentine, in which the rubber swells and becomes transparent without however actually dissolving. Attempts have been made to use caoutchouc oil as a solvent, but it has not yet been adopted on the large scale.

This varnish contains but very little solid matter, because a very large proportion of solvent must be used to obtain a homogeneous product, and there is no advantage in distilling off a portion of the solvent because the thickened residual varnish would dry too slowly.

It is important to use thoroughly dry rubber. Before treatment the rubber is cut into thin strips and dried for several days at a temperature of 40° to 50° C. The rubber dried in this manner is more easily acted on by solvents, and the resulting solution is of better quality.

If benzol be the solvent used, 1 part of caoutchouc is heated on the water bath with 8 parts of benzol.

With spirits of turpentine as solvent, 1 part of the rubber is covered with 4 parts of the solvent, and left to stand for forty-eight hours; there is afterwards added 4 parts of spirits of turpentine, and complete solution effected by heating on the water bath.

Finally, colourless solutions may be obtained by first treating the rubber with carbon disulphide thus: The rubber is covered with twice its weight of carbon disulphide, which causes it to swell, and contact is continued until a homogeneous jelly is obtained, which on the addition of benzol dissolves completely; the carbon disulphide may be distilled off on the water bath.

A colourless solution is thus obtained and in less time than with benzol alone.

GUTTA-PERCHA VARNISH.

This varnish—like that of india-rubber—is principally used in waterproofing paper or skins.

The gutta-percha ought to be dried with care.

Map Varnish.—A gutta-percha varnish is the one particularly selected for coating maps and manuscripts. Ten parts of gutta-percha cut into thin strips and well dried are placed in a vessel containing 40 parts of bisulphide of carbon and 20 parts of eucalyptus oil ; the whole is allowed to digest and agitated from time to time until the whole is dissolved, after which it is set aside to clarify and the clear liquid decanted. If this solution be too thick it is thinned down with benzol. Objects before being covered with this varnish ought to be very dry. It is very durable, and its surface can be written upon, which is an advantage.

But generally, and for leather in particular, linseed oil is the only solvent used, or a mixture of linseed oil and benzol.

COLLODION VARNISH.

Collodion or pyroxyline varnish is very extensively employed and for very varied purposes. The more so as a great flexibility may be imparted to it by the addition of 2 parts of castor oil. The solvent usually employed is the mixture of alcohol and ether : 1 part of collodion in 3 parts of alcohol and 18 parts of ether.

Photographic Varnish.—For photographic purposes 1 part of collodion is dissolved in 1 part of alcohol and 1½ parts of ether.

The addition of 25 per cent. of bisulphide of carbon or benzol produces products of different colour and brilliancy.

The collodion ought to be perfectly dry, and to effect this it is placed underneath a bell-jar over sulphuric acid for about forty-eight hours.

Camphor to the extent of even 50 per cent. is often added to the collodion.

The mixture of alcohol and ether is often replaced by other liquids, such as acetone, wood-spirit, acetate of amyl.

Acetone yields opaque varnishes analogous to that obtained by the addition of camphor; wood-spirit has the same effect when it contains acetone in appreciable quantity.

When acetate of amyl is used—in which collodion dissolves very easily—a bright, colourless, transparent varnish is obtained; moreover, the coating is much more malleable than that yielded by the ordinary solution. This is how the varnish so widely known under the name of "zapon" is made. "Zapon" spreads evenly over glass and gives a more homogeneous coating than other collodion varnishes; on the other hand, it dries more slowly.

Valenta proceeds as follows: 150 parts of gun cotton are dissolved in 1000 parts of acetone, and there is then added 2000 parts of acetate of amyl and 2000 parts of benzene. The solution is clarified by decantation and filtration. This varnish yields a supple adherent coating which does not shell off.

Finally, it has been proposed to impart to wood a coating of collodion varnish, *i.e.*, to prime it with collodion and then to cover this coat which has filled up the pores of the wood with different varnishes, which then become very lustrous—thanks to the layer of collodion—without either requiring rubbing or polishing.

It is also with collodion that the numerous compositions known as "non-oxidisable bronzes" are prepared. Thus: 5 parts of collodion are dissolved in 95 parts of acetyl acetic ether, and 25 parts of powdered bronze are then ground up in this solution; then, to hinder the decomposition of the nitrated product, 25 parts of gum dammar are dissolved in 75 parts of acetate of amyl.

HALLE'S COLLODION VARNISH.

Amyl acetate	4 galls.
Benzol	4 ,,
Pyroxyline	2½ lbs.

Ferner dissolves nitro-cellulose in a mixture of amyl acetate and amyl alcohol.

	I. Parts.	II. Parts.
Nitro-cellulose	100	100
Amyl acetate	150	400
Amyl alcohol	150	400

CELLULOID VARNISHES.

Celluloid varnishes, the introduction of which is comparatively recent, are colourless, very brilliant and adherent. They do not crack or rupture, and they may be easily coloured. They possess therefore almost all the qualities desired in a varnish, the more so as they may be rendered very supple by the addition of an oil such as castor oil.

Celluloid varnishes have a greater resistance than collodion varnishes, even a thick coating does not shell off, whilst collodion varnishes must be applied in thin layers; moreover, there is perfect brilliancy and adherence with coats of celluloid varnishes about ⅛ of an inch thick.

This varnish is therefore employed very advantageously for paper and all the varied objects in which durability and lustre is desired.

Celluloid should be perfectly dry before being treated. This is effected in the same way as with collodion, by placing it in a bell-jar for a certain time over sulphuric acid.

The same solvents may be used for celluloid as for collodion—that is to say, a mixture of alcohol and ether, or pure acetone, or acetone and amylic alcohol—so that the varnish may dry more slowly, or acetate of amyl, which gives a varnish analogous to "zapon" varnish.

If a mixture of acetate of amyl and acetone be used photographers' crystal varnish is obtained, which is applied in the cold, giving a perfectly transparent coat. By adding a small quantity of gum resin a layer which has not such a polish is obtained, and thus better able to bear "retouching".

When it is desired to apply celluloid varnish upon a coating of collodion, a few drops of water must be added to the varnish, so as to prevent it from dissolving the layer of collodion.

Celluloid varnishes are generally thinned down with wood-spirit.

Finally, aniline dyes dissolved in very strong alcohol are used to colour celluloid varnishes, and the necessary quantity is carefully mixed with the varnish whilst stirring.

	I. Parts.	II. Parts.	III. Parts.	IV. Parts.	V. Parts.
Celluloid	5	10	—	5	5
Amyl acetate	16	30	—	50	25
Acetone	16	30	5	—	25
Ether (sulp. meth.)	16	30	—	—	—
Camphor	—	4	5	—	—
Alcohol	—	—	50	—	—

ALUMINA SOAP VARNISH.

When an ordinary soap is precipitated by alum or sulphate of alumina the precipitate formed by the combination of alumina with the fatty acids dissolves very easily in spirits of turpentine. This solution is used in the form of an ordinary spirit varnish, which, although it does not present all the brilliancy of dammar varnish, has the advantage of not decomposing at even a high temperature and does not "blow".

RESINATE AND COLOURED RESINATE VARNISHES.

Resinates may be substituted for resins in varnish-making. Coloured resinates are generally used. Some—like resinate of copper,[1] which is of a beautiful emerald-green tint—are coloured naturally. The desired shade is imparted to colourless resinates by aniline dyes. Coloured resinates in the dry state have a fresh appearance, and those used in varnish-

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[1] Made by neutralising a solution of one equivalent (106 parts) of dry sodium carbonate with rosin, and precipitating with 249·5 parts of sulphate of copper free from iron.

making are insoluble in water; weak acids and alkalies have no action on these resinates, but, on the other hand, they dissolve very easily in alcohol, spirits of turpentine, benzol, ether and chloroform as well as in melted wax, resins, oils and boiled linseed oil. This facility of solution and their beautiful colour cause them to be greatly used, and they have been advantageously applied upon metal, wood, paper, skin, glass, wax, linoleum and cloth.

Preparation of Coloured Resinates.—A rosin soap is made by heating 100 parts of pale rosin with 33 parts of soda crystals in 1000 parts of water, and adding to the solution cooled to 50° C. a solution of colouring matter. A solution of a metallic salt is then added, such as the chloride of magnesia, and the solution filtered from the insoluble coloured resinate, which is well washed and dried at a very gentle heat. The dried product constitutes in reality a true coloured lake.

When spirit varnishes are made from resinates Lohmann's method may be followed. Thirty parts of well-dried resinate of magnesia, dyed to the requisite tint, are dissolved in 80 parts of benzol and 20 parts of chloroform; there is afterwards added 150 parts of a pale solution of 1½ per cent. of caoutchouc in sulphide of carbon and benzol.

Coloured resinates have also been prepared from dammar; to accomplish this a solution of caustic potash is added to a solution of dammar in spirits of turpentine. The whole is boiled to volatilise the spirit; the residue dissolves completely in water, and yields on the addition of a solution of metallic salts precipitates which dissolve partially in ether. These solutions have been employed as varnishes.

ANALYSIS AND VALUATION OF SPIRIT VARNISHES.

Technical Valuation.—In the valuation of spirit varnishes two great points must be carefully attended to, *viz.*, the physical properties and the chemical composition. In examining the physical properties, the drying properties have to be determined. It is a *sine quâ non* that the varnish should not dry tacky. If it does it should be rejected. The quicker the varnish dries, other things being equal, the more valuable and economical it is, as less time is lost by the workmen in waiting for a previous coat to dry before applying the next. Body, brilliancy, transparency, colour, elasticity, the hardness, as well as the capacity of the varnish to withstand wear and tear; the action of the weather and the alternations of temperature produced by day and night, summer and winter, etc., have each and all to be studied before a definite conclusion can be arrived at, having regard to the special object in view; the climate in which the varnish is to be used and the temperature or temperatures to which the object may be exposed during the whole course of its existence. A varnish, therefore, which may be very valuable for one purpose may be worthless, or worse than worthless, for another.

The valuation of varnish, therefore, depends almost entirely upon the experience of the expert. Through the long handling of varnish he can distinguish almost intuitively good varnish from bad. He calls to his aid the senses of sight, touch and smell. The presence of Manilla copal in spirit varnish may always be detected by its peculiar aromatic odour. The perception of this smell in a varnish at once recalls to the expert that Manilla has a tendency to string on application. He therefore at once presses a drop between the finger and thumb, and if Manilla be present after the spirit has evaporated to a certain extent the resin may be drawn out into long strings. It is, however, to be observed that certain other resins, *e.g.*, common rosin, are often added to Manilla to counteract this tendency.

Although the valuation of varnish is mainly technical, some processes to be indicated hereafter afford rigorously comparative results.

Chemical Analysis: Determination of Solvent.—From the chemical point of view, the analysis of varnish consists in the determination of solvent, or solvents, and resin, or resins, and their relative proportion, and should be so expressed as to constitute a formula from which the intelligent varnish-maker may match any sample of varnish submitted to him, and thus enable him to quote a price or submit a tender for the supply of the same in large or small quantities as the case may be.

The *smell* of the varnish gives a good indication of the solvent used, which may further be easily recognised by the determination of its *boiling-point.*

Determination of Boiling-point.—This may be done by heating a certain quantity of varnish in a small flask, and collecting the vapours either in a condenser or in a flask containing water. A thermometer placed in the vapour indicates the boiling-point, and consequently the nature of the solvent used.

In cases where the analyst has only a very small quantity of varnish at his disposal—sometimes no more than an ounce, and where he may have to make numerous samples for comparative trial against the sample submitted—every drop of that sample is precious. Now, in some cases, if the varnish were heated to the boiling-point of the solvent used, say, in the case of a metallic resinate such as resinate of copper, the latter would be liable to become reduced and change from green to a ruby-red, and if the whole of the sample of varnish were thus used the analyst would be left in the lurch.

In such and similar cases only small quantities of the varnish should be distilled, but, wherever practicable, enough should be taken to yield a distillate sufficiently large for its density to be taken without inconvenience.

But the boiling-point may be determined sufficiently accurately for technical purposes without distillation, and by the use of only a single drop of liquid. The liquid is placed in a glass tube, drawn out and sealed at the bottom. A capillary tube sealed up a little above its lower open extremity is introduced, and the whole is attached to a thermometer, and treated as in the determination of a melting-point.

Before the liquid reaches the boiling-point, single air bubbles proceed from the small volume of air in the capillary below the closed constriction; these become gradually more numerous till an uninterrupted thread of small bells of vapour is established. At this moment the thermometer shows the exact boiling-point of the liquid. The operation should be repeated several times and the mean of the observations taken. The capillary prevents boiling and must be renewed for each experiment.

Should the solvent consist of several volatile liquids, the operation goes on in the same manner according to the different boiling-points of the different liquids; moreover, these may be separated the one from the other by fractional distillation and their relative proportions ascertained.

Resin determination.—The nature of the resin or resins forming the base of the varnish is determined in the residue either after evaporation of the solvent or after distillation thereof.

Its appearance and physical properties will afford valuable indications, provided always, on the one hand, that all the solvent has been got rid of by evaporation or distillation, and, on the other hand, that these processes have not been carried so far as to start the destructive distillation of the resin or resins, and, finally, that no chemical change has been produced causing either oxidation or reduction. Otherwise the characteristics of the original resin or resins may be so altered in regard to colour, hardness, etc., as to prevent recognition.

The solubility of this residue in the following different solvents is then carefully studied, *viz.*, ether, alcohol, carbon disulphide, acetone, methyl alcohol, amylic alcohol, petroleum, ether and benzene, chloroform and spirits of turpentine.

The same solvents are used in the case of a mixture of resins, with the addition of glacial acetic acid. This latter solvent is used to identify mastic, which is the only resin insoluble in that reagent. The solubility of the different resins in the solvents indicated is shown in the following table:—

ACTION OF SOLVENTS UPON THE PRINCIPAL RESINS (LIVACHE).

Resins.	Ether.	Alcohol.	Benzol.	Acetone.	Methylic Alcohol.	Amylic Alcohol.	Petroleum Ether.	Bisulphide of Carbon.	Chloroform.	Spirits of Turpentine.
AMBER	Consists of 88 to 90 per cent. of a resin completely insoluble in all solvents, and 12 per cent. of a resin which dissolves more or less completely in alcohol ether, benzol, etc.									
COPALS :—										
Hard: Zanzibar	Insoluble	Insoluble	partially dissolve	Insoluble	Insoluble	partially soluble	Insoluble or almost insoluble	Insoluble	partially soluble	The greater number are insoluble, but all are at least partially soluble in the oxidised essence; complete solution is effected by repeated treatment with oxidised spirits
Hard: Mozambique	do.	do.		do.	do.			do.		
Hard: Madagascar	do.	do.		do.	do.			do.		
Medium Hard: Sierra Leone	do.	soluble 10%.		do.	do.			partially soluble		
Medium Hard: Angola Red	partially soluble or yielding a jelly which dissolves on the addition of hot alcohol			partially soluble	do.					
Medium Hard: Benguela		partly sol.								
Medium Hard: Angola Bastard		almost completely soluble			partially soluble					
Medium Hard: Pebble Copal										
Medium Hard: Akra										
Medium Hard: Benin										
Medium Hard: Loango										
Medium Hard: Gaboon										
Medium Hard: Congo										
Medium Hard: Angola White										
Soft—South American	soluble	almost insol.								
DAMMAR	partly sol.	only partially soluble in the cold, soluble with the aid of heat	soluble	insoluble	partially soluble	partially soluble	soluble	partly sol.	soluble	soluble
SHELLAC	insoluble	soluble	almost insol.	do.	do.	do.	partially sol.	do.	insoluble	partially sol.
MASTIC	soluble	soluble in absolute, partly soluble in dilute, alcohol	soluble	partly sol.	do.	soluble	almost insol.	incomp. sol.	soluble	soluble in the hot
SANDARACH	soluble	soluble	almost insol.	almost insoluble in cold, soluble in hot	do.	soluble	part. sol.	do.	insoluble	incomp. sol.
ROSIN	partly sol.	soluble	soluble	soluble	soluble	soluble	soluble	soluble	soluble	partly sol.
ELEMI	do.	sol. in hot	do.	do.	do.	do.	do.	do.	do.	soluble
VENICE TURPENTINE	do.	soluble	do.	do.	do.	do.	do.	do.	do.	part. sol.
BENZOIN	do.	do.	insoluble	insoluble	part. soluble	part. sol.	part. sol.	insoluble	part. sol.	do.
ASPHALTUM	do.	only very par.	soluble	do.	insoluble	do.	insoluble	do.	do.	do.

When the resin is not completely soluble in alcohol the solution is thrown upon a tared filter, washed with boiling alcohol, and the residue dried at 100°. With residues insoluble or difficultly soluble in alcohol, spirits of turpentine is used as the solvent. The solution is filtered and the insoluble residue weighed; the solvent is distilled in a current of steam, and the residue treated as in the preceding paragraph.

In this case the acid figure and the Kottstorfer figure are always a little low, for a portion of the free acids are carried away in the distillation process. The quantity of iodine absorbed is on the contrary a little too high.

The solubilities of the different resins entering into the composition of varnish may be so identical or so badly defined that no sound inference can be drawn by which they can be differentiated the one from the other. In such cases what is known as the *constants* of the resin have to be determined.

These are:—

(1) *The acid value* (free acid), that is to say, the number of milligrammes of caustic potash required to exactly neutralise 1 gramme of resin in alcoholic solution. The acid figure is determined by heating 1 gramme of the finely pulverised resin with 95 per cent. alcohol in a glass flask attached to a reflux condenser until complete solution is effected. Two c.c. of a dilute solution of phenol phthalein are added, and the solution titrated with standard alkali.

(2) *The Köttstorfer value* (corresponding to the total acids after complete saponification), that is to say, the number of milligrammes of caustic potash required to saturate 1 gramme of resin in alcoholic solution in presence of an excess of potash. The Köttstorfer value is determined by heating 1 gramme of the resin with 25 cubic centimetres of standardised alcoholic potash from five to fifteen minutes in a flask attached to a reflux condenser; the whole is then diluted with 100 cubic centimetres of alcohol, and again brought to the boil, and the uncombined alkali is titrated with standard hydrochloric acid in presence of phenol phthalein.

Chemical Constants of Resins.

Resin.	Variety.	Acid Value.	Saponification Value.	Iodine Absorption.		Bromine Absorption.
				With Residue.	Without Residue.	
Amber		15·4	86·8	—	—	—
		—	160·7	—	—	53·53
		—	145·0	—	—	—
		—	144·6	—	—	—
	Fused	0·0	38·9	—	—	—
	,,	0·0	33·2	—	—	—
	,,	0·0	36·0	4·8	3·3	—
Anime	Demerara Rough	26·6	73·6	—	—	—
	Zanzibar Fine	18·2	73·6	—	—	—
		25·2	87·5	—	—	—
		—	95·4	—	—	60·22
Asphaltum		—	4·7	22·2	3·5	—
Benzoin		136·3	164·7	—	57·4	—
		134·1	164·5	—	56·6	—
Copal	Manilla Soft	131·6	184·0	—	—	—
	,, Borneo	141·4	176·7	—	—	—
	,, Singapore	128·8	194·1	—	—	—
	Sierra Leone Clean	84·0	129·0	—	—	—
	,, Rough	72·8	138·5	—	—	—
	Akra Rough	46·2	131·6	—	—	—
	Angola White Rough	57·4	133·0	—	—	—
	,,	—	130·9	—	—	—
	,,	—	132·2	—	—	—
	,,	—	129·7	—	—	—
	,, Fused	93·6	118·8	—	—	—
	,,	93·4	117·8	—	—	—
	,, Red Clean Fine	60·2	136·2	—	—	—
	,, ,,	—	148·0	—	—	—
	,, ,,	—	146·4	—	—	—
	,, ,, Fused	30·5	110·7			
	,, ,, ,,	30·0	109·8	34·8	22·3	—
	Unknown	57·4	122·2			
		—	123·9	—	—	83·93

	Reduced to ¼ by boiling	—	128·9	—	—	84·52
	Boiled	—	111·1	—	—	—
	Zanzibar	—	92·1	—	—	—
	,,	—	89·6	—	—	—
	,, Fused	—	36·8	12·6	7·6	—
	,, ,,	—	34·6			—
Dammar	Batavian	22·4	36·4	—	—	—
		26·6	31·1	—	—	—
		—	52·3	—	—	117·94
		33·0	47·1	63·6	61·1	—
		30·6	46·5	63·5	60·5	—
Dragon's Blood		—	—	72·4	55·5	—
Elemi		15·7	28·6	—	—	—
		—	32·9	—	—	122·23
		22·3	25·1	—	85·1	—
		22·0	24·0	—	80·9	—
Gamboge		81·2	not determinable	—	—	—
		79·4	—	—	—	—
Kauri	Medium	63·0	99·3	70·9	70·0	—
	Fine	51·8	77·1	—	—	—
		—	128·8	—	—	108·22
Mastic		64·5	93·8	61·1	53·2	—
		63·6	92·3	61·2	53·1	—
Rosin	Refined	179·2	187·1	—	—	—
	,,	177·8	195·7	—	—	—
	Ordinary	169·4	176·1	—	—	—
	,,	166·6	190·1	—	—	—
	Window Glass	159·1	170·4	—	—	—
	E.	168·5	189·9	—	—	—
	A. Black	155·7	195·1	—	—	—
		146·5	168·2	—	116·8	—
	French	146·0	167·1	—	115·7	—
	Refined	—	181·0	—	—	112·7
Sandarach		141·4	174·4	—	66·8	—
,,		138·7	170·0	—	63·8	—
Shellac	Liver	65·1	213·3	—	8·3	6·0
,,	Orange	60·0	211·6	—	—	—
Storax		129·5	195·8	64·7	58·6	—
Turpentine	Venice	70·1	102·6	—	145·3	—

(3) The Ester value is got by deducting the acid value from the Köttstorfer.

(4) *The Hubhl or iodine absorption figure*, that is to say, the quantity of iodine fixed or absorbed by 100 parts of resin in alcoholic solution.

Process.—One gramme of the resin is dissolved in 50 c.c. of hot alcohol and allowed to cool, and 50 c.c. of a standard solution of iodine added. If the iodine be decolorised, a fresh quantity of the iodine solution is added until the mixture becomes of a permanent red-brown colour. The whole is allowed to stand for twenty-four hours, after which the iodine in excess is estimated in the following manner. A solution of potassium iodide is added until on the addition of water or a salt of mercury no further precipitate of iodine or mercuric iodide is obtained. Too great an excess must be avoided. The liquid is then diluted with 5 to 6 volumes of water. The resin separates in flocks. Starch paste is added and the solution titrated with hyposulphite of soda. The end of the operation is indicated by the decoloration of the iodide of starch. Analytical results for each resin are given in the preceding table.

QUALITATIVE ANALYSIS OF RESINS—HIRSCHSOHN'S SCHEME.

The following tables are summarised from the elaborate scheme of Hirschsohn's. The reagents to be used are :—

1. Sulphuric acid H_2SO_4, specific gravity 1·820.
2. Alcoholic hydrochloric acid (95 per cent. alcohol saturated with *dry* HCl).
3. Bromine solution, 1 in 20 of chloroform.
4. Saturated solution of bleaching powder in distilled water at ordinary temperature.
5. Solution of 1 part of ferric chloride Fe_2Cl_6 in 10 of 95 per cent. alcohol.
6. Saturated solution of *neutral* lead acetate $Pb(A_2)$ in 95 per cent. alcohol.
7. Solution of ammonia, specific gravity ·980.

8. Solution of pure Na_2CO_3 in distilled water.

9. Frohdes test, 1 centigramme sodium molybdate in 1 c.c. sulphuric acid.

10. Impure chloral hydrate containing alcoholate.

11. Saturated solution of iodine in petroleum spirit boiling at 60° C.

12. Petroleum spirit boiling between 35° and 40° C.

GROUP I.—COMPLETELY SOLUBLE IN CHLOROFORM.

Sub-Group I.—*Completely Soluble in Ether.*

Experiment.	Observation.	Inference.
Add alcohol to ethereal solution.	A.—Turbidity (if none pass on to B).	
Add Fe_2Cl_6 to alcoholic solution.	I. Turbidity disappears on boiling.	
Treat with chloral.	Violet coloration.	Canada balsam.
	II. No turbidity.	
	(*a*) The resin is liquid.	
Treat with petrol. spirit.	Dissolves.	
Add bromine test to chloroform solution.	1. Yellowish, passing through violet to blue coloration.	Maranham copaiba.
	2. No colour.	Para copaiba.
	(*b*) The resin is solid.	
Treat with petrol. spirit.	Dissolves partially.	
Treat with iodine solution.	Red violet coloration.	Mastic (ordinary).
Add alcohol to ethereal solution.	B.—Clear solution.	
Treat resin with alcohol.	I. Dissolves completely (if none pass on to II.).	
Add Fe_2Cl_6 to alcoholic solution.	1. Blue coloration.	
[Add $Pb(A)_2$ to alcoholic solution.]	(*a*) Precipitate—H_2SO_4 dissolves resin with a cherry red coloration.	Guaiacum.
	(*b*) No precipitate—H_2SO_4 solution yellowish brown.	Carana resin. (Aceyta Americana.)
	2. Brownish or greenish coloration.	
[Do.]	(*a*) Precipitate (if none pass on to (*c*)) not dissolved on boiling (if it dissolves pass on to (*b*)).	
[Treat resin with solution of Na_2CO_3.]	(1) Dissolves partially at ordinary temperature.	
[Dissolve resin in petrol. spirit, evaporate solvent, test residue with chloral.]	Gradually becomes reddish violet with blue streaks.	Coniferous resins.
	(2) Little or no action.	
[Do.]	i. Colourless. Chloral test faint green.	Bombay mastic.
[Do.]	ii. Dark brown. Chloral test brown.	Main resin.
[Do.]	iii. Yellow brown. Chloral test dull violet.	Carana resin.
[Do.]	iv. Yellow brown. Chloral test and bromine solution a magnificent violet.	Carana, [illegible]

Experiment.	Observation.	Inference.
[Add $Pb(A)_2$ to alcoholic solution.]	(*b*) Precipitate dissolves on boiling.	
[Bromine solution.]	(*a*) Red coloration.	Peruvian guaiacum.
	(*b*) No coloration.	Alexandria mastic.
[Add $Pb(A)_2$ to alcoholic solution.]	No precipitate. Ammonia gives turbidity.	Dragon's blood.
Treat resin with alcohol.	II. Imperfect solution.	
[Add $Pb(A)_2$ to alcoholic solution.]	(1) Turbidity which disappears on warming.	Brazilian copaiba.
	(2) No precipitate. Resin crystalline. Does not dissolve on boiling Na_2CO_3.	
[Bromine solution.]	(*a*) Gradually colours green.	
[Alcoholic HCl.]	Colours violet, blue or brown.	Elemi.
	(*b*) Colours violet.	Elemi.
	(*c*) No colour.	Elemi (Amyris Elemifera).

Group I., Sub-Group II.—*Imperfectly Soluble in Ether.*

Experiment.	Observation.	Inference.
Treat resin with alcohol.	A.—Dissolves perfectly.	
Treat evaporation residue of a petrol. spirit extract with H_2SO_4.	I. Cherry red coloration. No cinnamic acid.	Siam Benzoin.
	II. No coloration or faint brown, contains cinnamic acid.	Sumatra benzoin. Tolu balsam.
	III. Yellow brown passing to violet.	Black Peru balsam.
	B.—Incomplete solution.	
[Add Fe_2Cl_6 to alcoholic solution.]	I. Precipitate does not redissolve on boiling and is insoluble in ether.	Brazilian copal.
	II. No turbidity or slight disappearing on boiling.	
[Add alcohol to ethereal solution of resin.]	1. Turbidity.	
Alcoholic HCl.	(*a*) Brownish coloration.	
Treat with chloral evaporation residue of petrol. spirit extract.	(1) Greenish coloration.	Dammar.
	(*b*) Brick red.	
[Do.]	(2) Carmine red to violet.	White Peru balsam.
	2. Clear solution.	
Add ammonia to alcoholic solution.	(*a*) Clear solution.	
Add bromine to alcoholic solution.	(1) Blue coloration.	Ceradia resin.
	(*b*) Turbid solution.	
[Do., do.]	(2) Greenish coloration.	Mecca balsam.

GROUP II.—IMPERFECTLY SOLUBLE OR INSOLUBLE IN CHLOROFORM.

Sub-Group I.—*Completely Soluble in Ether.*

Experiment.	Observation	Inference
Treat resin with ether.	1. A clear solution. Red solution.	Dragon's blood, fro Pterocarpus Draco.
Add ammonia to alcoholic solution.	2. Solution yellow or colourless.	
To alcoholic solution add $Pb(A)_2$.	(*a*) No precipitate.	Podocarpus resin.
	(*b*) A precipitate permanent on boiling.	Sandarach.

Sub-Group II.—*Imperfectly Soluble in Ether.*

Experiment.	Observation.	Inference.
Treat ether solution with alcohol.	A.—Turbidity.	
Add ammonia to alcoholic solution.	I. A clear mixture.	
	1. The mixture is yellow.	
Dissolve resin H_2SO_4.	Solution yellowish brown, and gives clear violet mixture with alcohol.	Eryops resin.
	2. The mixture is carmine red.	Sonora lac.
	II. A turbid mixture.	
Fe_2Cl_6.	1. Green colour. The drug contains cinnamic acid.	
$Pb(A)_2$.	A precipitate.	Liquid storax.
	2. Brownish or not at all.	
	(*a*) The drug contains cinnamic acid.	
$Pb(A)_2$.	(1) No precipitate.	Liquidambar balsam.
	(*b*) The drug does not contain cinnamic acid.	Euphorbia Tirucalli resin.
$Pb(A)_2$.	(2) A precipitate.	
Treat ether solution of resin with alcohol.	B.—No Turbidity.	
Treat resin with alcohol.	I. Complete solution.	
Fe_2Cl_6.	Dark brown or black.	
	1. Alcohol solution red.	
$Pb(A)_2$.	(*a*) No precipitate.	Xanthorrhoea Quadrangularis resin.
Chloroform extract.	(1) Colourless.	
$Pb(A)_2$.	(*b*) Turbidity.	
Chloroform extract.	(2) Yellow.	Xanthorrhoea Arborea.
	2. Alcohol solution yellow	
$Pb(A)_2$	A precipitate.	Yellow xanthorrhoea.
	II. Incomplete solution.	
Treat alcoholic solution of resin with ammonia.	1. Clear Mixture.	
	(1) Violet.	
$Pb(A)_2$.	Violet precipitate.	Shellac.
	(2) Yellow or colourless solution.	

Experiment.	Observation.	Inference.
Add Fe_2Cl_6 to alcoholic extract.	1. Black coloration.	
$Pb(A)_2$.	No precipitate.	Gamboge.
Do.	2. Precipitate which neither dissolves on heating nor soluble in ether.	
$Pb(A)_2$.	A precipitate.	
Treat resin with mixture of alcohol and ether.	(*a*) Dissolves easily.	
Treat chloroform solution of resin with bromine.	i. Precipitates resin completely.	Australian copal.
	ii. No precipitate.	Manilla copal.
	(*b*) Dissolves imperfectly.	African copal.
Treat alcoholic solution of resin with ammonia.	B.—Turbidity.	
Fe_2Cl_6.	(*a*) Precipitate insoluble on boiling and in hot ether.	Borneo copal.
	(*b*) No precipitate.	
Treat resin with mixture of alcohol and ether.	(1) Dissolves completely.	
Treat petroleum spirit extract with chloral.	Blue to blue violet coloration.	Balsam of *Liquidambar* Straciflua.
	(2) Dissolves incompletely.	
	I. The resin contains sulphur.	
Subject resin to dry distillation.	i. Yields umbelliferone.	
Treat petrol. spirit extract with HCl.	(*a*) Orange coloration.	
Do. Chloral hydrate.	Green coloration.	Persian Sagapenum.
Do. HCl.	(*b*) Blue violet.	
Do. Chloral hydrate.	Rose colour to raspberry red and violet.	Levant Sagapenum.
Do. HCl.	(*c*) No coloration.	
Treat resin with H_2SO_4.	Dissolves with yellow-brown colour and blue fluorescence.	
Treat resin with HNO_3.	Malachite green.	Ordinary asafœtida.
	ii. Yields no umbelliferone.	
Treat resin with Na_2CO_3.	(*a*) Bright brown extract on which neither $Pb(A)_2$ nor $(A)_2$ have any action.	Asafœtida from ferula alliacea.
	(*b*) Emulsion which cannot be filtered.	
$Pb(A)_2$.	(1) No precipitate. Iodine no action.	Indian bdellium.
	(2) Precipitate immediate or in short time.	
	Redissolves on heating.	
	Iodine no change.	African bdellium.
	II. The resin contains no sulphur.	
Subject resin to dry distillation.	(1) Yields umbelliferone.	
Treat petroleum spirit extract with HCl.	Is coloured.	
	(*a*) Orange.	
Do. Chloral.	Green.	Persian galbanum.
Do. HCl.	(*b*) Violet red.	Comparatively fresh commercial (Levant) galbanum.
Do. Chloral.	Green.	
Do. HCl.	(*c*) Violet blue.	

Experiment.	Observation	Inference.
Treat petroleum spirit extract with chloral.	Carmine red.	Levant galbanum, older
Do. HCl.	(*d*) No colour.	
Do. chloral.	Light brown.	African ammoniacum.
	(2) Yields no umbelliferone.	
Treat resin with solution of bleaching powder.	(*a*) Orange yellow coloration.	Persian ammoniacum.
	(*b*) No coloration.	
$Pb(A)_2$.	1. No precipitate.	
Iodine.	i. Change.	
Chloral.	Greenish.	Olibanum.
Iodine.	ii. Not altered.	
Chloral.	No coloration.	Indian myrrh.
$Pb(A)_2$.	2. Precipitate.	
Bromine.	(*a*) Violet red coloration.	
Chloral.	Violet.	Ordinary myrrh.
Bromine.	(*b*) No coloration or only yellowish.	
Fe_2Cl_6.	(1) Green.	Opoponax.
Do.	(2) Brownish.	Euphorbium.

SECTION VII.

DRYING OILS, OIL CRUSHING, REFINING AND BOILING, INCLUDING MANUFACTURE OF OIL FOR (*a*) RUBBER SUBSTITUTE, (*b*) LINOLEUM, (*c*) PRINTERS' INK.

Drying Oils.

This name is given to those vegetable oils which possess the property of gradually absorbing oxygen from the air at the ordinary temperatures, so as to produce, as a final result, solid elastic substances.

The non-drying oils, on the other hand, though they likewise absorb oxygen, do not produce a solid, elastic substance, provided that they have not been subjected to an elevation of temperature. Even after a very long time they simply thicken, but do not solidify. It has been shown by Livache that if the absorption of oxygen was accompanied by a sufficient elevation of temperature the non-drying oils were transformed into a product identical with that yielded by the drying oils (Livache, *Comptes Rendus de l'Académie de Sciences*, 1895).

When a drying oil is transformed into a solid elastic body it is said to "dry".

There is no strict line of demarcation existing between the drying and the non-drying oils. The one class merges, by insensible gradations, into the other. Accordingly, although it is quite easy to differentiate between typical drying and typical non-drying oils, such as between linseed oil and colza oil, yet it is not so with some other oils. Cotton-seed oil, for instance, is classed by some authorities as a drying oil, by others as a semi-drying, whilst some classify it as non-drying. The same remark applies to castor oil.

The principal characteristics by which the drying oils may be differentiated from the non-drying oils will be detailed in the description which follows of the methods of detecting adulterations in linseed oil.

The drying oils employed by varnish manufacturers are linseed oil, from *Linum usitatissimum* (the flax plant); poppy-seed oil from the *Papaver somniferum* (the opium poppy); walnut oil from *Juglans regia;* hemp-seed oil from *Cannabis sativa;* castor oil from *Ricinus communis;* cotton-seed oil from *Bombax* (species).

Many other kinds of drying oils occur in the different organs of various plants, but they are but little used in actual practice; amongst these are cucumber oil from *Cucumus sativus;* grape-seed oil from *Vitis vinifera;* bankoul oil from *Aleurites triloba.*

It is necessary, however, to make special mention of the very siccative oil, *Paulownie of Japan*, the fine oil of the *Dipterocarpus crispaletus*, the *Kai-dan-long* of the Annamites, and *Eloecocca*, Japanese wood oil, the best drying oil known.

LINSEED OIL—CRUSHING THE LINSEED.

The first stage in the crushing of linseed, according to the Anglo-American process, is to pass it through the rolls (Fig. 13).

ROLLS.

The linseed is conveyed to the hopper by an elevator or shoot, and is distributed to the crushing rolls (Fig. 13) by a fluted feed roller the same length as the feed rolls placed at the bottom of the hopper. When the seed passes the feed roll it falls on a guide-plate, which carries it between the first and second rolls. After passing between these rolls and being partially crushed it falls on a guide-plate on the other side, which carries it between the second and third rolls, where it is crushed more fully. It then falls on another guide-plate, which carries it between the third and fourth rolls, where it is ground more fully still. In the larger rolls it falls on a fourth guide-plate, where it is conveyed between the fourth and fifth rolls and receives the final grinding.

It will thus be seen that the seed has been crushed four times in its passage through the rolls. The rolls, being brought into contact by a combination of a screw and spring, give a smooth and easily regulated pressure. When the seed falls from the bottom roll the grinding is much more complete and perfect than in seed that has passed through rolls and under stones of the old description.

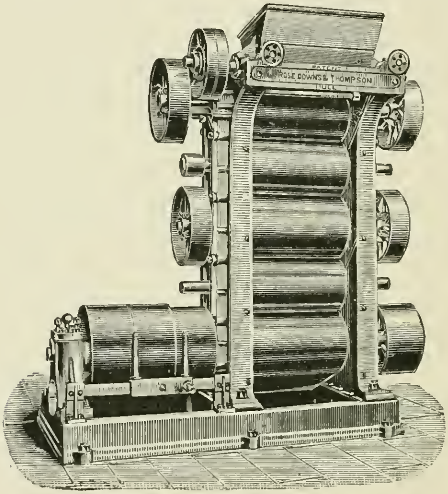

Fig. 13.—Rolls for Crushing Linseed.

Steam-Heating Kettle and Moulding Machine.

After passing the rolls the seed falls into a screw conveyer, which places it in an elevator that raises it to the steam-jacketed kettle (Fig. 14, A), where the seed is heated and damped by a jet of steam and agitated by a revolving stirrer. These kettles are made of cast-iron, and are constructed in the strongest and best manner. There is only one steam joint in them, and that is faced in a lathe or planed quite true. There is therefore little liability to leakage—always a dreadful nuisance.

When lagged, the kettle body is fitted with a wooden frame and covered with felt, and the felt is enclosed in iron sheeting.

It will be observed that up to this point in the process the operations have been automatic.

In connection with the kettle is worked the moulding machine (Fig. 14, B). This is worked by a man, who by its means measures out sufficient seed for a cake, forms it of the proper shape, cases it in a strip of bagging, and then passes it to the pressman to place in the presses.

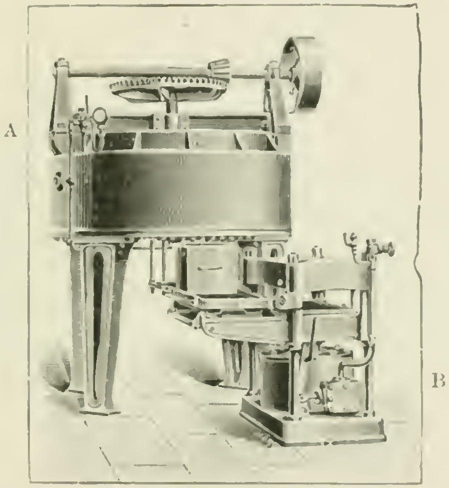

Fig. 14.—A, Steam Kettle; B, Moulding Machine.

Hydraulic Presses.

These presses (Fig. 18, A) each hold sixteen cakes, and are furnished with steel cylinders 16 inches in diameter, which carry a working pressure of one and three-quarter tons on each square inch of the ram. All the columns, cylinders, rams and heads are planed and turned accurately to gauges, so as to ensure that every part will take its due proportion of strain and no more; the pockets that take the columns are not cast as is usual, with fitting strips top and bottom, but are all solid throughout, and are all machined out of the solid to gauges. This method of treating the pockets has the effect of almost entirely preventing the columns from breaking.

In certain types of presses the pockets for the columns are bored and the columns are fitted with steel nuts.

The plates between which the seed is pressed are all well fitted and have a corrugated surface. If a brand be required on the cake as, say, letters about 3 inches long are clearly cut into one side of the plate and all the remainder is corrugated. These plates are all made of wrought and cast malleable iron, and the columns are made of best scrap iron or mild steel. The cylinder is crucible cast steel.

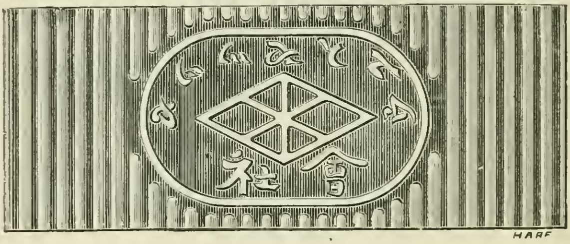

FIG. 15.

PUMPS.

The pumps are made of the highest class of crucible cast steel and are all bored out of the solid. Two of the pump rams are 2½ inches in diameter and have a stroke of 7 inches. These rams give only a limited pressure, and the arrangements are such as to give the limited pressure on each press in about fourteen seconds. The pumps then stop automatically. The work is then taken up by two other pumps having rams 1 inch in diameter and a stroke of 7 inches, and is continued by them until a gross pressure of two tons per square inch is attained. This is the maximum and is arrived at in less than a minute.

The oil as it comes from the presses falls into the tank underneath, which serves as a foundation for the presses, and is pumped from there into the store cisterns.

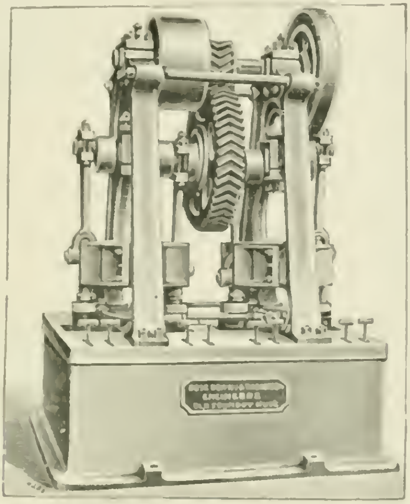

FIG. 16.—PUMPS.

CAKE PARING AND MOULDING MACHINE.

The cakes as they come from the presses require paring. The parer strips his cake and lays a number of these on a stool or table convenient to his machine (Fig. 17). He then takes up one and lays it on the machine, having one side parallel to the trough and overlapping it about an inch, one end being placed against a fence, either to the right or left, depending on what stroke the machine is taking. The knife block passes along and cuts one side clean and straight.

The cake is now turned over and the other edge of the same is treated in a similar manner. He now turns the cake half round, places one side against a fence, and cuts off the oily part at one end, then reverses the cake and does the same with the other end. The cake is now ready for the market, and has been pared by two double strokes of the knives, the speed of which is about thirty per minute. When required, fences or gauges are added that give all the cakes one exact length and breadth, but when these are used more parings are made, as the fences have to be set to suit the worst cakes.

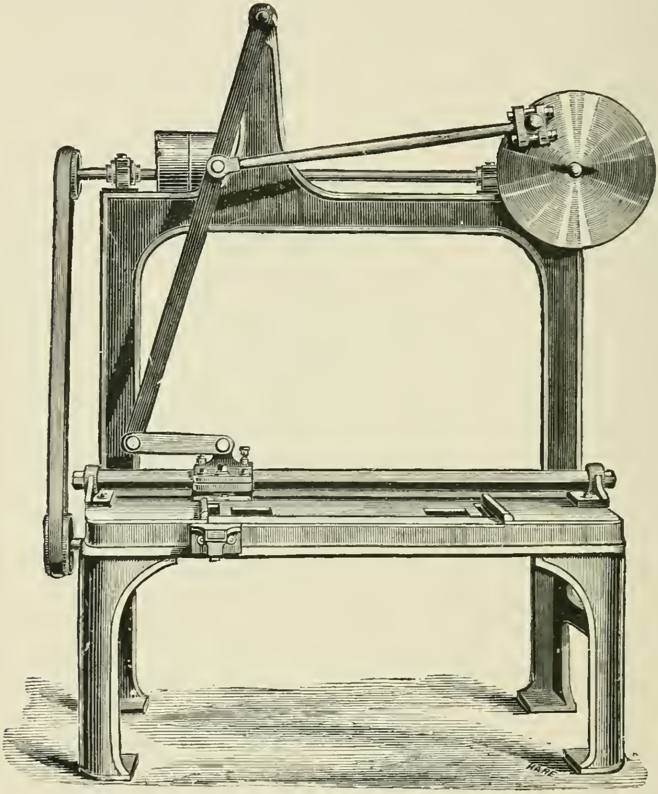

FIG. 17.—CAKE PARING MACHINE.

These machines can be driven by a shaft either parallel with or at right angles to the knife bar, and are suitable for paring either parallel or taper cakes.

Edge Runners for Grinding Cake Parings.

The parings from the cake-paring machine are taken to the edge runners (Fig. 18) to be ground. These are made of the best Derbyshire grit stone, and are furnished with a set of cast-iron centres. One centre has a long boss on it, which passes right through the stone and the centre on the opposite side, and has a long brass fixed on each end of it. These centres are securely fixed to the stones with bolts that pass through them, and they are also run into the stones with lead. On the outside centre there is a cap fixed with screws, quite oil-tight, and large enough to hold several pints of oil. The spindle on which the stone revolves passes through the centre of the upright shaft, and the stone centre has a cap fixed on each end of it with cotters. These caps are enclosed in the oil-cups described above, and revolve among the oil contained in them, thus causing such a perfect lubrication of the spindle as to make it quite unnecessary to withdraw it oftener than once in several months. The casting that carries the vertical shaft passes through the bedstone and goes beneath it, projecting beyond its circumference at six points, where it is attached to the circular pan above by six brackets. The top of this casting is fitted with a "step," constructed in several parts which can be removed or replaced in a few minutes without disturbing the driving wheels. The vertical shaft is made of cast-iron, about 7½ inches in diameter, having an oblong chamber or naval formed in the centre, into which is fitted a strong cast bush having a brass bush at each end through which the stone spindle passes.

The bush is free to rise and fall in the naval as material is added to or taken from the stones. All the sweepers and sweeper frames are made of wrought iron. The circular pan is made of cast-iron, dished slightly, and having a rim round its circumference 7 inches deep. All the bearings are brass, made very heavy and unusually long. The ground parings are carried from the edge runners by a conveyer, from which they fall into the seed kettle.

The process of refining the crude linseed oil is as follows:—

The crude oil is taken from the store tanks by a force pump into a lead-lined cistern, where it is mixed with the necessary chemicals from another cistern. After these have done their work and the process completed, as described hereafter, the oil is run into a third tank, where it is agitated by air from an air-pump to complete the bleaching and refining process.

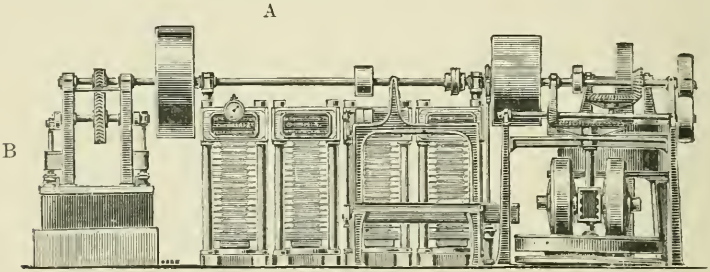

Fig. 18.

The accompanying illustration shows a complete set of the plant above described with the exception of the oil-refinery, constituting what is known as the Anglo-American Oil Mill, No. 4. It forms the standard size or unit of this machinery; larger mills are made up of multiples of this mill, and contain from eight presses upwards.

The Anglo-American machinery, as manufactured by Messrs. Rose, Downs & Thompson, is worked in sets of four presses as shown, and a mill can contain any number of sets. One set occupies a space 36 feet by 28 feet by 16 feet high, and takes 45 horse-power to work it. Additional sets will take about 35 horse-power each.

The complete process is therefore as follows. The seed passes through rolls, from these an elevator places it in the kettle, from this it passes to the moulding machine, where it is measured, formed and compressed, and then passed to the presses. The cakes, after being pressed, are cut to size in the paring machines and the parings reduced to meal by the small edge-stones. The parings are then returned by an elevator to the kettle to be re-worked. The presses stand in a tank, into which the expressed oil falls and from which it is forced to the store tanks or filters by a pump. Fig. 19 shows an illustration of filter press.

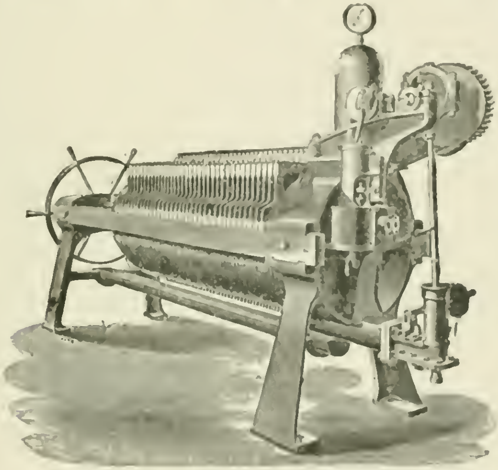

FIG. 19.—FILTER PRESS FOR OIL.

LINSEED OIL—CHEMICAL AND PHYSICAL PROPERTIES.

Linseed oil has a density varying from ·9325 to ·9348 at 15° C.; it dissolves in 5 parts of boiling, and in 40 parts of cold, alcohol. It commences to thicken at – 16° C., solidifying at 27·5° C. to a yellow mass.

Acid value varies.
Saponification value 193-197.
Elaidin test. It does not solidify.

Maumené's test	111° C.
,, absolute	$306 = \left(\frac{111 \times 100}{\text{divided by heat giving out in mixing 50 c.c. sulphuric and 50 c.c. water.}}\right)$
Iodine absorption figure	156
Bromine ,,	10·000
Acetyl figure	8·7
Fatty acids, melting-point	23° C.
,, solidification-point	21° C.
,, density	·9205
Deviation (polarisation)	Nil

Adulterations.—Linseed oil may possibly be adulterated with fish oil, mustard-seed oil, rosin, rosin oil, linoleic acid, cotton seed, hemp seed, camelina, poppy seed and rape. The principal adulterant is rosin oil.

Linseed oil, containing fish oils, darkens with chlorine. When mustard-seed oil is present, it is because the linseed has previously been adulterated or contaminated therewith. The presence of this oil, which diminishes the drying capabilities, is detected by exposing the sample to a temperature of 10° to 12° C. After a few days a yellow, flaky deposit is formed.

Rosin is indicated by a great increase in density, and may be further detected by heating the oil with twice its volume of alcohol, which dissolves the resin. The alcoholic solution is decanted, and an alcoholic solution of basic acetate of lead added, which, in the presence of rosin, produces a very abundant flocculent precipitate.

Rosin oil may be detected by the great increase in density and by a polarimetric examination. Any right-handed deviation indicates rosin oil.

It may be, quantitatively, estimated by saponifying 5 c.c. of the oil with 25 c.c. of alcoholic potash (80 of potash to 1000 of alcohol) on the water bath evaporating to dryness, dissolving the resulting soap in water, transferring to a separating funnel, and repeatedly extracting with ether. The collected ether extracts are distilled, and the residue consisting of rosin oil or mineral oil weighed.

Rosin oil dissolves in glacial acetic acid. Mineral oil is insoluble in that reagent and is of less density than rosin oil.

Linoleic acid has been added to linseed oil for some time back. It may easily be detected by its solubility in alcohol and by the greatly increased acid value.

Other vegetable oils may be detected by the lowering of the density.

Testing of Linseed Oil.—This is very often limited to taking the gravity, at 15° C., by the oleometer, and to acid coloration tests. Sulphuric acid of density 1·638 is added to the oil in a saucer; linseed oil takes a green colour (Livache).

A better test is the sulphuric acid spot test. One drop of concentrated sulphuric acid on 10 drops of oil. Pure linseed oil passes gradually from orange through red to dark brown. If even 5 per cent. of rosin oil be present the spot is dark brown at first, passing quickly to black. Fish-liver oils give a violet spot with this test. Anhydrous perbromide of tin applied similarly gives a violet spot in the presence of rosin oil.

One volume of an aqua regia (consisting of 24 volumes of hydrochloric acid of density 1·156, and 1 volume of nitric acid of density 1·333) is caused to fall on 5 volumes of oil; after four to six minutes the oil becomes greenish yellow. Finally, if to the oil thus treated there be added 10 volumes of a solution of soda of 1·350 density, in the case of genuine oil a yellow coloration is produced, the mixture remaining fluid.

Iodine absorption.—The iodine absorption (Hubhls) of the oil should be determined. The oil ought also to be submitted to different reagents to detect extraneous oils by the coloration produced, also to Maumene's test.

Maumené's Test.

1. The sulphuric acid to be used in this experiment should be pure, of specific gravity 1·845, and should be kept

in a well-stoppered and capped bottle. The stopper should not be left out a moment longer than that required to extract the necessary quantity. Have the acid in a bottle 6 inches high, with a thermometer inside.

2. Counterbalance a glass tube, on foot, standing heat (about 1½ inches in diameter and 7 ounces capacity), and weigh in accurately 50 grammes of the oil.

3. Immerse both the sulphuric acid and the tube containing the oil in water contained in a tin vessel of 5 inches deep, 2 quarts capacity, and apply heat. As soon as both the acid and the oil are at a temperature of 27° C., draw out 10 c.c. of the acid with a pipette and let it flow gradually at the rate of 1 c.c. every five seconds into the oil, without touching the sides, stirring very energetically all the time with the thermometer. After all the acid is in, continue to stir exactly for half a minute, then move the thermometer more slowly, noting the exact degree at which it ceases to rise.

A pure sample of oil should be tested in the same manner precisely, immediately before the suspected article. No two persons, unless actually working side by side, will ever get absolutely identical figures, and rarely then. So delicate is the rise of temperature, that the same operator must needs be careful to obtain constant figures on repeating the experiment on the same oil.

In the case of linseed oil and fish oils the action is very energetic, abundant fumes being given off, whilst the mixture, assuming a gluey consistence, thorough mixing of the acid and oil is a matter of great difficulty. Moreover, it is not at all easy to obtain satisfactory readings of the thermometer, the more so as the latter has to be used to stir the oil. It is therefore advisable to mix linseed, etc., oils in known proportion with an oil on which sulphuric acid has no great action, such as petroleum lubricating oils.

Knowing the heat given out by mixing the mineral oil alone with sulphuric acid, it is easy to calculate how far its admixture with linseed oil has affected the latter.

But beyond these different tests, viewing the matter in a practical light, the drying properties of the oil ought to be determined before everything else. This is done by pouring a certain quantity of oil into a cup or saucer so as to have a layer 1 millimetre in thickness, $\frac{39\cdot37}{1000}$ inches, and exposed to the air at a temperature of 40° to 80° C. After a certain time, which for good quality oil should not exceed three days, the layer ought to dry without tackiness.

Livache's Precipitated Lead Test.

The transformation of a drying oil into a solid elastic substance is due to an absorption of oxygen. Linseed oil absorbs 14 to 16 per cent. of its weight, and it is easy to directly measure the quantity absorbed by imbibing in the oil a little lead well washed and dried *in vacuo*, obtained by precipitating a lead salt by a zinc plate; the whole is exposed to air as long as the weight varies; the increase in weight indicates the amount of oxygen absorbed.

By this process, when conducted with the necessary precautions, the quantity of oxygen absorbed by any sample of linseed oil may be rapidly ascertained, and, if need be, whether a pure oil is being dealt with or an adulterated oil, or an oil which has already undergone oxidation to a certain extent. In all cases it can readily be ascertained whether an oil corresponds with a given sample.

This reaction ought to draw the attention of practical men, because samples are often met with which, although equally genuine, take longer time to dry. Now, the precipitated lead test indicates the difference in duration of the time taken by the oil for complete oxidation, and thus predicts beforehand the results which any oil will give on the large scale in regard to drying propensities.

Process.—One gramme or thereabouts of the precipitated lead is weighed in a tared watch glass, then 0·5 gramme of oil is dropped upon it drop by drop, spacing out the drops in such a way that a little dry lead rests between each of them. At the end of two days the following results were noted :—

	Increase per cent.
Linseed oil	14·3
Walnut oil	7·9
Poppy-seed oil	6·8
Cotton-seed oil	5·9
Beech oil	4·3

The following oils showed no results during the first two days. After a week the following increase was noted :—

	Increase per cent.
Colza	2·9
Sesamum	2·4
Earth-nut	1·8
Rape	2·9
Olive	1·7

Bishop's Silica and Resinate of Manganese Test.

1. Ten grammes of the oil are weighed in a capsule, and 2 grammes exactly of the resinate of manganese added. The capsule is put into the water bath, stirring it from time to time until the complete solution of the resinate, from five to ten minutes being sufficient for this. It is then cooled.

2. One gramme of silica is weighed in a capsule with a flat bottom furnished with a little glass stirrer. With the aid of a dropping tube, a quantity of oil, as near as possible approaching 1·02 grammes (*i.e.*, 1 gramme of oil plus 0·02 of resinate), is allowed to fall drop by drop over the whole of the surface. Both the weight of the oil and the total weight are noted. By means of the stirrer the oil is mixed with the silica in such a way as to have a divided mass, perfectly homogeneous, covering the whole of the bottom of the capsule. This is left at a temperature of from 17° to 25° C. in the case of drying oils, of 20° to 30° C. for the others, and weighed at the end of varying periods ; for instance, six hours, sixteen hours, twenty-two hours—that is to say, three

times in the twenty-four hours. After each weighing the surface was renewed by agitation with the stirrer. The degree of oxidation is furnished by the maximum augmentation multiplied by 100 when 1·02 grammes weighed exactly is operated on. Using pure linseed oil and the same oil with 2 and 4 per cent. of resinate the following were the results. The linseed oil had a specific gravity of 0·9322 at 15° C., and through the experiments the temperature ranged from 20° to 25° C. :—

Increase per cent. at the end of	No Drier added.	2 per cent. of Resinate added.	4 per cent. of Resinate added.
6 hours	0	12·35	14·10
22 ,,	0·50	15·65	15·50
24 ,,	0·80	15·85	15·30
30 ,,	2·50	16·25	15·30
48 ,,	7·30	15·65	14·90
72 ,,	15·00	14·65	14·10
96 ,,	16·10	15·15	13·60
120 ,,	15·30	14·05	13·20
144 ,,	14·90	13·75	13·20
168 ,,	14·30	13·35	13·20
288 ,,	14·00	13·25	13·10

These experiments showed in a decisive fashion the energetic action of resinate of manganese as a drier, since this salt produced the total oxidation of the oil three or four times as quickly as in operating with simply divided oil. It also confirms the conclusions of the experiments of Cloez and those more recently made by Mulder, Bauer, Hazura and Livache on the formation not only of solid products but also of volatile products. If the oxidation is too strong, as in the case where 4 per cent. of resinate was added, a final result is obtained very much more quickly and at the same time a feebler increase, this phenomenon being explained by reason of the formation of a higher proportion of volatile products. In the experiments that follow, therefore, 2 per cent. of resinate has been used :—

Increase per cent. at the end of	Pure Linseed oil. Sp. gr. 0·9322. Temperature 17° to 23° C.	Linseed oil containing 5 per cent. of Rosin oil and 5 per cent. of Mineral oil. Sp. gr. 0·9323. Temperature 17° to 23° C.
6 hours	13·50	11·50
22 ,,	16·30	14·80
24 ,,	16·40	14·90
30 ,,	16·20	14·80
48 ,,	15·90	14·60
120 ,,	14·80	13·80

The influence of temperature is very great, and it would seem useful in the case of linseed oils to operate at a temperature not exceeding 28° C., but above 17° C., in order to effect the oxidation in as short a time as possible. The experiments showed also the inferiority of exotic linseed oils of a low specific gravity, and also showed that it is possible to establish by a comparison the inferior quality arising from clever adulteration.

Oils.	Specific gravity.	Average degree of maximum oxidation.	Proportion.
French Linseed - - -	0·9327	17·05	—
Linseed from La Plata -	0·9304	15·20	1·12
Hemp-seed - - - -	0·9287	14·40	1·18
French poppy-seed - -	0·924	14·20	1·20
Commercial walnut - -	0·924	19·70	1·23
Demargarinated cotton-seed	0·923	3·45	1·80
Non-demargarinated cotton-seed - - - - -	0·924	8·60	1·98
Senegal sesamum - -	0·9215	8·70	1·96
Indian sesamum - -	0·921	7·40	2·30
African earth-nut - -	0·916	6·70	2·54
White earth-nut - -	0·916	6·50	2·62
French colza - - -	0·9142	6·40	2·66
Indian colza - - -	0·9137	5·85	2·91
Olive - - - - - -	0·9155	5·30	3·21

The degree of oxidation can therefore be advantageously used to control the iodine value, and in many circumstances it is even capable of replacing it with advantage. In fact, in addition to its great simplicity and the minimum cost of carrying it out, which makes it a very practical method for the industrial laboratory, this method furnishes very often a more rational and more complete indication than the Hubl number, as it permits many minor interesting details to be noticed. In consequence, this process will serve to fix the value and establish the nature of the identity of an oil, whether taken separately or compared with a type. Moreover, it may be applied in the case of certain mixtures, not only of oils among themselves, but also of commercial products, such as lard and alimentary fats, which often contain very variable quantities of vegetable oils. In this last case, instead of oxidising directly either the melted and clarified fat or the fluid part extracted by pressure, the liquid fatty acids may be operated upon, separated from the solid acids by Halphen's process. The oil is saponified, the soap dissolved in water, and precipitated with acetate of lead. The lead soap is extracted with ether, and the ethereal solution of the lead salts of the non-saturated fatty acids is precipitated with weak sulphuric acid. Indications still more interesting and more precise are obtained by taking the iodine value before and after oxidation. The determination of the degree of oxidation gives results in immediate connection with the value of the oils for certain of their industrial purposes, and, as has been shown, certain falsifications can be detected by the process. Moreover, it will show whether recently crushed or old tanked oil is being dealt with.

Refractometric and Polarimetric Tests.

The investigations of Bishop and Peter on the opticity of a number of oils show that, with the exception of croton oil and rosin oil, the only dextro-rotations are produced by sesame (high) and olive oil (feeble), all the others, including linseed oil, being either optically inactive or having a slight levo-rotatory power. The saponification test is reliable in presence of rosin oil or compounds of resin acid with metallic oxides and mineral oils, but is uncertain when resin is present.

A much more delicate test, however, is that given by Hefelmann's "refractometer," by the aid of which even small quantities of rosin, rosin oil, metallic resinates and mineral oils can be detected, as will be gathered from the following table of observations made with homogeneous (sodium) light :—

Refraction of Linseed Oil by Hefelmann's Refractometer.

	Refractometer Number at 200° C.
Pure linseed oil (raw)	80·5 to 82·2
,, ,, ,, boiled	80·5 to 84·2
Linseed oil + 20 per cent. rosin	above 100·0
,, ,, + 10 ,, ,, ,,	94·5
,, ,, + 5 ,, ,, ,,	88·4
,, ,, + 20 ,, ,, rosin oil	above 100·0
,, ,, boiled + 20 per cent. resin oil	above 100·0
,, ,, ,, + 10 ,, ,, ,, ,,	92·7
,, ,, ,, + 5 ,, ,, ,, ,,	88·2
,, ,, ,, + 15 ,, ,, cotton-seed oil	78·7
,, ,, ,, + 15 ,, ,, rape oil	79·1
,, ,, + 10 per cent. mineral oil	89·6
,, ,, boiled + about 30 per cent. manganese resinate	84·9

Optical Deviation of Drying, etc., Oils with Jean's Refractometer.

Raw Linseed Oil.	
Laboratory standard sample	+ 53°
Refined raw linseed	+ 49°
Raw linseed + 20 per cent. rosin oil	+ 67°
,, + 20 ,, hemp seed	+ 47°
,, + 20 ,, mineral oil	+ 17°
Poppy (œillette)	+ 29°
,, old	+ 35°
Poppy (pavot) Calcutta	+ 27·5°
,, old	+ 33°
Hemp seed	+ 30° to 32°
Walnut	+ 36°
Ravison	+ 25°
Colza	+ 17 to 18°
Rosin oil[1]	+ 78°

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[1] With some samples the whole field of the refractometer is black.

BOILED LINSEED OIL—CHEMICAL AND PHYSICAL DATA OF.

	Refraction at 25° C.	Polarisation.	Iodine number.	Saponification number.	Saponification (turbidity) test
Litharge boiled oil 1	84·1	feebly—	163·0	188·9	clear
,, ,, 2	84·2	,,	167·1	—	,,
,, ,, 3	84·1	,,	165·0	—	,,
,, ,, 4	82·8	,,	167·5	188·4	,,
,, ,, 5	82·9	,,	162·3	188·7	,,
Manganese boiled oil 1	80·6	,,	172·1	188·9	,,
,, ,, 2	84·0	,,	170·4	186·1	,,
,, ,, 3	82·9	,,	167·7	190·2	,,
,, ,, with 3 p.c. resin oil 4	84·9	±0	164·8	185·4	slightly turbid
Pure linseed oil	80·6/84·2	feebly—	160/180	180/190	clear
Litharge boiled oil 6	95·8	strongly +	143·6	160·8	quite turbid
,, ,, 7	96·3	,,	158·3	169·2	,,
,, ,, 8	89·6	,,	151·7	169·3	,,
,, ,, 9	91·9	,,	153·4	167·5	,,
Manganese boiled oil 5	90·0	,,	161·1	163·3	,,
,, ,, 6	over 100·0	,,	129·8	163·6	,,
Boiled oil + 20 p.c. rosin	,, 100·0	slightly +	acid number 15·6	197·6	slightly turbid

Therefore, if a sample of raw or boiled linseed oil, examined at 25° C., is found to give a refractometer number greater than 84·5, it is open to the suspicion of containing an admixture of rosin, rosin oil, mineral resinates, or mineral oil. On the other hand, if the refraction falls below 80, then additions of slightly or non-drying fatty oils must be looked for. Very small percentages are not detected by the refractometer, but such are not likely to occur in practice.

In how far the refractometer test coincides with the results of chemical analysis, see table, page 245. All oils exhibiting normal refractions were found pure, and those refracting abnormally adulterated. The foregoing were purchased as pure boiled oil, with the exception of the last, containing 20 per cent. of rosin, which was prepared in the laboratory. Out of fifteen commercial samples, nine were pure and six contained rosin oil, the only adulterant detected.

A slight turbidity on the addition of water to the saponified oil (p. 243) indicates the presence of small admixtures of resin or metallic resinates. (The production of a dark, flocculent, heavy precipitate in the case of manganese boiled oil should be disregarded as common to all boiled oil of this class. It cannot, therefore, be considered as indicative of the presence of rosin oil or mineral oil.) The polarisation test gives either neutral or slightly dextro results, and both saponification and iodine numbers fall within the ordinary limits, so that no differentiation between slight quantities of added resin or metallic resinates is possible.

WALNUT OIL.

Density at 15° C.	·9285.
Solidification-point—	
Thickens between	– 15° and – 18° C.
Solidifies at	– 30° C.

Solubility in alcohol	Dissolves in 22 times its weight
Maumené's test	99° C.
„ relative	275
Elaidin test	liquid mass
Absorption figures:—	
Iodine	144
Bromine	7·37
Acetyl	7·3
Fatty Acids, fluid at ordinary temperature:—	
Density at 15° C.	912
Polarisation	Neutral

This oil, known as nut oil, is a *drying* oil, and must not be confounded with other *non-drying* nut oils. It is obtained from the kernel of the common walnut, *Juglans regia*. (Juglans = Jovis glans or Joves acorn.)

Artists' Oil.—The kernels are peeled and pressed in the cold, when they yield a rather cloudy oil difficult to clarify, of a pale yellowish green tint, equal to, if not superior, in drying propensities to linseed oil, with an agreeable smell and nutty flavour.

Virgin Oil.—The walnut kernels are not peeled, but the fully-matured nuts are stored for two or three months until decomposition sets in, when the kernels are pressed, yielding in the cold 25 per cent. of their weight of virgin oil. The oil expressed in this manner is used as an edible oil, and as a substitute for linseed oil.

Second Quality Oil.—The marc from the first pressing is ground with hot water and again pressed, and the dark inferior oil, with an acrid taste and smell, thus obtained is used as a paint oil.

Walnut oil dries to a more elastic film or coat, with less tendency to crack than linseed oil. For fine, delicate and white colours the better qualities of walnut oil are highly prized by artists on account of their being almost colourless, but their cost is too great for general use.

Adulterations.—It is adulterated by mixing it with cotton, poppy-seed, sesame and earthnut oils, which are recognised by the colour tests, iodine absorption, by Maumené's test or the freezing-point.

When pure the sulphuric acid test gives a brown coloration, aqua regia a yellow colour, and on the addition of soda orange striæ.

Precipitated lead gives an oxygen absorption of 8·5 to 7·5 per cent.

Poppy-Seed Oil.

Density.—Between ·9242 and ·925 at 15° C.

Solubility in alcohol.—1 part dissolves in 6 of boiling, and 25 of cold, alcohol.

Solidification-point.—Thickens at – 18° C. Solidifies at – 20° C., when purified solidifies at – 18° C. (Mileau).

Elaidin test	Liquid mass
Maumené's test	80° C.
,, relative	222
Absorption figures:—	
Iodine	133
Bromine	835
Acetyl	131
Fatty acids:—	
Solidification-point	16° C.
Melting-point	20·5° C.
Density	·9082

European poppy-seed oil is more drying than that obtained from Indian seed, but the former is more costly.

Adulterations.—Sesame oil and cotton-seed oil, which may be detected by the colour tests.

By the precipitated lead test poppy-seed absorbs 7 per cent. of oxygen.

Hemp-Seed Oil.

Density.—Between ·925 and ·9276 at 15° C.

Solubility in alcohol.—Soluble in all proportions in hot or in 30 per cent. of cold alcohol.

Solidification-point.—Thickens between – 15° and – 16° C., and solidifies at – 27·5° C.

Adulterations.—Generally used to adulterate linseed oil; detected in latter by lower iodine absorption and greater length of time in drying.

Sulphuric acid gives a deep brown coloration, aqua regia a green, the addition of soda producing bright brown striæ.

COTTON-SEED OIL.

Density at 15° C., ·9322, refined, ·9222.

Solubility in alcohol.—In 16 times its weight.

Solidification-point, –1·5° C.

,, Stearine-freed oil, – 8° C.

Elaidin test	Liquid mass
Maumené's test	52° C.
,, relative	141° C.
Absorption figures:—	
Iodine	105
Bromine	645
Acetyl	15·3
Fatty Acids:—	
Melting-point	between 36° and 37·5° C.
Solidification-point	between 32·5° and 35° C.
Density at 15° C.	·9097
Deviation	Variable

Cotton-seed oil dries fairly; it is not often adulterated, and is frequently used to adulterate linseed oil.

The black coloration produced by the reduction of nitrate of silver in presence of the saponification products of the refined oil is characteristic, and the oil can thus be detected when used to sophisticate other oils.

The precipitated lead test gives an oxygen absorption of between 5 and 6 per cent.

CASTOR OIL.

Density at 15° C.	·9611
Purified oil	·965 (Mileau)

Solubility in alcohol.—Completely soluble in cold alcohol. Characteristic.

Solidification-point:—	
Thickens at	– 16° C.
Solidifies at	– 18° C.
Elaidin test	Yields ricinelaidin
Maumené's test	40° C.
,, relative	111° C.
Absorption figures:—	
Iodine	84
Bromine	579
Acetyl	153

Fatty Acids:—

Melting-point	12° C.
Solidification-point	4° C.
Polarisation deviation	43° C.

This oil is not generally adulterated. Sulphuric acid gives a yellow coloration, aqua regia a green, and the addition of soda yields bright red striæ.

If resin oil be suspected, it may be detected by agitating the suspected sample with its own volume of nitric acid of specific gravity 1·31. The acid which is colourless with pure oil becomes of a more or less deep brownish yellow according to the amount of resin oil present.[1]

Castor oil is distinguished from all other oils by its complete solubility in alcohol. Magnesium ricinoleate is also soluble in alcohol. Both may be used in spirit varnishes to produce elasticity.

WOOD OIL (JAPANESE).

Specific gravity at about 12° C.	·9385
Freezing-point	below – 17° C.
Iodine number	165·7
Saponification number	194
Hehner's number	96·4
Unsaponifiable matter	·54 per cent.
Temperature elevation	372
Free acid (reckoned as oleic)	3·84 per cent.
Melting-point of the fatty acids	37°
Freezing-point of same	34°
Iodine number of same	150
Viscosity (taken with Redwood's viscosimeter, 12½°)	41

Wood oil is the best drying oil known. It is but slightly fluid, colourless, inodorous and insipid.

When spread upon a glass plate it dries completely in a few hours, but covers the glass with a milk-white opaque film. But the oil dries transparent when previously heated with litharge.

Cloez published the results of a careful study of this oil in the *Comptes Rendus de l'Académie de Sciences*, vols. 81, 82 and 83.

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[1] Moreover, the solubility in alcohol would be diminished *pro rata*. For quantitative estimation, see under "Linseed Oil," p. 236.

It is produced by the oil tree of China and Cochin China, the *Eloecocca vernica* (Tong Yeou), the seeds of which contain 53 per cent. of oil, 80 per cent. of which is got by crushing. When heated to 200° C. it thickens; at 260° to 280° C. the oil gelatinises. It should therefore not be heated above 180° C. Unlike other drying oils it does not dry from without inwards, but the process goes on simultaneously through the thickness of the coat of oil. This oil is well adapted for mixing with linseed oil, the latter component giving elasticity and the former hardness and resistance, as well as an increase in drying properties.

The attention of varnish-manufacturers is directed to this oil. It is a known fact (says Livache) that large quantities are yearly imported into England, but for what purpose is unknown. Now its remarkable drying capabilities in the cold, and without previous heating, suggests to him its direct use in England in the manufacture of high-class varnishes.

But against its being used to any extent in England, at least until recently, is the fact that the oil merchants, on the other hand, advertised it as being *extensively used on the continent.*

Chemical Constitution of Drying Oils.

Since all oils consist of carbon, hydrogen and oxygen in almost identical proportions, it may well be asked what principle it is that is present in certain oils and causes them to dry, and which must therefore be absent in those oils which do not dry.

Linolein.

Mulder made a particular study of this question, and found that the principal constituent of linseed, poppy-seed and walnut oils—as well as that of all drying oils—was linolein, a substance formed by the combination of linoleic acid with glycerine. His conclusions have been partially confirmed by Hazura and Friedreich, who found in poppy-seed and walnut oils fatty acids analogous to, if not identical with, linoleic acid.

More recently, however, as a result of fresh experiments, Hazura has been led to regard linoleic acid as composed of two distinct acids, *linolic* and *linolenic*. But Reformabsky disputes these conclusions.

As a consequence of still more recent researches by Livache (*Comptes Rendus de l'Académie de Sciences*, 1895), he holds that the theory that the drying properties of linseed oil is entirely due to the presence of linolein can hardly be maintained. In fact all other oils, whether of vegetable or animal origin, are capable of being transformed into a solid product analogous to that which linseed oil is so easily converted, and that whether taken individually or as the component parts of a mixture, provided always that they be submitted to the action of heat.

According to the present state of our knowledge of this subject, it seems that the best explanation that can be given of the drying properties of oils is the following: *All* the different glycerides which enter into the composition of a drying oil play a part in the transformation of the oil into a solid body, but the greater the proportion of one or several glycerides analogous to linolein the more rapidly is this transformation effected, and at a lower temperature. All the glycerides present in oils and fats undergo this change more or less slowly, as if by a kind of metamorphosis.

Looking at the matter from this standpoint, we are able to explain the differences—in drying properties—of different drying oils. Should in fact the glycerides, analogous to linolein, be present but in small quantity, we can understand that the other glycerides will only dry slowly, and very often we must resort to the aid of heat to accomplish the object in view. Whatever explanation we adopt, we must bear in mind that the drying oils are those which may be quickly converted into a solid elastic substance at the ordinary temperature.

Experience has further shown that this transformation can only take place in the presence of oxygen, and with a rapidity which varies much according to the heat and light the oil is exposed to, and the previous treatment to which it has been subjected.

LINOXIN.

In the case of linseed oil Mulder called the resulting solid product linoxin. He found that it did not differ from linoleic acid but by containing a larger proportion of oxygen, whilst at the same time all the glycerine had disappeared, whilst making reservations necessitated by the still imperfect state of our knowledge regarding the composition of the different solid bodies obtained by the oxidation of different oils, we shall retain the name of linoxin. For this body, no matter what oil it may be derived from, presents the same properties of elasticity, insolubility in the usual solvents, etc., etc.

Linoxin consists of a perfectly dry elastic mass, of a more or less deep yellow or brown colour according to the manufacture of the oil from which it had been derived. For a long time it was believed to be perfectly insoluble in the different menstrua in which oils dissolve. When exposed to their action it at first undergoes no change, but if the action be prolonged it increases in transparency, swells like india-rubber, and at the same time a small proportion dissolves.

On drying the resulting swollen substance separately, there still remains an elastic mass, but very friable between the fingers, crumbling to particles, with no tendency to re-unite. On the other hand, by evaporating the solvent there is left a tacky residue of low melting-point. The oxidation product of a drying oil, therefore, presents many analogies with caoutchouc, being composed like it of two elements, one of which dissolves in different menstrua, whilst the other swells and disintegrates. The dissolved product recovered on evaporation of the liquid solvents acts as a real cement, reuniting the insoluble portions, first swollen and then disintegrated, yielding as a result a continuous, elastic mass, consisting on the one hand of the soluble, and on the other hand of the insoluble, portion of the original linoxin.

From what we have said it follows that before an oil can dry, the linolein must be in a position to become oxidised, and the more this is facilitated the quicker does the oil dry, as the oxidation of the other glycerides would appear to be one of the functions of the oxidation of the linolein.

Now by suitable means the oxygen may be caused to act either upon the linolein, *i.e.*, upon linoleic acid combined with glycerine, or upon the linoleic acid separated from glycerine, or finally upon suitable chemical combinations of linoleic acid with metallic oxides, *i.e.*, upon linoleates.

Before taking up the drying properties of oils from an industrial point of view, we must first study the conditions under which this oxidation takes place.

Action of Oxygen upon Linolein.

If we take the best and quickest drying oil, *viz.*, linseed oil, and expose it to the air in a thin layer, we soon see it change to a solid substance.

If we perform the same experiment in a vessel containing air placed mouth downwards over mercury, we see the same change take place, but the volume of air confined over the mercury diminishes in volume owing to the absorption of oxygen. Finally, if the quantity of air suffices, the oil is converted into a solid product which has increased in weight proportionally with the oxygen which existed in the air and which has disappeared, whilst the residual gas is composed of nitrogen, together with a small quantity of carbonic acid, and volatile acids of the methane series.

The transformation of the oil and consequently of the linolein is therefore due to the action of oxygen.

Mulder obtained this solid body by exposing the oil on plates to the action of the air, and after detaching, he treated the product with ether, alcohol and water so as to wash away any unoxidised oil or other soluble matter: and he finally obtained a more or less elastic white substance which analysis showed to be a product of the oxidation of the anhydride of linoleic acid, linoxin.

On the other hand, he experimented directly upon linoleic acid exposed in a thin layer to the air, and obtained very easily the same solid linoxin, but the change into a perfectly dry substance was of longer duration than in the case of linolein. But the products are identical in composition and properties, and as no glycerine is found in the product of the oxidation of linolein by the air, it would appear that the oxygen of the air first acts upon the glycerine, yielding such bodies as carbonic acid, water, etc., which disappear, and then upon the linoleic acid of the linolein, converting it into a solid body, linoxin. The product is identical whether we use air or oxygen.

Different circumstances influence the rapidity of drying.

An increase of temperature exercises a very decided influence; oxidation takes place more rapidly, whilst at the same time, the oil being more fluid, the oxidation is more thorough owing to the oxygen penetrating the oil better and more deeply.

The intensity of the sun's rays has a very decided action. Oil exposed to direct sunlight dried in four days, whilst the same oil in similar circumstances, but in semi-darkness, took fifteen days to dry.

According to Cloez, when the light is caused to pass through coloured glass the oxidation varies with the colour. The maximum amount of oxidation takes place with colourless glass, but with blue, red, green, or yellow, a longer time is required the nearer the colour approaches to yellow.

When oil is exposed to air in darkness, it is longer before the oxidation process sets in, and it takes a very long time for complete oxidation.

Action of Oxygen upon Linoleates.

Linolein is very easily saponified, yielding soaps; potash, soda and ammonia yield soaps which readily dissolve in water. Baryta, lime, the oxides of zinc, copper and lead, yield soaps, insoluble in water but soluble in ether.

The most suitable combination to study is that of linoleic acid with oxide of lead; if we dissolve this linoleate of lead in ether, and if we expose the liquid in a thin layer upon a plate of glass, the white solid residue remaining on evaporation of the ether, which is at first soft, becomes in a few days very hard, owing to the absorption of oxygen.

Linoxic Acid.

If we suspend this hard, brittle salt of lead in alcohol, and pass a current of sulphuretted hydrogen through the alcohol, we obtain, after filtering off the sulphide of lead, an alcoholic solution from which water precipitates a white substance which analysis shows to be that oxidation product of linoleic acid to which the name of linoxic acid has been given.

If instead of separating this alcohol in the cold by the simple addition of water, we evaporate the alcoholic solution by the aid of heat, we also obtain a viscous residue, but of a blood-red tint.

Linoxic acid is therefore met with in two colours—white or red—according as it has been prepared in the cold or the hot, but the composition is the same in both cases.

But whilst viscous linoxic acid exposed to the air changes to dry elastic linoxin, on the contrary, when combined with lead—although it also suffers this alteration—it becomes more and more friable.

If we resume what we have determined in regard to linoleic acid we find that—

1. Linoleic acid combined with glycerine, in the state of linolein, yields progressively in a more or less short period of time linoxin, a solid elastic body, a basis for colours and varnishes.

2. Free linoleic acid yields fairly quickly a viscous compound (linoxic acid) which afterwards changes to linoxin, but after a longer period of time than in the preceding case.

3. Linoleic acid combined with oxide of lead, *i.e.*, *linoleate* of lead, dries fairly rapidly in consequence of the formation of *linoxate* of lead, but this product changes afterwards into a friable, brittle substance. As a result of these experiments, it naturally follows that, to ensure a dry, elastic product, we ought preferably to cause the oxygen to act upon the linolein and to avoid, as far as possible, either the presence of linoleic acid, which would take a longer time to dry, remaining at the outset in a viscous state for rather a long time in consequence of the formation of linoxic acid, or the presence of linoleate of lead, which would give a brittle, friable product. Owing to the difficulty of separating linolein from the other principles entering into the composition of oils in actual practice, we have to oxidise the oil itself.

Having established these principles, experience shows that the drying properties of a drying oil are increased under certain conditions, which it is important to study in detail. With this end in view, we shall study how the drying properties of linseed oil—the best drying oil—may be increased, for whatever we may determine regarding it holds good, keeping to the same proportions with other drying oils.

METHODS OF INCREASING THE DRYING PROPERTIES OF A DRYING OIL.

The rapidity of drying is influenced by :—

1. The degree or extent to which the oil has been refined. 2. The time which has elapsed since its extraction. 3. The conditions under which it has been preserved. 4. Heat. 5. Addition of certain substances.

1. *Extent to which Oil has been Refined.*—The oil as it issues from the press contains water and impurities, *mucilage* "*foots*". It is evident that to obtain a continuous elastic mass, it would be advantageous to get rid of these substances.

This may be done, naturally, by allowing the oil to clarify itself by simple deposition, by filtration, or by chemical treatment.

The latter, which is generally done by treating the oil with sulphuric acid, frees it from substances which play no part in the drying of the oil, because they do not fix oxygen; if, in fact, we expose to the air comparatively freshly pressed oil, and the same oil, after treatment with sulphuric acid, then freed by washing from all trace of acid, we find that the oil in the second case absorbs a larger quantity of oxygen.

Further, it is evident that the paler the oil the whiter will be the final product, *i.e.*, the dried coat of linseed oil thus demonstrating that the oil ought to be bleached before application.

2. *Age of the Oil.*—Oil preserved from contact with the air absorbs oxygen the more rapidly the longer it has been stored.

This has been said to be due to a polymerisation of certain principles of the oil. Dr. Fahrion thinks that the non-saturated fatty acids enter into combination with themselves to form complex addition products.

3. *Method of Storing the Oil.*—Leaving the length of time out of account, if the oil has been stored in contact with air it will already have absorbed a certain quantity of oxygen.

Now experience shows that if, in the beginning, the oil does not absorb oxygen but rather slowly, this absorption, once fairly started, goes on afterwards much more rapidly.

In the case of such an oil the absorption of oxygen will take place much more rapidly when applied to any surface exposed to the air.

4. *Action of Heat.*—The temperature at which an oil is placed in the presence of air has an influence on the rapidity of drying of that oil. Chevreul demonstrated that linseed oil dries more rapidly at a temperature of 25° to 28° C. than at a temperature of from 15° to 18° C.

Further, if we expose raw linseed oil under the same conditions of temperature to the action of air, and the same oil, after having been previously submitted to the action of heat, the drying property is altered. If, for instance, we heat linseed oil for three hours in such a manner that it only disengages a few gaseous bubbles from time to time, the drying properties of the oil are greatly increased; as has been shown by Chevreul it takes only about half the time to dry.

Dr. Fahrion states that when boiled in a deep pan, so that oxygen of the air cannot intervene to any great extent, the polymerisation of the non-saturated fatty acids are accelerated with the formation of complex substances which absorb oxygen more rapidly.

Chevreul, on the contrary, found by prolonging the heating of the oil—the temperature, etc., remaining constant—that oil heated for five hours took longer to dry than oil heated for only three hours. It is probable that under the influence of prolonged heating a certain proportion of the glycerine is destroyed (as the smell of acrolein would appear to indicate) with the simultaneous liberation of linoleic acid. We have previously seen that linoleic acid takes longer to dry than linolein. Probably this is the reason why

Mulder found that oil heated to between 70° and 100° C. took longer to dry than raw oil.

What we can say is that :—

(1) A regulated temperature stimulates the drying properties of raw oil.

(2) Exposed to the same temperature raw oil dries less quickly than when it has been previously submitted to the action of heat, provided always that the heat is regulated in such a manner that the oil does not decompose in any way so as to liberate free linoleic acid.

5. *Addition of Certain Substances : Driers.*—It has long been known that the addition of certain substances, such as white lead, litharge, black oxide of manganese, etc., stimulates the drying properties of a drying oil. Sometimes these substances may be added in the cold, sometimes the mixture is heated.

Let us consider in the first instance the lead compounds which have been so long employed as driers. The process of oil boiling in most general use is that in which the oil is simply heated with a lead compound. The increased drying properties of the oil has by some been attributed to the formation of linoleates of lead with the simultaneous production of free linoleic acid on heating, whilst others aver that the oil becomes oxidised at the expense of the linoleate of lead, as borne out by the fact that a certain proportion of metallic lead is found at the end of the operation.

This double explanation is not satisfactory for the following reasons. In the first place experience teaches that linoleate of lead only imparts a fictitious drying property because it very soon becomes brittle and friable, whilst on the other hand linoleic acid dries less quickly than linoxin. It is, however, an incontestable fact that an oil heated with oxide of lead dries quicker than raw oil, or oil heated by itself alone. We can therefore ask in the second place whether it is not at the expense of the oxygen of the oxide of lead that the oil increases in drying properties.

If we recall the fact that linseed oil of good quality exposed to the air absorbs 16 to 18 per cent. of its weight of oxygen before it dries completely, and on the other hand we reflect that in actual practice the quantity of oxide of lead, litharge, or red lead, does not exceed 3 to 8 per cent. at the highest estimate of the oil to be boiled, it will be seen that the amount of oxygen which this oxide of lead could yield would be less than 1 per cent. instead of the 16 to 18 per cent. which are necessary, and consequently that it can only play a very secondary part as far as the direct oxidation of the oil is concerned, much more so as a part of this oxide does not yield up any of its oxygen to the oil. Nevertheless the presence of a certain quantity of metallic lead at the end of the operation proves that a corresponding quantity of oxygen has started the oxidising process, and we have seen that in studying the action of oxygen upon raw oil that the drying properties of an oil increase more rapidly after the oil has reached a certain stage of oxidation. Taking into consideration the remarkable increase in drying properties, this reaction would in any case appear to be of a secondary nature.

The same objections and the same explanations hold good in regard to the function of manganese compounds as driers.

INFLUENCE OF SURFACE ON RAPIDITY OF DRYING.

But a better explanation may be given founded upon an observation of Chevreul, who showed that if we spread linseed oil on lead foil, freed from all traces of oxide, the drying qualities of the oil increase in a remarkable manner. Without doubt the oil cannot in this case absorb any oxygen but what the air can in its normal state furnish it with. The greater rapidity of drying can only therefore be attributed to the presence of the metal, leaving out of account the combination in which it may enter the oil.

But we may go further. If we shake raw oil along with very porous metallic lead—obtained by precipitating a salt of lead by another metal—in a flask deprived of air, we find that without applying any heat whatever the oil dries much more quickly than before treatment, and in this case it is certain that there has not been any oxygen supplied to the oil. The only certain modification which the oil has undergone is the presence of a small proportion of lead, and we are thus led to conclude that the reason the oil dries quicker is due solely to the presence of this small quantity of lead.

In the case of manganese we cannot avail ourselves directly of this experiment, but by an artifice we can place the oil in the same conditions with regard to manganese. We have only to stir the oil previously treated with the precipitated lead with a solution of a salt of manganese, the sulphate for example which by double decomposition will yield sulphate of lead insoluble in the oil, whilst at the same time the manganese takes the place of the lead. Working in this manner it has been found that the resultant oil dries quicker, better even than that obtained by heating raw oil with a manganese salt.

It is by studying the action of a manganese-treated oil that an explanation has been found of the cause of the increase in drying. If we spread such an oil in a thin layer in contact with air, it will be observed to assume a brown tint due to the passage of the oxide of manganese present to a higher degree of oxidation owing to the absorption of oxygen from the air, but this brown coloration fades as the oil becomes more viscous, finally disappearing altogether. The oxygen of the peroxide of manganese is used up in oxidising the oil, and in the end a perfectly colourless solid mass is obtained.

This coloration followed by complete decoloration shows that the oxide of manganese has played the part of an intermediary or carrier of oxygen, becoming easily peroxidised, and afterwards giving up its excess of oxygen to the oil. The action of oxide of lead is probably similar but less energetic. We are, in fact, face to face with one of those observations, the inference to be drawn from which has been defined by M. Berthelot as follows: "We see by these observations how latent energy, and energy capable in theory of producing exothermal phenomena, but which under given circumstances do not produce such phenomena, may be rendered manifest by the intervention of certain agents acting solely as intermediates or go-betweens, and capable of developing indefinite reactions. This is the foundation of the whole theory of thermochemistry as enunciated by me in 1865" ("Action by Presence," *Comptes Rendus de l'Académie de Sciences*, 1889, t. 109, p. 546). In a litharge or manganese-treated oil the lead or the manganese plays the role of intermediaries, taking the oxygen from the air and giving it up to the oil, which becomes oxidised more rapidly than without the aid of these intermediaries.

Vincent defined this action as catalytic, but only as far as manganese was concerned. Litharge and lead salts in his opinion dissolved in the oil, and thus intensified its drying properties: whilst manganese salts simply acted as carriers.[1]

It was natural to examine whether other metals could perform the same function as lead and manganese. It was found most convenient to take a litharge-treated oil and to stir it with a salt of the different metals to be tested, the acid of which would give with lead a salt insoluble in the oil. Working in this manner Livache found that litharge-treated oil spread on a glass plate dried in twenty-four hours, when manganese replaced the lead in six hours, whilst, when the lead was replaced by copper, zinc or cobalt, the oil took thirty

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[1] In virtue of the fact that both resinate and linoleate of manganese dissolve in the oil, Vincent's hypothesis requires modification.

to thirty-six hours to dry, and finally the oils in which lead was replaced by nickel, iron, chromium, etc., did not dry completely until after forty-eight hours.

A very important point to be borne in mind is the fact that an oil indirectly treated with manganese by the substitution of manganese for the lead of a litharge-treated oil dries much faster than an oil directly treated with manganese.

Lead and Manganese Salts.

We shall see, in actual practice, that besides the oxides of lead and manganese we may employ certain salts of these oxides. But their choice would appear to be subordinate to their degree of solubility in the oil, and to the manner in which they behave when heated along with the oil. Further all the numerous salts proposed have been discarded with the exception of the acetate (sugar) of lead and borate of manganese, and the reason is not far to seek. These salts in fact decompose when heated and yield as a final result either the oxide of lead or finely divided metallic lead, or oxide of manganese of which the valuable function is well known.

1. *Acetate of Lead.*—Let us take the acetate of lead. We know that this salt melts in its water of crystallisation at about 75° C. Above 100° C. it loses water and a little acid, yielding the sesquibasic acetate, which towards 280° C. is completely decomposed, giving off carbonic acid and acetone and leaving as a residue metallic lead in an extremely fine state of division (spongy lead). We have seen the important role this plays in stimulating the drying properties of oil.

2. *Borate of Manganese.*—Again, on the other hand we have in borate of manganese a very unstable salt, as is the case with borates in general. The affinity of boracic acid for oxide of manganese being but very feeble, the latter is liberated by the action of heat: the employment of this salt is therefore a useful roundabout way of introducing oxide of manganese into the oil.

As experiment showed that non-oxidised metallic lead, in a very porous condition, increased the drying properties of the oil, it was interesting to study the action upon oil of other metals capable of being easily precipitated. But the oil dried no quicker when treated with precipitated tin or copper Besides these results might have been foreseen, for Chevreul showed that linseed oil, spread upon well-polished lead, dried much more rapidly than when spread upon copper, brass, zinc or iron. With these metals the oil did not dry any faster than upon plates of glazed or unglazed porcelain, glass or plaster of Paris.

With regard to the numerous other substances besides the salts of manganese and lead which have been proposed to be added to drying oils to hasten their drying properties, none of them would appear to exert any beneficial influence whatever. Chevreul made a comparative study of the oxides of zinc and lead and showed that the latter had no sensible influence. He further pointed out that certain substances even acted in a contrary manner, retarding instead of hastening the drying of the oil ; of such are the oxide of antimony and the arseniate of the protoxide of antimony. If certain substances appear to act, the reason is to be found in the fact that they have been used in conjunction with the application of heat, and their apparent beneficial action is due to that alone.

According to Andés the following substances may be regarded as *absolutely useless :* all organic matters (sepia, dog excrement, bread, onions, garlic), red oxide of mercury, verdigris, lime, brass, zinc, alum, hydrated oxide of iron, boracic acid, oxide of antimony, gypsum, vermilion, pumice-stone, animal charcoal.

Weak Oxidising Agents.—White lead, sulphate of lead, carbonate of lead, basic acetate of lead, black oxide of manganese, hydrate of protoxide of manganese, sulphate of zinc, oxide of zinc, umber.

Energetic Oxidising Agents.—Air—acting through its oxygen —red lead, litharge and the different oxides of lead, borate of manganese and the hydrated peroxide of manganese.

Recently Kassner has suggested the use of the plumbates of the alkaline earths. These are prepared by heating in a suitable furnace two molecules of baryta, strontia or lime, or their corresponding carbonates with one molecule of oxide of lead.

The plumbate of baryta is dense black, that of strontian, brown, and that of lime bright red. Kastner is of opinion that the drying property of the oil is increased by the richness in oxygen and the introduction of lead ; he holds further that the introduction of the alkaline earths themselves may produce oleates, which after drying assume a consistency of remarkable elasticity. Summing up we ought therefore to confine ourselves in the use of driers to lead and manganese, to their oxides, and in certain cases to some one of their salts.

Catalytic Action of Driers.

An experiment of Chevreul lends support to the theory of looking upon manganese and lead as simply performing the function of intermediaries or oxygen carriers, *viz.* : If we make a mixture of raw linseed oil and manganese-boiled oil, the liquid resulting from this mixture has a much greater oxygen absorbing power than either of the liquids constituting the mixtures taken separately.

It is very evident from this experiment that the quantity of oxygen absorbed during the same period of time is not proportional to the quantity of oxide of manganese contained in the oil, since the mixture absorbs more oxygen, although bulk for bulk it contains less oxide of manganese than the original manganese-boiled oil. The only feasible explanation is this : If the raw oil in the mixture absorbs a greater quantity of oxygen in the same space of time, it is simply because this oxygen is supplied to it under more favourable conditions. Now these conditions are precisely the fixation of oxygen by the oxide of manganese, which forthwith gives it up to the oil; there is thus a continual transport of oxygen from the air to the manganese and thence to the oxidisable principles of the oil.

It seems to us that we may from the foregoing statements draw the following deduction, *viz.*: That an oil will dry in a better manner the less combined linoleic acid it contains, because linoleate of lead absorbs less oxygen than linolein and becomes brittle; all we have to do is to introduce a quantity of lead or manganese sufficient to extract from the air the quantity of oxygen necessary to oxidise the linolein as fast as this oxidation proceeds. A large quantity of lead or manganese does not hasten the oxidation—the intensity of which is the sole function of the drying quality of the oil used—but would on the contrary yield a less elastic and slower forming final product.

Again, summing up the proper conditions under which the drying properties of an oil are stimulated we can deduce the following principles:—

1. The oil ought to be refined and clarified.
2. There is an advantage in allowing the oil to age before using it; if need be, oxidation may be started.
3. It is more preferable to use oil which has been heated than to use raw oil.
4. The drying of raw oil or heated oil is hastened by the addition of either lead or manganese compounds in known quantities, or by metallic lead in a fine state of division.

It remains to consider how in actual practice we can confirm to the principles enunciated.

OIL REFINING AND BOILING.

Purification and Clarification.

The oil obtained from oleaginous seeds by pressure is very impure, and owing to the presence of easily decomposable foreign substances, amongst which albuminoid principles predominate, it cannot be used in its raw, unrefined condition. It must therefore be refined.

The first stage of the refining process is mechanical, the second chemical. The oil is purified mechanically by conveying it as it comes from the presses into a reservoir, from whence a pump distributes it to the filter presses, in which the greater part of the foreign substances which it holds in suspension are separated.

Chemical purification consists in treating the oil, after it has been previously mechanically clarified, in a methodical manner with dehydrating agents such as concentrated sulphuric acid.

By Acids: Thenard's Process.—By using a quantity of not too strong acid (1 to 1½ per cent. of 66° B.), the acid exerts its action on the foreign matters contained in the oil in preference to the oil itself—it acts at first by absorbing moisture; it then attacks the foreign matters and transforms them into a carbonaceous mass, which imparts to the oil a brown coloration, which, according to Livache, quickly deposits as a flocculent precipitate. When the acid has completely charred these substances water is turned on to dilute the acid, so as to prevent it acting on the oil. After being energetically agitated with injection of air, and repeated washing with water heated by an open steam pipe, the oil is conveyed into large tanks and left to settle. It only remains to separate the purified oil by decantation. According to Hartley, however, oil treated in this manner is itself frequently charred, or some impurity therein which dissolving in the oil imparts thereto a brown coloration, which is not removed by the subsequent bleaching process to which the natural colouring principles of the oil are amenable. This brown colour in his experiments sometimes separated as a flocculent precipitate, but only after prolonged subsidence. Success in this process, in fact, depends upon adding just enough acid of the proper strength and no more as will be sufficient to char the mucilage without attacking the oil itself. Hartley states that he succeeded well with acid of 30 per cent. strength, which, whilst charring the mucilage, did not attack the oil when left in contact with it.

It must be borne in mind that however expert a chemist may be, that fact alone does not constitute him an oil refiner, and, as a matter of fact, linseed oil can be refined and bleached perfectly water-white by sulphuric acid alone by so-called ignorant workmen who have the advantage of being practical.]

This oil is not, however, free from acid. Linseed oil containing even a small proportion of sulphuric acid when heated to the high temperature incidental to varnish-making would char. This, of course, would be obviated by the use in such a case of a basic drier, such as litharge or manganese, but then only if kept continually in contact with every particle of oil by agitation. Metallic salts decomposable by sulphuric acid, such as sugar of lead, would act similarly.

In any case the oil is not quite bright. In France the oil after treatment with acid and subsequent washing is conveyed into capacious metallic tanks, upon the bottom gratings of which a layer of sawdust is deposited, and then a bed of cotton waste, and then a grating of willow ensures an even distribution of oil over the filtering material. This aggregation of filtering materials is preferable to the different mixtures often recommended, such as sand and sawdust, shale, mould, etc., and the use of canvas filter bags. These filters are placed in an atmosphere maintained at a temperature of 25° to 30° C. The oil, which escapes by a stop-cock at the bottom, passes into a second similar filter.

The selection of sawdust is said not to be a matter of indifference from the point of view of the quality of the oil, for pine sawdust, for example, imparts to the oil a reddish tint, whilst that of oak turns it blackish. Notwithstanding its relatively high price, poplar sawdust should be used.

A very practical method of rapid filtration was given by Ure. The filter is composed of three parts, of which the filtering material, charcoal, waste, etc., occupies the middle. The oil, placed in an adjacent reservoir, communicates on one hand with the lower compartment, and on the other hand by a pipe leading from its bottom with a reservoir of water placed at a sufficient elevation. The water displaces the oil, and causes it to pass through the filtering material, and to be transferred into the upper compartment.

[In these days of filter presses, and working on the large scale, trivial filtering processes like the above are rather out of date.]

Other processes of purifying oils chemically have been recommended, such as a concentrated solution of chloride of zinc, as suggested by Wagner and recommended by Hartley, heating the oil with freshly calcined magnesia; coagulation of the albuminous matter by warm steam pipes, or by the injection of air heated to 110° C.; addition of tannin extract or of salts of iron or alumina to precipitate albuminous matter; partial saponification with a small quantity of caustic lye, so as to carry down the foreign matters with the resultant soap in much the same manner as beer is refined with isinglass, a process employed with success in the purification of cotton-seed oil. Hartley recommends a concentrated solution of sulphate of manganese.

It would be superfluous to dwell longer upon these different processes. This short enumeration will be sufficient.

The oil thus purified is then stored in large reservoirs.

Bleaching of Oil.

In the manufacture of high-class varnishes, it is indispensably necessary to use an oil which has been deprived of its natural yellow colour. Even with an oil which only shows a very faint yellow tint the quality of the varnish into which it enters will be deteriorated. It is therefore necessary to bleach the oil.

The colouring principles of oils are derived from powerful colouring matters existing in the fruits and seeds from whence they have been extracted.

Hartley, who has studied these colouring matters, has identified four principal substances of very similar chemical composition: Xanthophyll and chlorophyll, both yellow, another, but blue, chlorophyll, and finally erythrophyll, of a red colour. These substances are not necessarily found in *all* oils, but nevertheless they are always present in linseed oil, and the variations in their relative proportions is the cause of the different colours which different samples of this oil exhibit.

If erythrophyll and a mixture of the two chlorophylls predominate we get a brown oil; if a greater proportion of chlorophyll be present the oil will have a greenish brown tint; finally, if zanthophyll predominates a pale yellow oil is the result. These substances are decolorised by sunlight, especially in contact with air; oxidation also destroys them; they are also easily decolorised by dilute acids. Alkalies and certain metallic salts first precipitate the chlorophyll, then in the long run, if added in excess, the other two substances are likewise precipitated; finally, they are rapidly decolorised by chlorine and hypochlorites.

Five processes of bleaching oils are in use based upon the preceding remarks:—

1. By sunlight alone, or by sunlight and air acting together.
2. By oxidising agents.
3. By acids.
4. By alkalies or metallic salts.
5. By chlorine.

1. *Action of Light.*—In decolorising oil the best result is obtained by the prolonged action of sunlight, and a superior article is obtained to that yielded by the use of chemical reagents.

When linseed oil is exposed in a thin layer to the direct action of sunlight, it bleaches in two hours. Working with large quantities the process is of course of longer duration, but by using large flasks of colourless glass, and exposing the oil in these to direct sunlight, bleaching proceeds very rapidly, and so that the oxygen of the air may aid in the operation the mouths of the flasks are simply plugged with cotton wool.

When it is desired to treat larger quantities the oil is placed in flat lead-lined or zinc-lined boxes about 40 inches long by 20 inches wide, and 6 to 10 inches deep, covered with a glass plate, and slightly inclined by raising one of the sides of the box about ½ inch or so; or the glass plate is made to overlap the sides of the box, so that no rain water gains access to the contents of the boxes, which are generally placed in the open air. Finally, two tubes lead into the box from the two opposite sides, so that the air on the surface of the oil is being constantly renewed. By means of this plant linseed oil can be bleached to a bright colourless oil in less than a fortnight.

The process can be further accelerated, according to Mulder, by previously filtering the oil through animal charcoal. It has also been suggested to expose the oil to sunlight in contact with animal charcoal for a week, and not to filter the oil until then.

2. *By Oxidising Agents.*—(*a*) *Peroxide of hydrogen*, which is now easily obtainable in commerce, has also been recommended. The oil is shaken with 5 to 10 per cent. of peroxide of hydrogen of 10 per cent. strength. This process would appear to be only adaptable to small quantities. Expense would debar its use on the large scale, and, as a matter of fact, quite irrespective of expense, both peroxides of hydrogen and sodium have no practical value as oil-bleaching agents, however energetic they ought to be theoretically in this respect.

(*b*) *Ozone.*—More recently attempts have been made to use ozone. It is made by causing a current of air to pass through a series of ordinary ozonising tubes, where it becomes richer and richer in ozone, being finally led to a receiver containing the oil to be oxidised. The oil is heated by a steam coil to about 40° to 50° C., and the ozonised air is admitted to the bottom of the vessel, and is made to pass through the whole of the oil by means of a tube pierced with very small holes.

E. Schrader and Otto Dumcke, who have made a study of this subject, found that ozone only acts upon the oil for a comparatively short time—it in fact stops very quickly. But if the oil thus treated be placed in white glass flasks in flat boxes, in contact with air and preferably in the sun, the action continues of its own accord bleaching and thickening the oil in a very short time, and causing it to dry much more rapidly.

Although information and particulars in regard to the bleaching of oils by ozone are far from definite, this process ought to receive the attention which it undoubtedly deserves.

(c) *By Permanganate or Bichromate of Potash.*—The permanganate, or, better still, the bichromate of potash, in conjunction with sulphuric acid, have long been used as bleaching agents. The process is conducted in wooden tanks lined with lead. For every 100 lbs. of oil about ½ lb. of bichromate of potash is mixed with double its weight of sulphuric acid, previously diluted with five times its bulk of water. This mixture is run into the oil in a thin stream, with constant stirring, which is kept up for an hour, the oil, if need be, being heated all the time by a steam coil. The liquids are allowed to separate, the lower layer drawn off, and the oil repeatedly washed with hot water.

This process is tedious, owing to the difficulty of eliminating even by acid the green hydrated oxide of chromium which dissolves in the oil. The same remark applies to bleaching with manganese compounds where a similar hitch occurs. There is a great loss of oil with both processes altogether incompatable with the extent of any to which the oil may be bleached. However, in both cases the oil may increase in drying properties owing to the action of the nascent oxygen developed in the processes in question.

By Linoleate of Manganese.—Blenkinsop and Hartley have proposed to bleach oil by oxide of manganese introduced in the state of linoleate of manganese dissolved in coal-tar naphtha. The process involves the use of heat. The oxygen used up in oxidising the oil and bleaching the colouring matters is restored to the manganese by a current of air as fast as it is deprived of it.

3. *By Acids : (a) By Nitric Acid, etc.*—Lawson has suggested dilute nitric acid. The process should be most carefully watched, so as to prevent any elevation of temperature. In England, according to Livache, use is made of a mixture of nitric acid and chlorate of potash in the proportion of 1 to 2 per cent. of the oil to be decolorised ; heat is applied, and the oil is then repeatedly washed with water. This process has been successfully employed with cotton-seed oil.

(*b*) *By Sulphuric Acid.*—Sulphuric acid has also been used, and gives very good results. The process is identical with that used in the purifying of oil by this acid, but it is recommended not to push the washing of the oil so far as to free it from all trace of acid, because oil bleached in this manner, and afterwards boiled with lead compounds, darkens much less during the process than when it does not contain any trace of sulphuric acid. The nitric acid and sulphuric acid processes are seldom used.[1]

4. *By Alkalies and Metallic Salts : (a) By Caustic Soda.*—Certain oils, containing resinous colouring principles, particularly cotton-seed oil, may be completely decolorised by agitation with a small quantity of *caustic potash* or *soda*

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[1] According to Livache, but the latter is extensively employed in England, and is the only really practical method of refining and bleaching in one operation.

(1 per cent.). Combination with the colouring principles ensues, and as a result these are precipitated.

(*b*) *By Carbonate of Potash.*—Five parts of the carbonate dissolved in 100 parts of water are added to the oil, and the mixture stirred to thorough incorporation; 25 parts of a 2 per cent. solution of chloride of calcium are then added. The oil bleaches rapidly, and it is then decanted and treated with a 5 per cent. solution of sulphuric acid, and afterwards washed until perfectly neutral.

(*c*) *By Ferrous Sulphate: Artists' Oil.*—Amongst the salts proposed, ferrous sulphate (green vitriol) is capable of giving good results, with small quantities of oil. *Process.*—One part of green vitriol is dissolved in 1½ parts of water, and this mixture is added to double its volume of linseed oil contained in a glass flask. The whole is then exposed to sunlight, and shaken at least once a day. The oil is generally bleached in from three to six weeks, according to the amount of sunshine. When the oil is decanted the green vitriol solution can be used over again for treating a fresh quantity of oil. This process is much in vogue with artists' colourmen, the bleached oil being sold as *artists'* oil.

(*d*) *By Basic Acetate of Lead.*—A solution of basic acetate of lead when agitated with oil eliminates its colouring principles very effectually.

(*e*) *By Sulphate of Lead.*—This process also gives good results. It is mixed with the oil to the consistency of cream, and exposed as in the preceding process to sunlight, with frequent agitation. After a time the oil is bleached. Two layers are found at the bottom of the flask, one consisting of sulphate of lead, the other of colouring matter. The sulphate of lead may be used over again. The rationale of the process is obscure. It may to a certain extent be looked upon as similar to the clarification of beer by isinglass.

(*f*) *By Complex Mixtures.*—More or less complex mixtures of different metallic salts are also used. We will only name the following, which gives good results, without seeking to demonstrate the precise reactions which simultaneously occur. To 100 parts of linseed oil are added 16 parts of water, containing $\frac{1}{2}$ part of black oxide of manganese, $\frac{1}{4}$ part of bichromate of potash, $\frac{1}{4}$ part of carbonate of soda, and $\frac{1}{4}$ part of common salt. The boiling solution is added to the oil; it is left to clarify, and the colourless oil is decanted.

5. *By Chlorine.*—Substances from which chlorine can be generated without the aid of heat are added to the oil. For instance, 5 parts of concentrated muriatic acid, 33 per cent., diluted with 4 times its weight of water, are added to 100 parts of oil. The whole is well stirred, whilst a solution of 1 part of bichromate of potash in 10 parts of water are added.

A mixture of red lead and hydrochloric acid may also be used. To 100 parts of oil $\frac{1}{2}$ part of red lead beaten up with $\frac{1}{2}$ part of oil is added, and the whole well stirred, whilst $\frac{1}{2}$ part of hydrochloric acid is added. During the next five days an additional $\frac{1}{2}$ part of hydrochloric acid is added daily, the whole being well stirred several times during the twenty-four hours. The decanted oil is run into large bottles, or placed in boxes lined with lead, and exposed to sunlight until the oil is perfectly colourless.

Summary.—The most rapid method of bleaching oils is by means of chemical reagents, especially chlorine, but it must not be forgotten that it is difficult to free the oil from all trace of these reagents, which in the end may exert a vexatious and injurious influence upon the final products manufactured from an oil bleached in this way. Certain bleaching agents, like chlorine and nitrous acid, are apt to form substitution compounds which alter altogether the nature of the oil, and not for the better. Had one space at his disposal, and a sufficient supply of refined oil to meet his wants in the meantime, the bleaching of oil by means of sunlight in flat boxes covered with glass plates cannot be too highly recommended. The slowness of the process would be largely compensated by the beauty and quality of the products manufactured from such an oil.

ACTION OF TIME.

Whatever theory one may adopt as to the cause, it is an undeniable fact that oil kept for a certain time gives a better coating on drying to that yielded by an oil fresh from the press. Speaking generally, linseed oil should not be used until after it has been "aged" for a year or two. Such oil is known as old tanked oil, and gives better results in varnish-making than can be obtained by any mere rapid process of bleaching freshly pressed raw oil.

METHODS OF STORING.

Galvanised iron reservoirs are to be preferred for storing oil. Their base should be very wide in proportion to their height; they are generally covered by a badly-fitting lid, so that the air may be constantly renewed on the surface of the oil, but it is preferable to use a tight-fitting lid so as to exclude dust. In this case a current of air is set up by two tubes placed on opposite sides of the reservoir, thus starting oxidation, and thereby increasing the drying properties of the oil. It is also of importance to maintain the oil at a constant temperature, about 15° to 20° C. It is advisable to draw off the "foots" two or three times a year.

OIL BOILING.

This operation is conducted generally in cast-iron pans, which are advantageously made in the form of an inverted truncated cone, so that in frothing up or priming the oil may spread over a larger surface.

Recently oil-boiling vessels have been constructed of enamelled iron, especially on the continent, but their use is for obvious reasons far from general in this country.

In general the pan is half filled with oil, and heated for three hours. For some purposes the oil is set on fire a few minutes before the fire is drawn.

At other times the oil is heated to a much higher temperature, *viz.*, 200° to 225° C., and this temperature is maintained in some cases as long as eight days. So that the heat may not accidentally rise too high a small quantity of tin is added to the oil, and care taken that the heat does not reach its melting-point, 228° C.

Should a viscous or a good bodied oil be required, the temperature is raised to 316° C., and kept thereat from 6 to 8 hours. But in the case of oils to be used in the preparation of pale varnishes, it is not desirable to heat the oil to too high a temperature, which would impart to it a brown colour and, moreover, start the destructive distillation of the oil, thus liberating a certain proportion of linoleic acid, which dries more slowly than linolein.

In order to produce a superior article the heat should not be raised so far as to start destructive distillation or "crack" the oil. The oil should rather be kept at a temperature which would bring about polymerisation, an object attained by slow boiling at a temperature regulated between 150° and 200° C. The lower the temperature at which the oil is kept the longer does the process last. This is the method adopted in England, where the oil is boiled for a very long time, often extending over a week; but by working in that way a very pale oil is obtained, presenting great elasticity and possessing properties which more than compensate for the length of time occupied in the process.

Treating Oil with Driers.

The above considerations of the influence of different driers show that those which have really a beneficial influence upon the drying of oils are lead and manganese either in the state of metals, oxides or salts.

In actual practice the oxides of lead and manganese or their salts are preferred; nevertheless the use of metallic lead has lately been tried with promising results.

Driers are generally incorporated with the oil by aid of heat. However, interesting attempts have been made to effect this result in the cold.

OIL BOILING BY NAKED FIRE.

1. When only small quantities of boiled oil are required, the most simple plan is to boil the oil in an iron or copper pan of about 20 gallons capacity, with an enlarged mouth to prevent the oil from frothing and threatening to prime over. This pan is at a certain height furnished with a collar or circular flange which supports it on the rim of a sheet-iron furnace, fed preferably with wood charcoal.

The pot being filled to the extent of half of its capacity with oil, and therefore containing about 10 or 11 gallons, the fire is lighted, and as soon as boiling commences the driers are added in small quantities at a time. The proportion and the nature of the driers used vary much, according to Andés; for the quantity of oil in the pot, either of the following mixtures may be used:—

2¼ lbs. of red lead and 2¼ lbs. litharge; or
2¼ lbs. of litharge and 2¼ lbs. sugar of lead; or
1½ lbs. of red lead and 3¾ lbs. sugar of lead; or
1½ lbs. to 3¾ lbs. of borate of manganese; or
2¼ lbs. of hydrated oxide of manganese.

The driers are previously ground as finely as possible, and the oil well stirred after each addition. As soon as the driers are all in and the frothing has ceased the pot is filled with oil just up to the neck, and the fire regulated so that the temperature does not rise above 220° C. by means of a thermometer with metallic framework. The operation is generally complete in three hours, when the pot is withdrawn from the fire, and the oil is set aside to clarify; or if it be desired to start to boil a fresh batch the oil is run into a sheet-iron or galvanised-iron reservoir.

Although this is a very simple arrangement, it has the great drawback that the pot full of hot oil has to be lifted down from the top of the furnace, thus greatly enhancing the risk of accidents. Further, when several consecutive boilings are made the heat is but badly utilised.

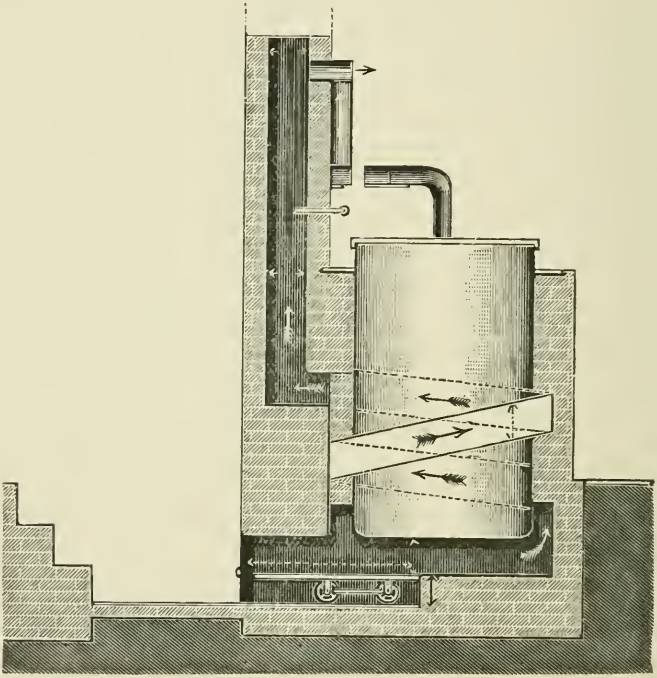

Fig. 20.—Plant for Oil Boiling by Naked Fire.

2. To remedy these inconvenient defects a furnace built in a framework of masonry is used. This furnace is covered with an iron plate, with a circular hole into which the pot fits.

More often, however, the fire is underground, thus enabling the workman to watch the operation at his ease, since the pot is then on the same level as the ground.

A rather different kind of pot (Fig. 21) is used to that illustrated in Fig. 20, and so constructed that the oil does not receive throughout its whole mass and at the outset the heat stored up in the brickwork. To obviate this the pan of about 20 to 22 gallons capacity, generally of enamelled cast-iron, is contracted in the middle, and egg-shaped at the base, so that the bottom only is exposed to the action of the heat. Otherwise the process is conducted in the same way as before, only the oil is heated a little longer, say, four hours instead of three.

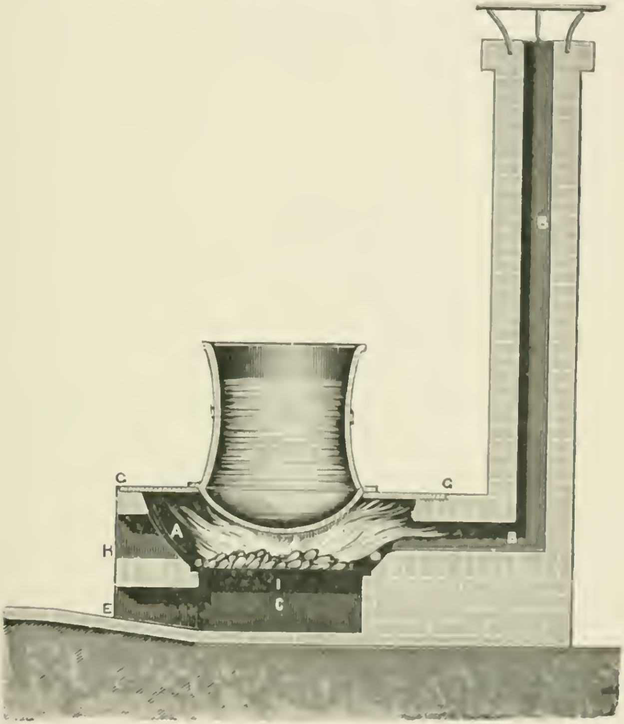

FIG. 21.—OIL BOILING POT. A, B, *Flue and Chimney Stack*. C, *Plate on which Lug of Pot rests.*

When large quantities of oil are to be boiled no good purpose is served by multiplying operations. It is better to use larger pans. In an extensive business the pans may measure 6 feet 6 inches high by 5 feet in diameter. Nevertheless we more often meet with pans having a capacity of 60 to 120 gallons. The pan is of strong iron plate about $\frac{1}{6}$ of an inch thick, is built into masonry in such a manner that the upper part controls a circular gutter of 4 to 5 centimetres deep, which, in case of the oil boiling over, conducts it to an adjacent receiver.

The pan can be so built into the brickwork that it may be heated on a part of its bottom and its sides, or, on the contrary, and preferably, on its sides alone. Accordingly, in the first case it rests on the brickwork in such a manner that the centre part of the bottom is exposed; in the second case, on the contrary, the bottom is supported by a small arch of brickwork, so as not to overheat the bottom part, thus preventing solid matters from tenaciously adhering to the bottom.

The cylindrical form of pan is not always adopted. We often come across pans almost conical in shape, the narrow part of which forms the bottom—a form which has the advantage of distributing the heat more evenly, and, owing to the larger section, of diminishing, in case of frothing, the chances of the oil running over.

Using this form of pan the process is as follows: The pan is half or at the most two-thirds filled with oil and at first heated gently, and when the oil reaches 100° C. the impurities which appear on the surface are skimmed off. The driers, previously ground very finely, are then added. It would not do to add them in the beginning, as they would fall to the bottom, and their beneficial action be to a great extent lost.

Andés gives the following proportion for 20 gallons of oil :—

2¼ lbs. red lead, 2⅕ lbs. litharge; or
2⅕ lbs. litharge, 2⅕ lbs. sugar of lead, or
1⅗ lbs. borate of manganese; or
2⅕ lbs. hydrate of manganese.

Boiling is kept up for five hours at a temperature not exceeding 220° C.

The temperature is regulated by means of a thermometer in metal framework, or, better still, by an aneroid thermometer, which is more legible, and not so fragile. Sometimes the heat may be regulated by adding some tin, and taking care that the heat does not rise to the melting-point of the latter, *viz.*, 228° C. The degree of heat is also ascertained by dipping the quill end of a feather into the oil, which ought to at once shrivel and curl up.

The more attention there is paid to ensure a constant regulated temperature, the brighter and paler will be the oil.

When very quick drying properties are aimed at the oil is boiled from one to two hours longer, and the amount of driers increased from 20 to 25 per cent.

At the time of adding the driers the oil should be well stirred either with an iron spatula, or preferably by means of mechanical agitators (a revolving shaft with arms, or a simple hanging chain fixed at both ends to a horizontal shaft, bent twice at right angles, fixed above the pan. The chain descends almost to the bottom of the pan and is turned by means of a crank some distance away).

Wood charcoal is the fuel used. This allows a constant temperature to be maintained if the fire be well regulated. Coke will suit equally well. So as to lessen the risk of the temperature rising too suddenly, which would colour the oil and cause it to overflow or aggravate the risk of fire, it has been suggested to use a movable grate, which can be lowered instantaneously, and thus cause the fuel to fall into a pit full of water. Movable car furnaces running upon rails, and thus capable of being easily removed, have also been employed.

Oil is sometimes heated by immersing the pan in a sand bath, or in a bath of a suitable alloy. But this plan is hardly to be recommended. In point of fact, if for any reason the temperature rises too high, the oil cannot be withdrawn from the source of heat—the same thing sometimes happens when the pan is built into masonry, for even in the case of a movable furnace the oil remains exposed to the action of the heat stored up in the brickwork.

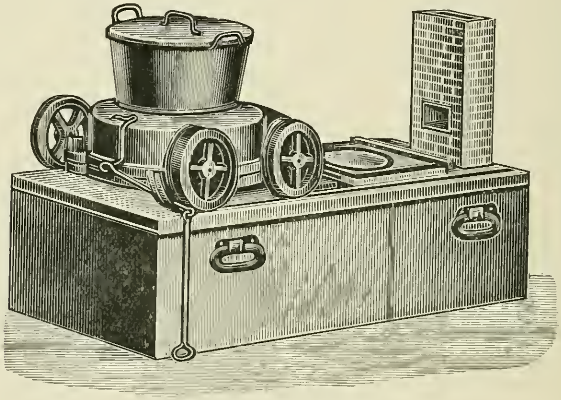

Fig. 22.—Movable Furnace.

To remedy this inconvenience, the pan, which sometimes measures more than a cubic yard, is mounted on a kind of trolley. The circular opening of the fire is on a level with the ground. If the temperature rises too high all that has to be done is to remove the trolley supporting the pan from the fire ; a movable hood with a counterpoise descends over the top of the pan and carries away the fumes. This arrangement not only enables the oil boiler or varnish-maker to watch the operation very easily, but at the same time preserves the metal of the pan. It therefore presents many advantages.

When the operation is terminated the oil is ladled out, and on the large scale pumped, into a galvanised iron tank. As soon as the pan is empty it is immediately re-charged with another batch, and generally without being cleaned in any way. Working in this manner, it has been found that the coating which forms on the sides of the pan prevents the oil from darkening, but care must be taken to remove any excess of driers from the bottom of the pan, otherwise unintentionally an excess of driers, which might have an injurious effect, may be added to the next batch. When after a time the skin becomes too thick it is scraped off. In certain factories this is only done once a year, or once in two years.

PREVENTION OF NUISANCE.

During boiling pungent, very inflammable, vapours are given off. When working with small quantities of oil all that is generally deemed necessary to prevent fire is to have a cover at hand to place on the pot should the oil take fire.

On the large scale, when the oil is heated in a pan, a hinged lid is fixed over the pan during the boiling by means of a cord. If the oil takes fire the cord burns, and the cover falls automatically on to the pan.

Different arrangements have been adopted so as to do away with the inconvenience of the pungent vapours. On the small scale, a simple hood connecting with a draught pipe of sufficient elevation is placed above the pot. A better plan is to fix a sheet-iron cap above the pot (Fig. 20), provided with an opening in front by which the process may be watched, or the contents of the pot stirred. This cap is connected by means of a pipe with a tall chimney shaft, the lower part of which is closed by a plug. Here the thick viscous products with a vile odour condense. They are removed from time to time by opening the plug. The more volatile products escape from the top.

But it is better not to let the volatile products escape directly into the air, but to cause them to pass through a condenser in connection with the chimney. A very simple arrangement consists in covering the boiling plant with a movable lid, provided with a window in the centre capable of being opened so as to allow of stirring the oil. On the side is a disengagement tube, which can be luted to a pipe connected with a condenser. The latter may be a pipe, shaped like an organ pipe leading into a vessel containing water, or it may consist of a vertical cylinder, the interior of which contains a coil of piping, through which cold water circulates. The vapour passes through the space *not* occupied by the pipes, and is in a great measure condensed. It is advisable to use an aspirator because the vapours are very heavy, but working with one or other of these arrangements condensation is almost complete. If need be the uncondensable vapours may be led through a fire to burn them as shown in Fig. 23, p. 291.

Often no previous condensation is attempted, but the vapours are led directly into the fire. At first it was supposed that explosions would occur, but the large quantity of air which is mixed with the volatile vapours does away with any danger. It is, however, necessary to be very careful that the oil in the pan does not catch fire when these products are conveyed directly to the fire which heats the pan, or to a special furnace. The oil may in fact take fire by the heavy vapours condensing in the conduit almost as soon as they leave the pan, and should there be any flaw in the arrangement, they may run slowly backwards towards the fire and there become inflamed, and thus by the flames running back heat to redness the connecting pipe, which is generally of sheet iron, and thereby set fire to the condensed products in proximity to the boiling oil, and thus to the oil itself. This is easily remedied by protecting the lower part of the conduit pipe by fire-clay bricks, and adding a damper (see Fig. 20, p. 280) so that communication may be shut off should the temperature rise too high.

If the vapours be condensed by means of a properly constructed condenser between the pan and the fire, no fears need be entertained.

CHOICE OF DRIER—MANGANESE DRIERS.

The preliminary study which we have made of the different driers shows that the best results are obtained by the use of lead and manganese. In particular it has been shown that an oil boiled in contact with manganese dries quicker than one boiled with lead. Further, when boiled oil is free from lead it has the advantage of not being altered by sulphur compounds.

If we go back to the receipts for the different driers to be added, we will find the employment of the borate and the hydrated oxide of manganese recommended to the exclusion of all lead compounds, and the boiling may be done as suggested.

The superiority of oils having manganese as their only drier is so decisive that attempts have been made to manufacture such oils directly. According to Andés this result may be attained in a very simple manner.

1. BORATE OF MANGANESE.

Two parts of very white borate of manganese, free from iron and finely ground, are added with constant stirring to 10 parts of oil heated to 100° C. When complete incorporation is effected the heat is raised to 200° C. One thousand parts of oil are heated in a pan until bubbles of gas commence to come off, when the preceding mixture is added in a thin stream. The heat is raised to about 200° to 220° C., and after twenty minutes' boiling an oil is obtained which rapidly dries to a bright elastic coat.

It is absolutely necessary only to use borate of manganese completely free from iron. It is prepared, according to Livache, by adding a solution of borax to a solution of chloride of manganese, so long as a precipitate is formed which is washed with water and dried. A perceptibly white product is thus obtained.

As manganese ores are apt to contain iron, lime and other impurities, this method of preparing borate of manganese is apt to give an impure product; a better method is that given on p. 312. In addition, the drying properties of this salt are such that it can transform linseed oil into a very drying oil at a temperature as low as 40° C.

If in a flask containing oil we suspend a small linen bag containing borate of manganese (3 of borate to 100 of oil), and if we place the flask in a warm place we obtain in fifteen days a quick-drying oil.

Nevertheless, we obtain a better quality oil if, instead of employing borate of manganese alone, we employ a mixture of borate of manganese and oxide of lead, which is practically the same, on account of the reaction which ensues as introducing oxide of manganese. Oxide of manganese may be added directly in the form of the hydrated oxide—a dense, brown product; finally it can be introduced by utilising reactions similar to that just indicated.

2. Hydrated Oxide of Manganese and Air.

One thousand parts of oil are heated to 70° to 80° C.; on the other hand, 3 parts of crystallised sulphate of manganese are dissolved in a very small quantity of water by the aid of heat; 10 parts of caustic potash dissolved in a small quantity of water are added; the whole well stirred and run into the oil. The mass turns brown, and brightens at the same time. An india-rubber tube, to the end of which a rosed nozzle is fixed, is introduced into the pan, and by means of a pump air is injected for four or five hours until the brown coloration has disappeared.

It has likewise been proposed to cause the oil to which the above mixture has been added to fall in drops inside a tube, where a current of air circulates the reverse way. The oil is again elevated, and made to fall again, and so on until the brown coloration disappears. It has been further proposed to use the natural black oxide of manganese—pyrolusite.

3. Black Oxide of Manganese and Sulphuric Acid.

A mixture of 2 parts of finely ground pyro-lusite and 2½ parts of sulphuric acid is added to 100 parts of linseed oil heated between 180° and 200° C. After about an hour a milk of lime, obtained by slacking 1 part of quick-lime, is added, the whole well stirred, and filtered through canvas.

4. *Various Processes.*—A very quick-drying oil whose dryer is manganese—free from lead—may be obtained in a roundabout way by slightly heating or simply agitating an oil which has been boiled with litharge, with borate or sulphate of manganese. The oxide of manganese replaces the oxide of lead, which is precipitated in the state of insoluble borate or sulphate.

Finally, a note may be made of certain processes but seldom used.

One thousand parts of linseed oil, for example, are heated with 5 per cent. of oleate of lead.

It has been proposed to replace the oleate of lead by the linoleate, or more recently by the resinate of manganese.

Zinc Driers.

In another process the oil is boiled after the addition of white lead, acetate of lead and sulphate of zinc.

The function of zinc sulphate in oil-boiling and varnish-making is obscure; it is generally added with the idea of imparting elasticity.

Again, attempts have been made to substitute lead salts by pure oxide of zinc, but after boiling the resultant oil dries slowly, and has no advantage over a litharge-boiled oil except that it is not blackened by sulphuretted hydrogen. It is much more profitable to precipitate the lead of a litharge-boiled oil by means of a suitable manganese salt such as the sulphate.

In certain cases it has been recommended to add oxide of zinc to the salts of manganese employed in oil boiling, but experience has shown that if the oxide of zinc did any good in these cases it was in consequence of the liberation of oxide of manganese. Leaving out of account the advantage —often very appreciable—of obtaining an oil which does not blacken in contact with sulphuretted hydrogen, it would appear that the oil does not dry quite so quickly as when a salt of manganese is added in presence of oxide of lead.

It has been recommended not to heat the oil with driers beyond 120° to 127° C., but to project into the oil after several hours' boiling $\frac{1}{100}$th of its weight of water, mixed with $\frac{1}{50}$th of its weight of litharge, in the form of fine drops. But this process, due to Bartky and tried in Germany, appears to be but little used.

Oil Boiling by Steam.

In boiling oil over a naked fire two very great inconveniences present themselves, *viz.*, the danger of fire and the dark tint which the oil assumes in contact with overheated surfaces. It has been tried to remedy these by boiling oil by means of a steam heat, and this method of boiling ensures perfect safety, economy and rapidity, advantages which are the more to be appreciated because this method produces bright-coloured oils.

Boiling by Steam Coil.—The most simple arrangement is to heat the oil in a pan by means of a steam coil. Steam at about 4 to 5 atmospheres is passed through the coil so as to heat the oil to about 150° C. A mechanical agitator spreads the heat uniformly through the bulk of the oil.

Suppose we are working with a pan capable of holding half a ton of oil. When the oil has been heated for half an hour the necessary quantities of driers are added, and the boiling continued for six hours, the mechanical agitator being kept at work all the time. The process is rendered more economical by using superheated steam. In this case the steam before entering the pan passes through a coil in a furnace capable of bringing it to 400° C. When the superheated steam in its passage through the coil in the pan has brought the oil to the desired temperature, it is then only necessary to maintain it at that temperature by means of the steam admission valve.

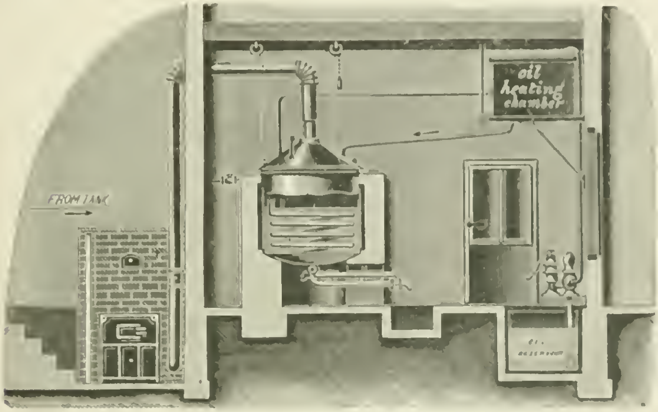

Fig. 23.—Oil Boiling by Superheated Steam Plant.

So as to render the process as economical as possible, the gases evolved from the furnace in which the steam was superheated are led underneath the pan and burnt so as to assist in heating the oil. But if this arrangement be economical, it possesses one of the disadvantages, the obviating of which we had in view when adopting the process of oil boiling by steam.

Boiling in Steam-Jacketed Pans.—In boiling oil by a steam coil there is always this serious disadvantage, and that is, there is a great difficulty in maintaining a uniform temperature throughout the mass of oil, even when continuous agitation is kept up. Effectual commingling of the oil and driers is not practicable; the latter settle to the bottom, and the coil of piping prevents the agitator from reaching them. Were it not for this drawback linseed oil could be very well boiled in a comparatively shallow, lead-lined, rectangular wooden tank lined with six or seven pound lead. This drawback, however, could be easily got over by pumping in air through a perforated pipe laid down so that each coil thereof alternated with a coil of the closed steam pipe. This, in conjunction with the mechanical agitator, would effectually prevent the drier from settling at the bottom.[1] It must likewise be borne in mind that certain manufacturers deprecate the continuous stirring of the oil as likely to produce bad results.

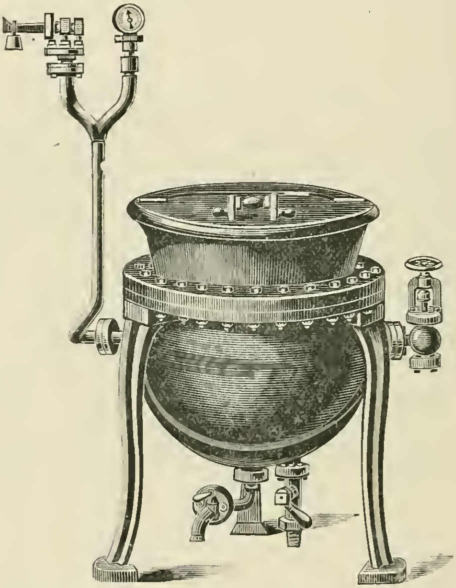

FIG. 24.—STEAM-JACKETED PAN FOR OIL BOILING, ETC., UNDER PRESSURE.

Andés obtained better results by using a steam-jacketed pan, 85 gallons capacity, made from strong iron plate, $\frac{1}{5}$

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[1] By suitable mechanical arrangements a boiling pan made in this way would be equally effectual and cost less, not only originally but also for maintenance, than the costly wrought iron or copper vessel now in vogue.

inch in thickness, and able to stand a pressure of 4 or 5 atmospheres; it is provided with a safety valve, blow-off cock, and a tap to run off condensed water.

Process.—About 40 gallons of oil are run into the pan, the steam turned on, and when the pressure is sufficient the blow-off cock is opened for a minute to allow the steam and air to escape; this is repeated several times, and the escape valve is regulated in such a manner that only the small quantity of spent steam, the replacement of which by superheated steam is necessary to keep the pan and contents at the desired regulated temperature, is allowed to escape.

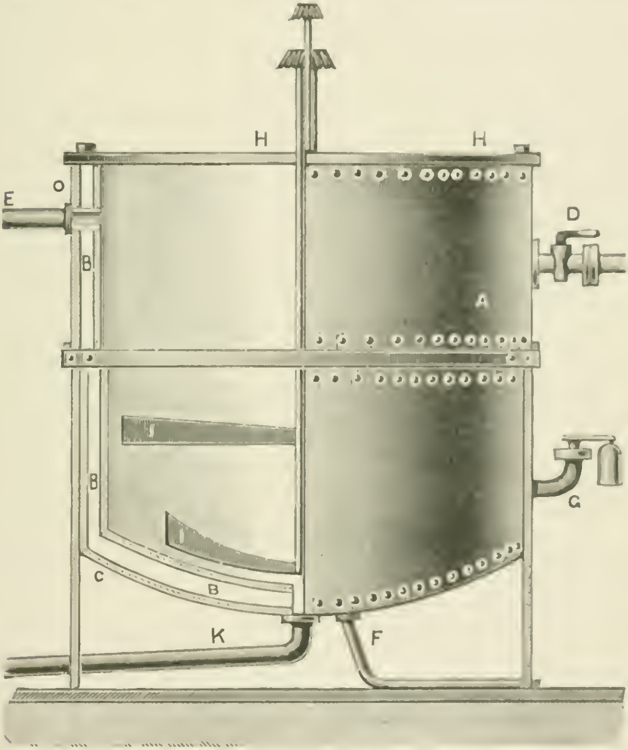

FIG. 25.—STEAM-JACKETED PAN WITH AGITATOR FOR OIL BOILING, ETC

The condensed water which forms in the beginning is run off when necessary.

The steam is regulated so as to keep the temperature between 125° and 132° C., and the process lasts from five to six hours, but by prolonging the boiling and using more driers better quality oil is obtained. If care be taken not to exceed 132° C., the resultant oil is very pale and bright—very remarkably so.

To stimulate oxidation during the boiling process, Andés fixes a mechanical agitator on the surface of the oil, which is propelled very slowly.

The same driers may be used as in boiling oil by fire; however, experience has shown that it is advantageous to alter the proportions. Only half of the quantities of lead compounds should be used, but double the quantities when working exclusively with hydrated oxide of manganese or manganese salts.

To economise, two steam pans may be wrought together, the steam passing from the steam-jacket of the one to that of the other. But it is perhaps more advisable to utilise the waste steam (as shown in Fig. 23) so as to heat in an intermediate tank the next batch of oil as it comes from the settling tanks, and previous to the actual boiling. This preliminary warning almost does away with the frothing of East Indian linseed oil, and the consequent liability of the oil to prime over into the condenser when it is heated in the jacketed pan in the usual way.

Vincent's Process for Oil Boiling by Steam.

In Vincent's process a pan, preferably of copper, is used, having a depth equal to its diameter, of two to ten tons capacity, and surrounded with a steam-jacket up to three-quarters of its depth, or to the oil level or a little above it, and capable of withstanding a working pressure of forty pounds to the square inch, into the interior of which the steam is led, as a source of heat, and so regulated as to register throughout the process a pressure within the jacket of thirty-five pounds to the square inch. The top of the pan is closed by a dome rivetted to it, and pierced with a manhole. In the centre is a stuffing-box through which pass two concentric vertical shafts—a hollow shaft encircling a solid one—armed with blades, at one half the height of the pan for one of the shafts, and at the bottom of the pan for the other. By means of exterior gearing these fans turn in opposite directions, and in so doing intersect each other, and thus by their "dashing and cutting" action cause energetic agitation of the oil and thorough incorporation therewith of the driers. From the dome the fumes are either led by a 6-inch pipe to a condenser or into the furnace. Finally, as soon as the pressure of steam on the pan registers thirty-five pounds to the square inch, air is injected under pressure by means of a pipe passing through the jacket into the bottom of the pan.

The oil to be boiled at one operation is first pumped into a large reservoir, where it is stored as long as possible; from this reservoir it is run into an intermediate tank traversed by a 1½-inch coil of iron piping through which the heated vapour from the pan passes, thereby economising expense besides facilitating the deposition of impurities. The oil thus heated to about 35° C. is pumped or run by gravitation (see Fig. 23) into the pan, and steam turned on in the jacket. As soon as the pressure marks 2 or 3 atmospheres, thirty-five pounds to the square inch, the agitators are put in motion and air pumped in. The finely pulverised drier, beaten up with a little oil to the consistency of treacle, is introduced into the pan slowly and in a thin stream through a funnel fixed in the dome as soon as the oil in the pan has become evenly and thoroughly heated through its bulk, or about half an hour after the steam indicates a pressure of thirty-five pounds. Introducing the drier in this highly comminuted condition prevents coagulation, and thus gives practical effect by complete diffusion throughout the oil to Vincent's theory of oil boiling by steam, which requires each particle of oil to be in contact with, or in proximity to, a particle of the drier used and the oxygen of the air at the same time.

Air is injected in this way so long as it is absorbed and does not cause the oil to froth up and prime over into the condenser.

It was thought at first it might be advisable to heat the air before injecting it into the pan, but this would appear to be useless, as it becomes so heated by the pressure to which it is subjected in overcoming the weight of the oil, lifting the heavy valves, etc., that the pipes through which it passes cannot be held in a firm grip in the hand, and not only so, but its passage through the oil cools the latter but very little. The process lasts four hours. By means of a 2-inch pipe in the bottom of the pan the oil is run into tanks where it deposits any undissolved driers, etc. It is through this discharge pipe that the air is injected into the oil. Injecting it in any other way leads to the injection pipe getting choked up with oil-skins, etc.

Boiling by Superheated Air.

Notwithstanding the advantages incidental to the process of oil boiling by steam, there is one drawback which has been considered so serious by some manufacturers as to lead them to give up this method. They found that even with superheated and thoroughly dried steam there was always a certain amount of moisture which acted on the metal of the pan, piercing it in holes and causing bursting or explosions.

They therefore substituted superheated air for superheated steam. This air is drawn by means of a fan from the superheater to the (steam) coil or jacket, and is again returned to the superheater, thus working in a continuous cycle. The difference between the specific heat of steam and that of air renders the economy of the hot air process very problematical. The coil in the pan is made of copper, which, although dearer than an iron one, has the advantage of not colouring the oil. As copper is also attacked by hot oil, it has been recommended to electro-plate it with silver. The coating need only be but very thin, as it undergoes no wear and tear.

RULES FOR, AND CHANGES WHICH TAKE PLACE IN, OIL BOILING.

If we wish to know what goes on during oil boiling we must pay great attention to three factors—the amount of driers, the temperature of boiling, and the length of time occupied in the process.

When working with naked fire, the quantity of drier varies from 2 to 4 per cent. of lead compounds and 1 to 2 per cent. of hydrated oxide of manganese. If we examine the deposit which forms at the bottom of the pan we find it to consist of a viscous mud, and when a drier with a lead base has been used, we also find a notable quantity of *metallic* lead, whilst the boiled oil only contains about 1 per cent. of *oxide* of lead. We may therefore conclude—firstly, that the oxidisable principles of the oil have reduced a certain quantity of oxide of lead; secondly, that a certain quantity of oxide of lead is thrown down in the form of lead salts insoluble in oil; thirdly, that a part of the oxide of lead enters into a soluble combination with the oil; this combination takes place by the simple saponification of certain glycerides of the oil, and consequently a corresponding quantity of glycerine is liberated. These different reactions are set up from the commencement at a moderate temperature.

In the case where manganese is employed instead of lead, the boiled oil contains about ½ per cent. of oxide of manganese.

Whilst studying the temperature in the different processes of oil boiling which we have examined, we have seen that the temperature ought to be regulated between 210° and 228° C. so as to ensure a boiled oil of good quality; and it is very easy to convince any one that this is the case. On the one hand, experience shows that an oil when heated commences to give off volatile products at about 230° to 236° C., with the formation of fatty acids which yield oxidation products of inferior quality, requiring longer time to dry. When it is required to produce a limpid oil with but little body (which is not always the case; take printers' ink, for example), it is advisable not to go beyond 230° C. On the other hand, as a consequence of the saponification of a small quantity of oil a corresponding quantity of glycerine is liberated; now if this glycerine remained, in the free state, in the oil the oxidation products would never dry perfectly but remain tacky. The glycerine must therefore he eliminated; this result is obtained by prolonged boiling, for the glycerine is partially carried away by the volatilisation of certain principles of the oil, which are given off in small proportion during the whole process, however carefully regulated, and even although the temperature remains lower than the temperature of distillation of glycerine.

Another portion of the glycerine decomposes under the action of prolonged heat, either as Hartley has shown in presence of metallic lead, as found at the end of the operation, or into volatile products such as acrolein, acrylic, formic, or acetic acids, and finally the larger quantity reacts upon the triglycerides of the oil forming diglycerides which afterwards oxidise and dry perfectly. Now this latter reaction occurs at 200° C. As a consequence of these facts it follows that to get good results the temperature should not be allowed to get below 200° C. nor rise to 230° C. In actual practice a temperature of 220° C. and 228° C. is aimed at.

Finally, the process lasts from three to six hours according to the size and shape of the pan, and the manner in which the furnace is built on which the pan is heated. The oil ought to be boiled long enough for the above reactions to take place, but, on the other hand, it is very certain that the boiled oil obtained as a final result will be better in quality the longer it has been boiled, for on prolonged boiling a polymerisation of the glycerides takes place, giving a final product which oxidises more rapidly and is very elastic after drying.

According to Livache, this is the reason why in England, instead of boiling rapidly so as to "crack" the oil as in France, the operation is carried on very slowly at the lowest possible temperature, the boiling proceeding for several days, sometimes for even a week.

When the oil is boiled by superheated steam, or superheated air, the temperature is regulated between 125° and 130° C. When working with steam, the latter is superheated to 4 or 5 atmospheres, relaxing in the steam coil or jacket to about 2 atmospheres. It will be readily understood, looking at the matter from the double point of view of safety and economy, how difficult it would be to superheat the steam to such a temperature that the steam in the coil or the jacket would be at 200° C., because it would be necessary to heat the oil to 15 atmospheres. Consequently the liberated glycerine remains to a larger extent in the oil, and thus prevents it from drying so well as fire-boiled oil. However, as the oil boiled in this way has the advantage of being very pale, endeavours have been made to get over the difficulty, either by using a larger proportion of manganese drier, so as to have a more energetic oxidation and thereby a splitting up of the glycerine, or, preferably, by producing direct oxidation by a current of hot air, or finally, by causing a current of air to circulate through the whole mass of heated oil; or by projecting on the surface of the oil a small quantity of water so as to carry away the glycerine in its train. However this may be, it is most likely that we have here the reason why many persons in the trade prefer oil boiled by fire-heat. Nevertheless, prolonged boiling by steam diminishes and even does away altogether with this disadvantage.

CLARIFICATION OF BOILED OIL.

When the boiling is finished the oil contains suspended solid matter—coagulated or carbonised organic matter, insoluble salts, undecomposed or unreduced drier, etc. In order to hasten deposition the oil ought to be left to rest as hot as possible, because it is then more fluid and limpid, thus facilitating the more rapid deposition of extraneous matter.

According to Andés, when working with small pans which can be easily handled, all that has to be done is to lift them off the fire and to let them stand for eight days at least. On the other hand, when working with large-sized pans built in masonry, they must be emptied quickly, either by a ladle or by a pump. as hot as possible, into a reservoir of the same size, where it is left to deposit for a fortnight at least.

After standing for this time the decanted oil may be sent into the market. If stored too long before being used, the oil, at first limpid, becomes cloudy, or at least dull, when knocked about in moving or transported in barrels. This is due to chemical changes, which supervene slowly throughout the mass. All that has to be done is to allow the oil to stand for a day or two after it has reached its destination, and to decant cautiously. The amount of deposit formed in this way should not exceed 1 to 2 per cent. of the oil.

Some oil boilers have been known to fill the boiled oil into drums for despatch abroad after scarcely twenty-four hours settling. It need scarcely be remarked that goods made in this way are liable to be returned.

When cotton-seed oil is cheaper than linseed oil it is sometimes attempted to boil it in the proportion of 10 to 20 per cent. with linseed oil. An oil produced in this way will never clarify, leaving altogether out of account its diminished drying properties.

The very prolonged storing of *boiled* oil, far from improving it, deteriorates it, especially in the case of lead-boiled oil. After from five to ten years the oil separates into two fractions, the one solid and brittle, whilst the other, retaining its fluidity, can still be used. Oils containing manganese alone do not alter in this way.

The deposit formed during oil boiling varies, according to the quality of the oil and the boiling process adopted, from 5 to 8 per cent. of the quantity of oil treated. It is of medium consistency, and, according to the quality of the oil, the colour varies from white to yellow. Andés states that impure, bad quality oil gives a white or yellow deposit, whilst well-purified good oil yields a brown, with no granular or crystalline appearance.

The loss in oil boiling and the cost may be compensated by adding 10 per cent. of thick rosin oil to the raw oil before boiling. This percentage does not affect the finished oil to any extent, if at all.

If the boiling process has been well conducted, *i.e.*, at a temperature not exceeding 220° C., this deposit constitutes the only loss. It is carefully collected, being worth sometimes as much as 50 to 60 per cent. of the oil used. It finds a use in the manufacture of mastic (cements), soap and low-quality paints, especially blacks.

PROPERTIES OF BOILED OIL.

Colour and Fluidity.—If we take linseed oil as the type of a drying oil, it ought after boiling to be a little less fluid than the raw oil, and vary in colour from pale yellow to deep yellow, or, at the furthest, reddish brown.

Smell.—The smell, without being pleasant, should not be nauseous.

Taste.—Taste similar to that of the raw oil, bringing to mind the vapours given off during boiling. A pungent bitter after taste on the back part of the palate indicates rosin oil.

Density.—According to Livache, an oil marking 29° with the Fisher's and Bex's oleometer, equal to a density of ·9325, will register on the other hand, after being well boiled with lead, 24°, equal to a density of ·9433, or after boiling with manganese 26°, equal to a density of ·9389.

But the density of a very good boiled oil made on the large scale in England according to Vincent's process, and drying to a non-tacky, firm, elastic, durable coating in six or seven hours and containing 10 per cent. of rosin oil, but no other impurity, has always been found to have a density not exceeding ·934.

Driers : Detection of.—To detect in a boiled oil the substances used as driers a small quantity of the oil is poured into a test tube and shaken with dilute sulphuric acid. A white precipitate becoming black on the addition of sulphuretted hydrogen indicates lead.

If no precipitation takes place, and if the sulphuric acid becomes green, blackening by the addition of sulphuretted hydrogen, the oil contains copper compounds.

When sulphuric acid gives no precipitate, if the acid be neutralised by ammonia on adding ammonium sulphide, iron gives a black, manganese a flesh, and zinc a white-coloured precipitate ; further, in the case of zinc, the acid solution gives a white precipitate with ammonia soluble in excess.

If, instead of using ammonia, we neutralise with carbonate of soda, iron yields a green precipitate, blackening in the air, and the original solution becomes blue on the addition of yellow prussiate of potash ; manganese gives a white precipitate blackening in the air.

Ignition Method.—The better plan is, however, to burn the oil with the usual precautions observed in the ash determination of vegetable substances, and after weighing the residue on ignition to make a systematic qualitative and then a quantitative examination thereof. Zinc oxide and salts are, however, liable to volatilise and escape recognition.

MANUFACTURE AND PROPERTIES OF DRIERS.

The principal substances used as "driers" or aids in the process of oil boiling are oxides and salts of lead and manganese.

LEAD COMPOUNDS USED AS DRIERS:—[1]

1. Litharge.
2. Red lead.
3. Acetate or sugar of lead.
4. Lead borate.
5. Lead linoleate.
6. Lead resinate.

LITHARGE.

Manufacture.—When metallic lead is heated in a current of air, it oxidises, and becomes converted into massicot PbO. 207 parts of lead yield 223 of litharge. When this is further heated, it fuses, and is known as litharge, or, from its scaly nature, as flake litharge. It does not, however, undergo any chemical change. When buying litharge for use as a drier, it should be guaranteed free from copper, which is often present in considerable quantity, and acts injuriously.

Properties.—Litharge should dissolve completely in dilute nitric acid and in acetic acid to a colourless solution. A green coloration would indicate copper. Any residue will be found to consist, most probably, of grit, acetic acid may leave a residue of non-oxidised blue lead. When litharge is heated with linseed oil for some time at the temperature at which that oil is said to "boil," but, more properly, at which it undergoes destructive distillation, it dissolves in the oil. According to practical varnish-makers, it dissolves at a temperature considerably below this point. In fact, in varnish-making and oil-boiling it is often found at the close of the operation reduced to metallic lead. In such cases very probably all its

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[1] Galen states that "white lead and litharge thicken and dry".—*De Meth. Med.*, iii., 4.

oxygen is seized hold of and absorbed by the "boiling" oil. Litharge assists in oil-boiling in three different ways:—

1. It may combine with any free linoleic acid in the oil at a comparatively low temperature to form linoleate of lead, which will dissolve in the oil as soon as it forms.

2. At a higher temperature it will eliminate glycerine from the oil and combine with the liberated linoleic acid to form linoleate of lead, which will again dissolve in the oil as soon as formed.

3. In the act of being reduced to metallic lead, it will part with its oxygen to the oil, and thus assist in its oxidation, and consequently increase the drying properties of the oil.

The drawback attendant on the use of litharge and all lead salts as driers is the dark colour which they impart to boiled oil, due most probably to the dissolved lead linoleate. This dark colour is intensified by the high temperature to which the oil must be brought, and kept at for some considerable time, before the litharge dissolves and plays its part.

Another drawback attendant on the use of litharge and other lead salts is the fact that the oil or varnish into which they enter cannot be used in conjunction with, or in juxtaposition to, pigments consisting of or containing sulphides liable to combine with lead compounds to form the black sulphide of lead. Sulphur emanations act similarly. Paintings or decorations injured in this way may be revivified by washing with a weak solution of peroxide of hydrogen, which converts the black sulphide of lead into white sulphate of lead without materially affecting the painting in any other way. This black coloration is very often due to the free sulphur contained in the pigment, and not to the combined sulphur, which, looked at from a rational point of view, must be regarded as more or less stable. But, on the other hand, linseed oil boiled over the naked fire with litharge gives an elastic, durable coat of greater lustre and less susceptible to dry superficially than manganese boiled oil. In a word, litharge boiled oil is more of the nature of a varnish than manganese boiled oil.

RED LEAD MINIUM.

Manufacture.—Red lead Pb_3O_4 is made by heating litharge in contact with air at 300° C., a temperature slightly below its point of fusion. The litharge should be free from copper. It gradually becomes converted into a fine red known as red lead.[1]

Composition.—The red leads of commerce differ in composition according to the length of time they have been furnaced. However, the proportion of oxygen fixed by the litharge never exceeds that which corresponds to the formula $2\,PbO, PbO_2 = Pb_3O_4$.

Properties.—When red lead is heated it darkens, and then, at a temperature above 300° C., it gives off oxygen, becoming reconverted into litharge.

Test for Purity.—When red lead is treated with nitric acid the two equivalents of PbO are dissolved as nitrate of lead, leaving the PbO_2 behind as a puce coloured residue which is perfectly insoluble in nitric acid, and only dissolves in dilute nitric acid after it has been reduced by oxalic acid. When this reagent is introduced into the test tube containing the nitric acid and red lead vigorous action ensues, the whole of the puce coloured oxide being converted into white crystalline nitrate, to which it is only necessary to add a little boiling water for complete and instantaneous solution.

Adulteration.—Any residue remaining is generally barytes. Sophistication with ground brick is only met with in books.

Red lead, owing to its containing more oxygen perhaps in a more available state, may be regarded as a more energetic drier than litharge.

Other Uses.—Besides its use as a drier, red lead is used in

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[1] A variety of red lead, made by igniting white lead, is sold as orange lead. Although dearer in price it is no more efficacious as a drier than ordinary red lead.

the manufacture of wall paper, sealing wax, crystal glass; mixed with white lead in oil it is used to form steam joints.

Red lead "substitutes" are of no use as driers.

LEAD ACETATE (NEUTRAL) Pb $(C_2H_3O_2)_2 + 3\ H_2O$.

SUGAR OF LEAD. *Molecular Weight*, 379.

Manufacture.—This salt was formerly known as *salt of Saturn*, or sugar of Saturn. It is prepared by neutralising acetic acid with litharge.

Properties.—It crystallises in oblique rhomboidal prisms, colourless, transparent and efflorescent. It is generally met with in the form of heavy compact crystalline masses, somewhat resembling loaf-sugar. Its odour is acetous, whilst its taste is both sweet and astringent, its after taste is bitter and disagreeable.

Solubility.—It dissolves in ½ part of cold water and in 8 parts of alcohol. Its solution slightly reddens blue litmus paper, and is partially decomposed by carbonic acid, which at the same time liberates a small quantity of acetic acid, which preserves the rest of the salt from the action of carbonic acid.

Action of heat.—Crystallised acetate of lead melts at 75·5° C., at 100° C. it loses water and a small quantity of acetic acid. It afterwards solidifies, but towards 280° C. it again melts. The dehydrated salt would appear to consist of a sesquibasic acetate. At higher temperatures it decomposes with disengagement of carbonic acid, acetic acid and acetone (see solvents), leaving a residue of very finely divided and highly combustible metallic lead. It is a very energetic drier; perhaps a deal of the metallic lead found by varnish-makers at the bottom of their pots may come from the complete decomposition of the acetate and not from the litharge often used in conjunction with it.

BASIC LEAD ACETATE.

The so-called neutral lead acetate results from the combination of two equivalents, *i.e.*, 120 parts of monobasic acetic acid with one equivalent, *i.e.*, 223 parts of the di-acid base litharge. When *two* equivalents of a *monobasic* acid combine with *one* equivalent of a *di-acid* base a neutral or normal salt is produced.

Manufacture.—But it is found in actual practice that certain neutral or normal salts, in their state of solution in water, can still dissolve and combine with a further proportion of base. Thus when one equivalent of neutral acetate of lead (p. 306) is dissolved in water, it combines on boiling with an additional equivalent of litharge to form the di-basic salt, $PbA_2Pb(HO)_2$. By digesting together one equivalent of sugar of lead with two equivalents of litharge the tribasic salt, $Pb(A)_2\ 2Pb(HO)_2$, is produced.

Properties.—These basic acetates, owing to the fact that their excess of litharge, or, rather, lead hydrate PbO,H_2O, is in a loose state of combination, easily give up this excess to weak acids, even carbonic acid, as exemplified in the manufacture of white lead. Should a raw linseed oil contain free linoleic acid, agitation with a solution of basic acetate of lead will convert the former into linoleate of lead, and if the agitation be renewed from time to time this linoleate of lead will dissolve in the oil imparting drying properties thereto, whilst the insoluble magma produced by the action of the basic acetate on the colouring matter and mucilage collects as an insoluble slime at the bottom of the vessel. Such is the principle of the refining of linseed oil by Liebig's method and the imparting of drying properties thereto in the cold by means of basic acetate of lead, a substance the utility of which is not appreciated either by the varnish-maker or colour-maker to the extent to which its intrinsic merits entitle it.

BORATE OF LEAD.

Borate of lead is a white substance made by precipitating the boracic acid in 382 parts (*i.e.*, one equivalent) of borax $Na_2B_4O_7 + 10$ aq. by one equivalent of a lead salt—that is to say, 331 parts of nitrate of lead $(PbNO_3)^2$ or 379 parts of neutral acetate of lead $Pb(C_2H_3O)_2 + 3$ aq. The mother liquor is filtered off the precipitate, well washed, filtered, pressed and dried in the usual way. This substance combines the drying properties of both lead and boracic acid. It does not darken the oil to quite the same extent as other lead driers.

Resinate of Lead.

The first stage in the manufacture of resinate of lead is the preparation of an alkaline solution of resinate of soda. Rosin being an acid body of variable degrees of acidity, different samples require different proportions of caustic alkali for exact neutralisation. In actual practice the best plan is to add rosin gradually and with constant stirring to a very dilute boiling solution of caustic alkali of known strength until the latter is exactly neutralised, which happens when the solution refuses to dissolve or take up any more rosin.

Starting from the data that every 62 parts of anhydrous sodium oxide (real alkali) present in solution as resinate of soda require for complete precipitation one equivalent of a lead salt, *viz.*, 379 parts lead acetate or 330 parts lead nitrate for complete precipitation, and knowing the original bulk of the alkaline solution used and its richness in real alkali, as determined by the hydrometer and the usual tables, it is easy to calculate the amount of lead salt necessary to displace the soda in the solution of alkaline resinate by lead, and thus throw down all the rosin as resinate of lead.

The dilute solution of the proper proportion of lead salt is gradually added with constant stirring to the equally dilute solution of alkaline resinate; the mother liquor is syphoned off the precipitated lead resinate, which is further well washed, drained and dried.

Linoleate of Lead.

Linoleate of lead is made by precipitating a solution of linseed oil soft soap. The soft soap is made by saponifying linseed oil with caustic potash in the usual way. 100 lbs. of linseed oil on an average require 19½ lbs. of pure caustic potash, equal to 16½ lbs. of anhydrous potassic oxide, for complete saponification.

Caustic potash is sold as containing so much per cent. of anhydrous caustic alkali; when pure it contains 83·93 per cent. It is, therefore, a mere matter of calculation if 100 lbs. of linseed oil require 19½ lbs. of caustic potash, when the strength is 83·93 per cent., to find how much will be required for complete saponification when the strength is 80 (or any other figure) per cent.

The soap is dissolved in five or six times its weight of water, and the linoleic acid precipitated therefrom by a dilute solution of a lead salt. The exact proportion of lead salt for complete precipitation depends upon the amount and the strength of the caustic alkali used. 112·2 parts of pure caustic potash, equal to 94·2 parts of anhydrous potassic oxide, require one equivalent of a lead salt, say 379 parts lead acetate or 330 parts lead nitrate. The lead salt solution is added gradually to the soft soap solution with constant stirring. The mother liquor is syphoned off the precipitated lead linoleate, which is then further washed, drained and dried.

MANGANESE DRIERS.

The manganese compounds used as driers are:—

1. Black oxide—MnO_2.
2. Manganese sulphate—$MnSO_4$.
3. Manganese acetate—$Mn(C_2H_3O_2)_2$.
4. Manganese borate.
5. Manganese oxalate.
6. Manganese linoleate—MnL.
7. Manganese resinate—MnR.

Metallic manganese is a reddish-grey coloured brittle metal, difficultly obtained from its naturally occurring oxides by heating strongly with charcoal. Finely divided metallic manganese has such affinity for oxygen that it decomposes water at the ordinary temperature, with evolution of hydrogen gas.

In order to preserve it in an unoxidised condition it must be preserved in an hermetically-sealed tube or under naphtha.

This strong affinity of metallic manganese, as well as its compounds, for oxygen constitutes in great part the value of manganese ores for commercial purposes. This value is further greatly enhanced by the fact that the oxidised compounds of manganese, under certain circumstances, easily part with a portion of their oxygen, becoming converted into lower oxides, and these lower oxides, in contact with the air, reabsorb oxygen, and are reconverted into the original higher oxides. The drying properties which manganese oxides and salts impart to oils boiled in contact with them is due to the fact of these oxides and salts being so easily deoxidised to a certain extent, and then just as easily reoxidised—*e.g.*,

$$3\,MnO_2 = Mn_3O_4 + O_2, \text{ and again}$$
$$Mn_3O_4 + O_2 = 3\,MnO_2.$$

BLACK OXIDE OF MANGANESE PYROLUSITE.

This is the source from which the various salts of manganese are prepared. Not only is it the most important, but also the most widely distributed of manganese minerals. It usually occurs in heavy compact masses, reniform, and sometimes with a fibrous or radiated structure. The colour is iron black to dark steel grey, and very often soils the fingers. It gives with unglazed porcelain a grey streak. The other manganese minerals give a brown streak. When heated alone it liberates considerable quantities of oxygen, and becomes converted into trimanganic tetroxide $Mn3O_4$. Pure pyrolusite has a specific gravity of 4·9.

In commerce pyrolusite is usually found as a coarse, dull, black powder, consisting of a mixture of manganese dioxide, sesquioxide and monoxide, and contaminated with the gangue, quartz, felspar, limestone, etc., which often amount to 40 or 50 per cent.

Besides being used directly as a drier, pyrolusite—*savon des verriers*—is extensively used in glass manufacture to burn up any carbonaceous matter and at the same time impart a slight violet tint, which masks the usual yellow tint of glass.

SULPHATE OF MANGANESE.

This beautiful rose-coloured salt is prepared on the large scale by heating pyrolusite with coal and dissolving the impure monoxides, to which it has been reduced, in sulphuric acid. A little muriatic acid is added towards the end of the operation.

The solution thus obtained is evaporated to dryness and afterwards exposed to a red heat, so as to decompose any sulphate of iron.

The pure sulphate is filtered and drained from the insoluble oxide of iron.

It crystallises with :—

7 H_2O below 6° C.
6 H_2O between 7° and 20° C.
3 to 4 H_2O at higher temperatures.
1 H_2O at 200° C.

The monohydrated salt is greyish-white in colour, and is the starting-point of the manufacture of all manganese driers.

MANGANESE ACETATE.

This salt is manufactured by doubled decomposition between acetate of lime and sulphate of manganese in equivalent proportions.

The reaction is as follows :—

$$Ca(A)_2 + MnSO_4 = CaSO_4 + Mn(A)_2.$$

The brown acetate of lime may be used. A solution of acetate of manganese marking 22° Twaddell, specific gravity 1·110, is used in oil-boiling by steam in the proportion of 36 gallons to 10 tons of oil in conjunction with a small quantity of lead driers, the greater the proportion of which the darker is the resultant boiled oil.

MANGANESE BORATE.

This is prepared by precipitating a solution, 382 parts, *i.e.*, one equivalent of borax, $Na_2B_4O_7$ 10 aq., with 169 parts, *i.e.*, one equivalent of the monohydrated sulphate of manganese, $MnSO_4H_2O$. The sulphate of manganese should be free from iron and the borax from excess of alkali. The iron may be removed by roasting the sulphate and redissolving. The excess of alkali may be neutralised by the addition of the requisite quantity of boracic acid. Precipitation should be effected in the cold. This substance, when pure and skilfully prepared, is a perfectly white powder, but whiteness is not a guarantee of purity, as it is often most grossly sophisticated with sulphate of lime, etc.

MANGANESE OXALATE.

Manganese oxalate is made by precipitating a solution of oxalate of soda by a manganese salt. In boiling the oxalic acid of the manganese oxalate is decomposed with evolution of CO_2, etc. Very energetic oxidising properties are claimed for the residual manganese.

MANGANESE LINOLEATE.

Manganese linoleate is made in the same way as lead linoleate. A linseed oil soft soap is precipitated by a solution of the monohydrated sulphate of manganese, 169 parts of which, or one equivalent, are equal to 112·2 parts, or two equivalents of pure caustic potash which contain 83·3 parts of anhydrous potassic oxide (the percentage of real alkali contained in pure caustic potash), and as 100 parts of linseed oil require 19½ parts of pure caustic potash for complete saponification, it is easy to make the necessary calculation when working with potash of any strength different from the pure, *viz.*, 83·3 per cent. of real alkali. Or potash ley of a certain gravity may be kept in stock, and its richness in real alkali ascertained from the customary tables and the gravity as indicated by the hydrometer. For 100 lbs. of linseed oil caustic potash equal to 16⅓ lbs. of real alkali must be used, starting initially with a weak solution and finishing with a more concentrated. The soft soap of commerce contains about 50 per cent. of water, and though often made from linseed oil is not invariably so, and, moreover, it is grossly adulterated. The varnish-maker, therefore, who makes his own driers, should, therefore, make also his own soft soap.

Manganese linoleate is precipitated, washed, pressed and dried in the same way as lead linoleate.[1]

RESINATE OF MANGANESE.

Resinate of manganese is made in the same way as lead resinate; for every 80 parts of pure caustic soda (containing 62 parts of pure anhydrous alkali sodium oxide) present in the solution of resinate of soda 169 parts of monohydrated sulphate of manganese are required for precipitation.

TREATMENT OF LINSEED OIL WITH DRIERS IN THE COLD.

Numerous attempts have been made to prepare quick-drying oils without the intervention of heat, the great inducement being economy and a paler oil. Few processes have, however, been published.

The simplest plan consists in passing the oil into a reservoir, from whence it falls upon plates superimposed at alternating inclinations and at certain distances one from another. The oil thus runs in a thin layer in a zigzag course from one plate to another and thickens as it absorbs oxygen; by

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[1] The oleate or the ricinoleate may be made in a similar manner.

using lead plates the action is more rapid. The oil when it reaches the bottom of the column of plates is pumped up to the top, and the same process gone through again, and so on until the oil has assumed the requisite consistency.

1. Bink's Method.

In Bink's process a very small quantity of drier (2 to 6 parts in the thousand), consisting of a mixture of oxide of manganese and a salt of lead is first added to the oil, then a current of air is passed through the mass until the desired quantity of oxygen is absorbed. But this process does not give satisfactory results without the aid of a moderate heat.

Bouis' Process.—Bouis proposed to introduce oleate of lead into the oil: the product is colourless and dries well enough. It has been proposed later to replace the oleate by the linoleate of lead, but the latter does not give the same result as it is insoluble in oil in the cold. Hartley and Blenkinsop dissolve the linoleate in naphtha and introduce it in solution in that vehicle into the oil and then blow air through it.

2. Liebig's Method.

Liebig recommended the agitation of the oil with water, litharge and basic acetate of lead. The latter salt is prepared by grinding as finely as possible 1 part of acetate of lead with 1 part of litharge to thorough incorporation, and placing the mixture in a porcelain basin, which is heated on the water bath and covered with another porcelain basin to prevent access of air. After an hour's heating a white mass is obtained, which is triturated with 5 parts of water. The solution on standing and clarifying contains the basic acetate of lead. It is diluted with its own bulk of water, and vigorously shaken with 20 parts of oil which has been triturated with 1 part of finely ground litharge. The mixture thus obtained is added to the oil to be treated, which it soon decolorises, and at the same time stimulates its drying properties. In those cases where the presence of lead would be prejudicial to the object for which the oil is to be used, it is agitated with a small quantity of sulphuric acid diluted with 3 parts water. The lead may also be eliminated by agitating the oil with a salt of manganese, the acid of which forms an insoluble lead salt—the sulphate, for example, or, better, the borate.

3. LIVACHE'S PROCESS.

Livache's process consists in agitating the oil in the cold with finely divided metallic lead, perfectly free from oxide. For this purpose he uses the spongy lead obtained by precipitating a lead solution by zinc plates.

For 1000 gallons of oil the proportions and process are as follows: 30 lbs. nitrate of lead are dissolved in 15 gallons of water; about 1 ounce of nitric acid is added, then 6 lbs. of metallic zinc in plates. The precipitated lead is placed in capacious funnels plugged with shavings, or, better still, with sea-weed, where it is rapidly washed with water; then a small quantity of oil is poured on very gently, so as to displace the water imbibed by the porous mass of precipitated spongy lead. When the oil runs away clear and limpid from the bottom of the funnel, and as a consequence thereof all the water has been displaced, the mud thus obtained is run into the tank containing the 1000 gallons of oil, where the whole is subjected to agitation. In working with small quantities the requisite agitation may be imparted by running the oil and lead into a cask, and rolling it about from time to time on the floor. The oil thus treated assumes a reddish tint, which disappears as soon as it oxidises in contact with air.

If the treatment has been efficient, the resulting oil is as fluid as the original, contains a small quantity of lead, and dries in twenty-four hours in the open air.

It is advisable to introduce manganese into the oil prepared in this way. Accordingly, a salt of manganese very soluble in oil, *viz.*, the nitrate, is added, say, 15 lbs. for the proportions given, and the whole frequently stirred for two or three days, after which it is allowed to stand and decanted.

If this oil were at once used, as it often contains an excess of nitrate of manganese, which deliquesces in the air, it might dry dull. To obviate this 7½ lbs. of dry precipitated oxide of lead is added, and the whole again subjected to agitation. After standing a perfectly clear oil is obtained, which, exposed to the open air in a thin layer, at the ordinary temperature dries in six hours.

It is necessary to state that during drying the absorption of oxygen produces, in consequence of the presence of oxide of manganese, a brown coloration, but the latter soon disappears, and finally a beautifully brilliant, perfectly dry and completely colourless coat is obtained.

Some manufacturers using this process have found the oil in certain cases to be tacky, perhaps owing to the presence of a small quantity of glycerine. This has led them to heat the oil treated first with finely divided lead and then with a salt of manganese, and it would appear that they have obtained very interesting results.

Employment of Boiled Oil in Painting.

Boiled oil or oil prepared as just indicated is a very important factor in painting. In fact, in house decoration raw linseed oil is the vehicle used in painting. It is ground along with *white*, that is to say, white lead, so as to obtain a soft paste, which is thinned down in various proportions with linseed oil and spirits of turpentine until the desired fluidity is obtained. If a certain shade is desired, the colouring matter beaten up with a small quantity of oil is added; finally, if very rapid drying is required, what is known as "Patent Driers" (siccative) are added. These may consist of (1) a solid powder (litharge, red lead or zumatic, *i.e.*, a mixture of oxide of zinc and borate of manganese); (2) litharge or manganese boiled oil when there is no reason to fear the painting, or by a drying oil the drying properties of which have been stimulated by appropriate treatment (precipitated lead, acetate of lead, etc.); or (3), finally, of special preparations the base of which is oil known under the name of liquid driers, but which are only used with coloured pigments, as they turn white tints yellow.

The paint thus formed slowly changes when spread upon any surface in a thin layer to a solid body, which forms a protective coating against external influences.

The paint ought not to dry too quickly, for the slower it dries the more it is capable of expanding, and thus lasting the longer.

The fluidity ought to be such that the paint flows easily from the brush whilst at the same time it does not run in streams, but forms a homogeneous coating. The coating must also be thin enough to prevent the formation of pellicles at the surface, which would hinder the part underneath from drying. It is therefore advisable to apply several successive coats. But each of these ought to be allowed to dry thoroughly before the succeeding one is applied.

A brilliant coating is not aimed at, the question of appearance being subordinate to that of resistance and durability. It is preferable to impart brilliancy by a final coat of varnish.

Patent Driers.

Concentrated Driers and Liquid Driers.

Concentrated driers consist of the product obtained by heating linseed oil at 250° to 300° C. to the consistency of sticking plaster, with a quantity of litharge, red lead, borate of manganese greater than 10 per cent. and even as high as 70 per cent. of the weight of the oil. Sugar of lead, oxide of zinc, etc., are added to or partially substituted for the preceding substances.

Concentrated driers are met with in the form of a thick viscous mass, generally of a deep brown colour.

A good product may be made by boiling in a small pot 7 parts of good and aged linseed oil—old tanked oil—with a mixture of 2 parts of litharge in powder and 2 parts of red lead. The oil at first assumes a beautiful red colour, which turns brownish as the temperature rises. Suddenly it thickens, looking like bronze in fusion, whilst at the same time abundant fumes are given off. A sample placed on a glass plate becomes in a minute perfectly solid, with no viscosity whatever.

Concentrated driers are used to make varnishes in the cold from raw linseed oil.

Liquid driers are made like concentrated driers, but when the pot is taken off the fire it is only allowed to cool for a few minutes, when it is thinned down with successive additions of spirits of turpentine, with constant stirring. It is then passed through coarse linen, and stored in closed vessels.

Liquid driers consist therefore of linseed oil saponified and oxidised to a greater or less extent, and afterwards dissolved in spirits of turpentine.

Numerous recipes have been given for the manufacture of liquid driers. It will be enough to indicate the following due to Andés :—

Concentrated Driers.

7 parts linseed oil.
2 parts litharge.
2 parts black oxide of manganese.
1 part red lead.
16 parts spirits of turpentine.

7 parts linseed oil.
2 parts sugar of lead.
2 parts red lead.
16 parts spirits of turpentine.

7 parts linseed oil.
1 part umber.
1 part litharge.
1 part oxide of manganese.
1 part sugar of lead.
16 parts spirits of turpentine.

Pale Liquid Driers.

7 parts of linseed oil.
3 parts pure dry white lead.
1½ parts sugar of lead.
16 parts spirits of turpentine.

7 parts of linseed oil.
2 parts borate of manganese.
2 parts zinc white.
16 parts spirits of turpentine.

7 parts linseed oil.
2 parts borate of manganese.
1½ parts acetate of lead.
16 parts spirits of turpentine.

In the manufacture of white liquid driers the mass does not become red on boiling, but white at first, and afterwards a very faint yellow.

Liquid driers are but little used for white colours—solid driers are preferred—but with other coloured paints very rapid drying is effected by simply adding, say, a few parts in 100 of liquid driers to the raw oil used. Here we have a case of oxidation analogous to that pointed out by Chevreul, *viz.*, when a manganese-boiled oil is added to raw oil.

Endeavours have been made by working in the cold to produce very pale liquid driers. One which is used very extensively is made by intimately mixing 100 parts of finely pulverised sugar of lead with 1200 parts of poppy-seed oil. This mixture is exposed to sunlight in a glass vessel, and frequently stirred. There is obtained in this way a perfectly colourless oil, which, when thinned down with 250 parts of spirits of turpentine, dries very rapidly, yielding a solid, durable coating.

Hartley and Blenkinsop's drier consists of zinc oxide mixed with a solution of linoleate of manganese dissolved in naphtha. One per cent. of manganese linoleate added to linseed oil in dilute solution renders it quick drying.

The so-called "terebine" very often consists simply of japanners' gold size thinned down with spirits of turpentine. Another process consists in fusing 2 parts of Manilla copal, which is then mixed with ½ part of linseed oil and 6 parts of concentrated driers made by one of the formulæ given above, after which the whole is incorporated with 14 parts of spirits of turpentine.

The bulk of the paste "patent driers" used in the trade consist of 90 per cent. or more of a mixture consisting of equal quantities of chalk and barytes and about 10 per cent. or less of actual driers.

When it is desired to prevent the paint from cracking, very good results are obtained by using spirits of turpentine, which has simply been shaken up with litharge and decanted. A liquid is thus obtained which does not affect the colour, and which also gives a very durable coating.

Driers with a Rosin Base.—Filsinger has proposed to replace oil driers by a mixture which approaches them very closely in their result. The principal ingredient is a rosin soap, prepared as follows: In a copper pan 50 parts of caustic soda are dissolved in 150 parts of water, and brought to the boil, then 100 parts of finely ground rosin are added, with constant stirring, and the liquid heated until limpid. The whole is allowed to cool, and the solution decanted from the resin soap in the bottom. This soap is dissolved in a small quantity of water, to which has been added a little sal ammoniac. The pigments are ground with this mixture. A paint is thus obtained which dries quickly, and which will take a coat of varnish, but if the process is economical and the appearance pleasant, the durability on the other hand is but very poor and the rationale of the process is very crude.

THICKENING OF LINSEED OIL BY OXIDATION.

According to Livache.—Whatever process we may adopt in the boiling of oil, the consistency or body of the resultant product is but little different from that of the raw oil.[1] Now, in many cases it is very advantageous to use an oil of good body, so as either to hasten the drying when it is used either directly or after it has been incorporated with varnish, or when it is desired to apply the oil to porous surfaces, wood, oil, etc., without having reason to fear of its being imbibed by such substances.

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[1] In the boiling of oil over a naked fire with lead driers any degree of body desired may be easily obtained, from a comparatively limpid oil to one approaching, if not equal to, rubber in consistency according to the nature and amount of the drier and the heat used in the process and the duration thereof. Vincent in fact complained of the trade not taking kindly to his oils owing to their limpidity. In some of the processes described under this heading the oil is not thickened to the extent imagined by Livache.

The simplest process consists in causing the oil to fall in a thin layer, in contact with air, as we have seen in the cold process of making drying oils, but the process is a long one, and oxidation becomes difficult as soon as the oil attains a certain thickness, even when working at a temperature of 50° to 60° C., by which the oil is kept fluid for a longer time. Further, a quantity of oil adheres to the plates, and there solidifies, causing a serious loss when it is not desired to convert the whole into a solid body. Finally, after a time a solid pellicle is formed on the surface of the oil, which prevents oxidation, and consequently the thickening from proceeding through the mass of oil.

OXIDATION OF A BENZENE SOLUTION OF LINSEED OIL.

Attempts have been made to artificially increase the fluidity of the oil by the use of a suitable solvent. By taking, for example, equal parts of manganese-boiled oil and benzene, and stirring the mixture in a closed vessel, with simultaneous injection of air, rapid absorption of oxygen takes place. This absorption is stimulated by aid of heat regulated between 40° and 50° C. If the air be continuously injected so as to supply the quantity of oxygen necessary for complete oxidation of the oil, a quantity which amounts at least to 16 per cent. by weight of the oil used, we very soon see the mixture thicken, and if finally we distil off the solvent a viscous residue is left on cooling. If the process be carried on so as to completely oxidise the oil, the liquid becomes cloudy owing to the formation of linoxin, which is insoluble in benzene; on separating the latter, the linoxin is obtained as a very dry, elastic solid.

ORDINARY PROCESS.

In actual practice boiling is continued for a long time, often for three or four days, and the temperature pushed as far as 250° to 300° C. By this means heavy bodied oils are produced, which can be thinned down by the addition of thinner bodied oil, boiled only from six to eight hours at 200° C. But boiling at so high a temperature has the disadvantage of liberating fatty acids, which take longer to oxidise, and render the oil fatty.

English, German and Dutch Practice.

In England and Germany preference is given to boiling oil by superheated steam or superheated air either in pans as flat as possible, so that the surrounding air may have free access, or in deep, closed pans, into which either cold or hot air is injected.

In certain manufactories, especially in Holland, no drier is used; it is considered preferable to oxidise the oil exclusively by the injection of hot or cold air. The product is superior, but the process rather slow.

Storage of High-Class Boiled Oil.

According to Livache.—When a choice boiled or thickened oil is manufactured, it should not be stored either in iron or copper vessels without electro-plating the interior with silver; this deposit need not be thick, as it is subject to no wear and tear, and owing to the low price of silver would not be so costly as would at first sight appear. The same result may be arrived at, *viz.*, prevention of discoloration by a layer of enamel which stands well and is economical. Wrought iron vessels "galvanised" on the inside surface with tea lead have no action on varnish nor on spirits of turpentine. One per cent. of tin added to the lead has the same effect.

In F. Walton's, Lion's, Hadfield's and other processes, air is made to act upon oil falling in drops, in such a manner as to present a large surface to oxidation. The *modus operandi* of such processes will be understood from Fig. 26.

Walton's Processes.

1. Walton's plant is composed of a reservoir, the bottom of which, pierced with a number of holes, rests upon a second

rectangular reservoir, of which two of the faces are made of glass so that the action of sunlight may intervene with the view of stimulating oxidation and bleaching the oil; the other two faces are made of perforated sheet zinc; through one of these faces air enters, the whole way up the column, and passes out through the other, but owing to the minuteness of the holes and to their conical shape the oil is retained. This quadrangular column is itself surrounded by a double envelope made either totally of glass, or having two faces at least of that material, and in which an arrangement of partitions causes a current of air to flow against one of the perforated zinc faces and to force its way through the opposite. This current should not be too strong, but yet strong enough to constantly renew the air in contact with the oil.

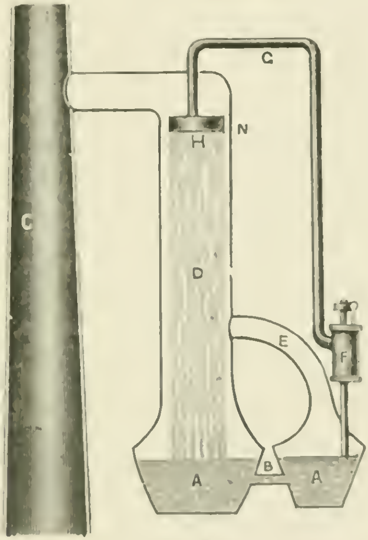

FIG. 26.

The oil at last falls into a steam-jacketed reservoir, by means of which it can be heated to a temperature between 100 and 170° C. Finally, by means of a pump similar to Fig. 26, F, the oil thus heated is propelled into the reservoir at the top of the column, through the openings of which it again passes to be submitted afresh to the action of the air. An automatic arrangement enables the flow of oil to be stopped should the pressure inside the column become too strong.

Raw oil, to which an appropriate drier has been added at the start, is that which is generally treated with this apparatus. Sugar of lead to the extent of 5 to 10 per cent. is the most suitable drier. However, oil previously boiled with manganese or litharge may also be treated, but air is propelled in this case at a lower temperature, say, 100° C.; nevertheless, in spite of this precaution, the resultant oil is darker than when raw oil is used.

2. Another arrangement by Walton consists in boiling the oil in large open pans; the hot oil is led into a chamber maintained at a suitable temperature by steam pipes. Here the oil which falls on the floor of the chamber is beaten with armed agitators, bringing it into the form of a spray which meets a current of air passing in the opposite direction; the oil falls back again into the chamber, which may be covered with glass, so that the action of sunlight may intervene.

STORER'S PROCESS.

Instead of *blowing* air into the oil, Dr. John Storer draws it in from the atmosphere by suction by the creation of a vortex current generated by a sort of screw propeller. By a mechanical arrangement the oil subjected to the vortex action flows into another compartment and returns to the surface to expel the vitiated air and to be again and again subjected to the same treatment. The screw propeller makes about 1500 revolutions a minute. It will be readily seen that the commingling of continually renewed fresh air, drier and oil is effected in a most complete and perfect manner by this process. The plant differs from Vincent's in the fact that the fumes are carried away, not by a dome rivetted to the pan and forming part of it, but by a hood suspended over the pan, thus allowing fresh air to be continually brought in contact with the oil without the necessity of pumping. The atmosphere above the pan is therefore always freer from decomposition products.

SOLIDIFICATION OF BOILED OIL—LINOLEUM MANUFACTURE.

When boiled oil is completely oxidised it is insoluble in the various solvents used in varnish-making, consequently manufacturers generally are content with a partially oxidised product which will dissolve.

But now-a-days there is a demand for completely oxidised and consequently solid oils, which are incorporated by mixing and grinding with a great number of substances, to which they impart impermeability, elasticity, as well as insolubility, not only in the various solvents for oil but also in acids. This is the case in the manufacture of linoleum, for example, and other similar substances formed by a mixture of solid linseed oil, powdered cork, sawdust and resin.

The process most generally adopted consists in spreading in a very thin layer boiled oil, or oil thickened after one of the processes described, so that it may be completely oxidised: if the layer be too thick a solid pellicle is formed on the surface when oxidation verges completion, which stops all further oxidation from going on underneath, and complete solidification is prevented.

When large quantities of solid oil are required it is necessary to have enormous surfaces at disposal, so that the layer of oil to be oxidised may be as thin as possible.

PASSING THE LINEN THROUGH THE OIL.

Linen cloths are passed through a bath of the oil to be oxidised, and thus become impregnated with the oil, and are afterwards suspended vertically so that the excess of oil drops off. When the layer of oil is dry the linen is passed through the bath once more and thus becomes charged afresh with oil, and again vertically exposed to the action of the air, and so on until a succession of solidified layers gives the thickness desired.

RUNNING THE OIL OVER THE LINEN.

The following is a more simple arrangement. Oil is made to fall upon several thousand square yards of linen suspended vertically. The oil being in a very thin layer absorbs the oxygen necessary to change it to the solid condition very rapidly. This absorption is facilitated by working in warm chambers. The sequence of operations which follow each other, almost automatically produces, very cheaply, a superposition of perfectly dry pellicles, amalgamated together to form a mass of considerable thickness.

SEPARATION OF SOLID OIL FROM LINEN.

In order to separate the solid oil from the linen, the latter is passed between two cylinders, heated by steam, and so adjusted that only the linen can pass through. In starting all that has to be done is to free the linen from solidified oil, for about an inch, and feed it in between the cylinders; these catch hold of the linen and draw it forward, leaving the solid oil behind. The linen can in this way be used over again to prepare a fresh batch of solid oil. But when complex substances are to be made from the solid oil, manufacturers do not trouble to separate the two, the more so as the linen does not last long. In that case the linen charged with oil is ground up and mixed with the other ingredients.

LIVACHE'S PROCESS.

Livache tried to make a direct utilisation of this oxidised oil which, when subjected to trituration with the majority of volatile solvents, spirits of turpentine, benzene, alcohol, ether, bisulphide of carbon, etc., is observed to swell, become transparent and then divide into pieces of extreme tenuity, and thus form a paste which may be spread out in a more or less thick layer. When the solvent has evaporated, an elastic homogeneous surface of oxidised oil is obtained, perfectly dry and of appreciable thickness.

These pastes of oxidised oil may be combined with caoutchouc pastes, or with solutions of india-rubber and other substances, soluble in the solvents used, so as to obtain after evaporation a homogeneous product of peculiar properties partaking of those of the different substances entering into its composition.

Printing and Lithographic Ink.

The principal constituent of printing ink is boiled oil, most generally linseed, sometimes hemp-seed, which is cheaper, but retains a bad smell. No attempt is made to produce a fluid quick-drying oil like that used in the manufacture of oil varnishes, *viz.*, by boiling the oil to a temperature not exceeding 220° C. On the contrary, we have to produce a partially resinified, partially decomposed oil which will leave no greasy stain on paper. This result is obtained by heating the oil above 250° C.

Refining Oil for Printing Ink.

The oil used should be of prime quality, so as to avoid the disagreeable smell and too deep colour of ordinary oil. It ought, however, to have been stored for at least a year or two, so that all solid impurities may have completely separated out. In other words, what is known as old tanked oil should be used, but it ought still to undergo a special refining with sulphuric acid. Accordingly the oil is mixed with a few per cents. of concentrated sulphuric acid of about 50° B., and heated for several hours by means of a steam coil to a temperature of 90° C., taking care not to go beyond 100° C. The oil is then pumped into another tank containing hot water, with which it is well washed, running off the wash water and agitating afresh, and so on until all trace of acid is removed. The oil may then be boiled.

As drying oils contain a certain quantity of fatty matter (palmitin) which does not dry, and causes the printing ink to blot or blur, it has been recommended to eliminate it by adding to every 1000 parts of oil 1 or 2 parts of fuming nitric acid, which decomposes the palmitin into glycerine and palmitic acid, which may thus be precipitated as insoluble palmitate of lead or manganese. This process seems, however, to present many difficulties, which have prevented it from being adopted to any extent. Moreover, fuming nitric acid, in consequence of being easily decomposed, is by itself a dangerous substance; this danger is intensified to the highest degree when the acid is mixed with organic matter. Dangerous explosions may result and the oil take fire.

Description of Pan.

The oil is boiled either in a cylindrical copper pan, the lower half of which fits into the brickwork of the furnace, where it is kept in position by a collar which at the same time acts as a gutter to collect any overflow, or in a cast-iron enamelled pan. The pan is manipulated with a chain or by an iron band encircling its top, and attached to which are strong arms, by means of which it can be moved with safety. The capacity is 60 to 85 gallons. But in certain important factories the boiling is done in pans which do not cube less than 600 to 700 gallons. In such cases a mechanical agitator is kept going during the boiling process, which may last two or three days.

Boiling Process.

The pan is filled to half or at most three-quarters of its capacity, and a moderate heat applied so as to get quit of all trace of moisture; when the boiling goes on quietly the fire is quickened until volatile, pungent and disagreeable smelling products are given off. This takes place at a temperature of 250° to 270° C. In certain factories the workman satisfies himself as to this being the case by dipping in organic matter, such as a feather, for instance, which ought to inflame; or by projecting fine drops of water on to the surface of the oil, which as they fall decompose and scintillate, or, in other words, present, in consequence of the absorption of oxygen by the oil and the inflammation of the hydrogen, the appearance of stars. The fire is now carefully watched, for the oil may froth over or the vapours ignite. The frothing over is parried by adding a little cold oil.

The oil becomes bright and more fluid at first, but, on being kept at this temperature for one or two hours, a brown red skin forms on the surface, which is skimmed off as rapidly as possible after it has formed, because it is this skin which colours the oil brown. The temperature is now raised to 310° C., and kept at that for half an hour to an hour, watching the process with the greatest of care, as the vapours are very apt to catch fire. A well-fitting lid and wet cloths ought therefore to be always at hand. The fire is now allowed to die down, so that the temperature may descend to 260° to 270° C., when the pan is withdrawn from the fire. It is covered with a lid, and let stand to cool.

Certain manufacturers, instead of cooling the oil after withdrawing it from the furnace, set fire to the escaping vapours, and let them burn for about five minutes—an operation they regard as indispensable in the production of a good article. Other manufacturers contend that the same result is obtained by boiling the oil a little longer, which has the further benefit of saving the appreciable loss of oil incurred during the combustion process, and, further, a much paler oil is obtained.

"STRING" TEST.

The exact moment when the pan ought to be removed from the fire is determined by the "string" test. A small sample of oil is taken from the pan on a wooden spatula, which is cooled rapidly by stirring it. A drop is taken and pressed between the finger and thumb, which are then gradually drawn apart as far as possible. The thread joining the finger and thumb together should stretch 1 to 2 inches without breaking, otherwise the oil is not sufficiently boiled.

Properties of Good Printing Ink.—Good printing ink has the requisite body of a pale, bright yellow colour—not deep yellow or brown—and is not cloudy from matters in suspension. It is very evident that the body of the oil will depend upon the purpose for which it is intended. For printing books and lithographic work an oil having a good body is required, whilst for newspaper printing a soft, almost fluid oil is used. According to Villon, who made an elaborate study of printing ink, five kinds of oil of different body or consistency are in the market, *viz.* :—

(1) Extra strong, (2) strong, (3) medium and (4) weak-bodied oils, and (5) drying oil. The strong-bodied oil (2) is used in summer for very particular work with the hand press. The medium oil (3) is used in ordinary weather. The weak-bodied oil (4) is used in winter with the mechanical press, whilst the drying oil (5) is used to prevent the formation of the oily ring around the letters.

DRIERS AND EXTRA STRONG-BODIED OIL.

Usually no driers are used, because oil boiled with driers would clog the type, and could not be removed even when washed with a hot alkaline solution. There is sometimes, however, an advantage in using such an oil, and to meet this want the oils known as "extra strong" and "drying" oils are manufactured.

The "extra strong-bodied" oil is made by boiling with lead salts, and the "drying" oil with an oxide or salt of manganese.

The "extra strong-bodied" oil is added to the "strong-bodied" oil. The former is made by heating to 150° C. for an hour

Linseed oil	200
Litharge	5
Umber	4
White lead	2

The drying property of this varnish is tested by spreading it on a glass plate, when it should be perfectly dry next day, with no air bells nor dulness. If it be dull 5 per cent. of spirits of turpentine must be added.

The drying oil is obtained by heating for fifteen hours at 275° C. oil to which 0·05 per cent. of the hydrated oxide of manganese has been added, or by boiling oil for twenty-four hours at 300° C., with 5 per cent. of black oxide of manganese. If borate of manganese be used 4 parts of this salt are beaten up with 25 parts of linseed oil, and run into 2000 parts of linseed oil, and the whole heated for half an hour at 250° C., or a quarter of an hour at 316° C. Some manufacturers add 2½ per cent. of oxalate of manganese, whilst applying heat for three hours at 150° to 160° C., or simply add a manganese soap (manganese linoleate) previously dissolved in coal-tar naphtha, petroleum ether, or spirits of turpentine.

USE OF ROSIN.

When great body is required, without having resource to prolonged boiling, rosin may be added. With ordinary black ink common rosin is used, but for fine, coloured inks pale American rosin is employed. The rosin is melted, filtered to eliminate rubbish, and added to the oil during boiling. When very high-class inks are required, balsam of Peru, Canada balsam, or copaiba balsam are used instead of rosin.

USE OF SOAP.

Finally, a certain quantity of soap is added to the oil, which imparts suppleness, but its principal object is to facilitate the cleansing of the type, enabling this to be done with a brush. The soap ought to be perfectly dry, that is to say, when left for a long time in thin slices; at 100° C. it ceases to lose weight. Ordinary yellow resin soap is used for common ink, but for fine inks white soap is used.

BLUE BLACK INK.

A little indigo blue is also added to the hot oil to impart lustre, or 1 per cent. of Prussian blue may be added as the oil commences to boil, which, as it dissolves, yields a beautiful blue black.

MANUFACTURE PROPER.

Generally in the actual manufacture of the ink, the boiled oil is moderately heated with the simple addition of rosin and soap alone until the mass becomes tranquil and homogeneous; the colour is then added and ground in the mill until perfectly cold.

Another process consists in melting the rosin until destructive distillation commences, and then to add soap in slices, then the boiled oil. Heat is now applied more strongly, so as to increase the fluidity of the mass, then the colour is added, and the whole run into a receiver, from whence it passes to the mill while still hot.

The nature and the quantity of the colouring matter depends upon the consistency of the oil and the object in view, but heavy, dense, mineral colours should, as far as possible, be excluded, using in preference light colours, which give a more homogeneous and impalpable ink.

Thus the following colours are habitually used, *viz.*, for

Black—lamp-black.

Red—lake, cochineal, carmine aniline colours; very rarely red lead or vermilion.

Blue—Prussian blue or indigo; ultramarine is inadmissible as it is easily bleached.

Yellow—yellow ochre or chrome yellow.

Green—mixtures of blue and yellow.

Brown—sepia and umber.

CHARACTERISTICS OF GOOD PRINTING INK.

Printing ink ought to satisfy several tests. (*a*) In the first place, it ought to be perfectly homogeneous, for the least particle of soiled matter (grit, etc.) will cause a blot in the printing. (*b*) It ought to suffer no change when exposed to the air for a long time, but when spread in a thin layer it should dry very quickly, without, however, commencing to do so on the type, from which it should be easily removed by washing. (*c*) The colour of dry printing ink should be rich and brilliant. (*d*) It should not penetrate the paper too deeply but deep enough to prevent it being detached from the surface. (*e*) Finally, it ought to have no bad smell.

The proportions of different substances entering into the composition of printing ink vary much. They may, however, be reduced to three types:—

	Best. Parts.	Medium. Parts.	Ordinary. Parts.
Boiled oil	100	100	100
Rosin	25	50	75
Soap	4	6	8
Drying oil	6	9	12

For ordinary, such as newspaper, work, great economy is necessary, hence in place of boiled oil there has been used a mixture of raw oil and thickened turpentine: the quantity of resin is increased and the requisite fluidity obtained by the addition of rosin oil.

The proportions given by Andés are as follows, according to the amount of body required:—

	Parts.	Parts.	Parts.
Raw linseed oil	140	110	90
Rosin oil	210	210	210
Rosin	210	210	210
Rosin (*i.e.*, yellow) soap	5	5	5
Thickened turpentine	5	5	5

We may add that the same products, but of inferior quality, are made very cheaply by replacing linseed oil by rosin oil, for which purpose the following recipes have been given :—

	Parts.	Parts.
Rosin oil	1000	1000
Rosin	400	780
Rosin soap	100	780
Ordinary soap	100	180

INK FOR STEEL ENGRAVING.

This oil, according to Villon, who made a special study of ink for steel engraving, is prepared like printing ink, the only difference being that it is boiled a little longer.

Linseed oil which has aged is the oil employed, or, better still, walnut oil; but this latter is never used alone, a little linseed, varying from 12 to 50 per cent., according to the quality desired, being always added to give it more viscosity.

The boiling process, although an old one, is that still used. It consists in heating 5 gallons of oil in a pot until it takes fire or gives off white fumes, which are set on fire. It is left to burn for five minutes, taken off the fire and placed on a furnace filled with hot ashes, where it is let burn a few minutes longer. A piece of stale bread, held by a pair of pliers, is dipped into the oil until it reddens, without, however, charring; it is replaced by a second slice, and that by a third—the bread is sometimes replaced by peeled onions. Although no explanation is given of this process, it would appear to be necessary. At any rate practical men think so. The pot is now covered and allowed to cool. There is thus obtained a *bright*, greenish oil (*huile claire*), more consistent than the raw oil, but with no body.

Strong-bodied oil (*huile forte*) is prepared by setting fire to the oil, letting it burn for five minutes, and stirring several times. The flames are put out and the oil again set fire, twice at least, and sometimes four times. The cooled oil strings like treacle.

The "fatty" oil is made by shortening the burning process, and is intermediate between the other two.

Now-a-days a process similar to that used in making ordinary printing ink is adopted.

The removal of grease by onions or bread is then effected by nitric acid, as previously indicated, or by magnesia. This latter, which is extensively used in England, consists in adding 2 per cent. of magnesia, boiling for an hour, and decanting.

The oil is then run into a pan capable of holding 42 gallons, and gradually heated to 182° C., three hours being occupied in attaining this point. About 1¼ lbs. of finely ground bichromate of potash is then added, and the temperature is raised to 300° C., stopping the frothing of the oil by adding a little cold oil, then it is kept for half an hour at a temperature between 315° and 320° C., after 1¼ lbs. of Prussian blue have been slowly added. "Strong"-bodied oil is thus obtained. To make "fatty" oil, the oil is only heated for five minutes to 300° to 305° C. With "clear" oil the heat does not rise above 275° C., which is maintained a quarter to half an hour, according to the object in view.

De Luxe Drawings, Ink for.—Villon gives the following proportions for ink to be used in the production of *de luxe* drawings:—

Limpid oil (huile claire)	1000
Strong-bodied oil (huile forte)	250
Black	500
Prussian blue	50
Rosin (yellow) soap	25

The Prussian blue is first added to the clear oil in a pan fitted with a mechanical agitator, and heated to 100° C. by a steam jacket. The heat is raised to 150° C., and the rosin soap is mixed with continual stirring. It is only when perfectly cold that the black is introduced into the mixture, then the huile forte (strong-bodied oil). The whole is then passed through rollers. Small quantities may be ground in a mortar worked by power.

Black Ink for Engraving Purposes.—Villon's researches resulted in his selection of the following formula as yielding a very black ink for engraving purposes, without reddish reflex, and of irreproachable composition, which keeps well, and each of the ingredients of which plays an important part:—

Huile claire (limpid oil)	1000
Huile forte (strong-bodied oil)	200
Carnanba wax	25
Paraffin	35
Yellow soap (rosin)	25
Paris violet	0·5
Prussian blue	40
Cork black	100
Blood black	50
Wineless black	200
Ivory black	150

Numerous other substances are also used to impart consistency and unctuosity, such as Canada balsam, ozckerit, asphaltum, stearic acid. It is for each manufacturer to select those materials best adapted for the object in view.

INDIA-RUBBER SUBSTITUTES.

Drying oils are used in the manufacture of a solid elastic compound, which can be used as a rubber substitute. Chloride of sulphur or nitric acid are the re-agents used.

CHLORIDE OF SULPHUR PROCESS.

By mixing linseed oil with 5 per cent. of chloride of sulphur the oil becomes very thick, becoming plastic with 15 to 20 per cent., the solidity increasing as the percentage rises to 25 per cent.

If the mixture be cooled solid plates are obtained, on which other layers can be superimposed in such a manner as to obtain plates thick enough for making printing rolls, boxes, knife handles, etc.

If a certain quantity of carbonate of lime be added the carbonic acid evolved converts the mixture into a white spongy mass, which is mixed with natural rubber.

Chloride of sulphur has the same action on other oils such as earthnut or colza.

NITRIC ACID PROCESS.

Dilute nitric acid and linseed oil yield a product somewhat analogous to the preceding; but in the latter case it would appear to be the sulphur which is the active agent, and which explains why we can have the same substance by using either drying or non-drying oils. In the present case, however, we are confronted with a rapid oxidation process exclusively confined to those oxidisable principles only found in drying oils.

The oil is first heated so far as to render it viscous; it is then boiled for a long time with dilute nitric acid. A solid elastic, brown substance, which does not stick to the fingers, is obtained analogous to caoutchouc, and whence its name of black artificial rubber.

The same substance is obtained with the different drying oils, but in proportion to the intensity of their drying properties. Linseed oil and walnut oil yield eight to ten times as much as poppy-seed oil.

PROPERTIES OF RUBBER SUBSTITUTE.

This substance does not melt, but may be differentiated in a very precise manner from the final product yielded by the complete oxidation of drying oils, *viz.*, linoxin, by the fact that it cannot be saponified by a concentrated aqueous solution of potash. On heating an emulsion is obtained which does not separate on the addition of an acid. With alcoholic potash, on the contrary, complete saponification takes place, and the addition of an acid liberates a mixture of fatty acids of complex constitution. With bisulphide of carbon there is an emulsion; in petroleum the substance swells without dissolving; in a mixture of alcohol and ether it swells and dissolves if a large quantity of ether be added, whilst it is precipitated if alcohol be added in too great proportion; finally, it is soluble in ether and in a large excess of spirits of turpentine.

Summary.—Rubber substitute, prepared from linseed oil and nitric acid, would appear to consist on the one hand of a mixture of substances present in the original oil and not sensibly changed by the nitric acid treatment, and on the other hand of products oxidised by the nitric acid and transformed into linoxin, the relative proportions of these substances masking the individual properties of each of them.

This substance is used either in solution in ether or in spirits of turpentine in waterproofing tissues, or is added directly in the solid state to india-rubber.[1]

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[1] *Synthesis of Genuine Rubber.*—The attempts that have been made to convert spirits of turpentine into real rubber have not as yet been carried out in practice.

SECTION VIII.

MANUFACTURE OF OIL VARNISHES.

OIL VARNISHES.

SPIRIT varnishes, consisting simply of a resin dissolved in spirit, are not adapted for outdoor work, or for application to any object subject to wear and tear. They certainly produce a brilliant coating, but with feeble powers for resisting either heat or moisture. It is therefore necessary to introduce an element which, in virtue of its elasticity and impermeability, will impart the necessary resistance, whilst at the same time it is so transparent that it does not attenuate, but in the smallest possible degree, the brilliancy of the varnish.

The substance which fulfils these conditions is the oxidation product of drying oils, and the varnishes into which it enters are called oil varnishes.

The use of a protective coating with linseed oil as a basis was indicated by the monk Theophilus so far back as the twelfth century.[1] But for a long time oil varnishes were only made in small quantities by each tradesman, in accordance with recipes transmitted to him, and according to the exigencies of his own personal work.

According to Livache, the first varnish factory was established in England in 1790, in France and Germany between 1820 and 1830, and in Austria in 1843.

The composition of oil varnish is simple, since it only contains resin, drying oil and a volatile solvent.

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[1] "Linseed, walnut and poppy seed oil were known to Theophilus and probably all of them to the Greek painters as drying oils fit to be used in painting. Linseed oil could not have remained long unknown to the Egyptians. Great cultivators of flax, skilful in the arts and in medicine, they could not have overlooked the production of an oil from the linseed nor have been ignorant of its peculiar properties."—*Hendry's Theophilus*, p. 94.

Nevertheless, there are many different qualities of oil varnish, which vary with the quality and relative proportion of the different ingredients and the method of manufacture.

The qualities desired in a varnish are: (1) colour. This should be as pale as possible so as not to appreciably alter the tone or tint of any object to which it may be applied or that of any colouring matters which may be added to the varnish; (2) consistency or body so as to be easily applied; (3) drying properties; (4) brilliancy; and (5) resistance.

(1) *Colour.*—The colour will be paler the more care has been taken in the selection of the resins and in selecting a pale oil, and the greater the attention with which the temperature necessary for the fusion of the resins and the process of manufacture has been regulated.

(2) *Consistency.*—This is principally determined by the relative proportion of the three ingredients, oil, resin and volatile solvent, according to the object in view.

(3) *Drying Properties.*—These will depend on the rapidity with which the volatile solvent evaporates, and, above all, on the quality of the drying oil. The more care taken in the boiling of the latter to ensure good drying properties, the better will the varnish dry.

(4) *Brilliancy and lustre* depend on the nature of the resin, which ought to form a sufficiently thick, continuous coat.

(5) *Durability and resistance* will vary with the proportion of linseed oil and the elasticity of its oxidation product.

Varnishes ought to combine in the best proportions the properties of the resins (brilliancy) and of the drying oils (elasticity). As far as durability is concerned, it depends, above all, on the resins used and the relative proportions of the different ingredients. Take, for instance, varnish made from the hardest resin, Zanzibar copal. This varnish may last for eighteen months at the most, but in the case of low quality varnish, especially quick-drying ones, the durability is lowered to a few months or even weeks. They are seen to lose their brilliancy, become dull, then, after the formation of fine cracks, assume a whitish appearance and finally to resolve into small pulverulent scales.

CHOICE OF RAW MATERIAL.

1. RESINS.

The resins employed ought to be carefully selected, as indicated in the preceding part whilst treating of the raw materials employed in the manufacture of varnish. Hard copals and amber have to be submitted to a high temperature before they dissolve. They are generally rendered soluble during the process of varnish-making; sometimes, however, the process is so modified that resins which have sometime previously been heated to the requisite extent (pyro-succin, pyro-copal) may be used.

The colour and brilliancy of the varnish will depend upon the amount of care exercised in not only the selection of the resins, but their previous treatment and the skill exercised in running them.

2. LINSEED OIL.

Linseed oil is the drying oil generally used. Its quality is of prime importance, for upon that depends the drying properties, elasticity, and the resistance and durability of the varnish. High-class varnishes can only be made from pure and perfectly refined oil. It ought to be "aged" or tanked for at least a year, no matter whether raw oil or manganese or lead-boiled oil be used. This oil ought previously to have absorbed a certain quantity of oxygen, so as to render it more drying. The temperature at which the oil decomposes ought to be taken into account, for at the high temperature at which copal melts certain oils darken very rapidly. The driers with which the oil has been boiled ought also to be considered. Manganese compounds are less hurtful than those of lead, because the linoxate of manganese is less brittle than linoxate of lead: further, there is less manganese absorbed by a manganese-boiled oil than there is of lead by a litharge-boiled oil.

Many manufacturers, chiefly in England, prefer to use only oil which has been boiled for a prolonged time without any addition of drier, and which has afterwards been stored for a long time; such an oil is pale in colour, is not altered by sulphur compounds, and durable varnishes are made from it even with soft copals.

3. Solvents.

The spirits of turpentine used as a solvent is the ordinary spirit; when the varnish is applied on a surface the spirit never evaporates completely, and the portion remaining oxidises and resinifies; being softer than the resins used, this residue imparts elasticity and durability to the coating.

Certain manufacturers prefer American spirit to French; the chief reason is that English varnishes, which are of excellent quality, are made with American spirits, and as the latter possesses a peculiar essence of lemon smell, this smell has become recognised as an indication of superior quality varnish.

Turpentine Substitutes.

Attempts have been made to replace spirits of turpentine by benzol, petroleum ether, and by mixtures of different solvents, etc., but except in certain particular instances the bad smell of these solvents and their peculiar toxic effects, and last, but not least, the danger of fire and explosion, have each and all caused the use of these solvents to be abandoned, especially for inside work, whether on the interior of buildings on land or ships at sea or in dock.[1]

Manufacture of Oil Varnish.

The quality of a varnish depends principally upon the hardness of the resins used, but a difficulty is met with in

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[1] Moreover, as each of the constituents of a mixture of different solvents may evaporate more or less quickly than the others, unequal drying results with all its attendant evils

the very beginning. The hard resins do not dissolve directly and at once in the solvents used, and they in particular refuse to dissolve in boiled oil or spirits of turpentine. It has been pointed out, when describing the properties of the raw materials used, that it was necessary to render these hard resins soluble by previously heating them to a rather high temperature. It is therefore necessary to modify the manufacturing process according to the manner in which the resins used behave when treated with different solvents.

(A.) VARNISHES MADE FROM RESINS WHICH DO NOT DISSOLVE DIRECTLY.

AMBER AND COPAL VARNISHES.

(1) *Addition of Oil to Melted Resin.*—The process most generally used consists in melting the resin in a copper vessel on the naked fire, and then to afterwards add first the oil and then the spirits of turpentine in suitable proportions.

Fusion of Resins.—Without going into detail in regard to the fusion of the resins, which has already been dealt with at some length (p. 157 *et seq.*), it must be borne in mind that the pot ought to have a capacity double that occupied by the resin used, so that there may be no risk of the material priming over, especially when it contains much water. It is equally necessary to recall the fact that it is better to work with small quantities at a time, say, 3 to 5 lbs. at the most, so as to obtain as pale products as possible, which cannot be done when 25 to 35 lbs. are "run" each time. The time occupied in working with small quantities must not be taken into account; it is more than compensated by the paler product.

Finally, copper pots must be used because it is necessary to heat to 300° C., and at this high temperature copper has more resistance than iron.

When the disengagement of fumes has ceased, and when it is thought that the resin is about fluid, the varnish-maker assures himself of this being the case by "sounding" the varnish with his spatula, which should be made of iron, because wooden spatulas besides darkening the varnish contain moisture and give rise to boiling or frothing, which may mask the fluidity. When the resin is completely melted and fluid it runs from the spatula in drops like water. It is indispensable to make absolutely certain in this manner of the resin being fluid, because if it be not so the addition of oil would yield a turbid viscous mass, or the spirits of turpentine would separate the resin in a viscous lumpy aggregation.

When treating certain copals containing much moisture it is better to let the resin remain on the fire for two or three minutes longer, after it has been melted, so as to drive off all trace of water, otherwise, although a clear product would be obtained on the addition of linseed oil, it would become turbid on the addition of spirits of turpentine.

Incorporation of Linseed Oil.—The raw linseed oil is heated separately, and the necessary quantity is added to the fluid resin, whilst stirring constantly. After incorporation some manufacturers allow this mixture to cool, mostly in the case of linseed oil, which has been previously boiled with driers, but in general it is preferable to heat it for some time, so as to bring it to the proper consistency or body. To prevent fire, the temperature should not exceed 300° C.

The process is often modified by running a certain number of potfuls of the mixture of fluid resin dissolved in oil into a large pan which is heated to about 140° C., and kept at that temperature until the oil has become sufficiently thick, and finally formed an elastic substance which imparts such suppleness and flexibility to the varnish that the expense and the increased length of the process are more than compensated.

Stringing.—The heating should be continued until the varnish, when spotted on a glass plate, yields a bright transparent product possessing the greatest possible consistency and capable of being drawn out into long elastic threads which do not break easily. Each drop falling on the glass plate should present in a decided manner the form of an arch. This result is obtained by prolonging the heating process in proportion to the thinness of the mixture at the beginning—that is to say, the more oil it contains the longer the process.

Incorporation of Spirits of Turpentine.—Whether working with a single pot or with a pan into which several potfuls have been emptied, the mixture is allowed to cool to 160° to 170° C., that is to about the boiling-point of spirits of turpentine, and the solvent, *i.e.*, spirits of turpentine, is slowly added, preferably from a jug with a long neck or spout, in a small stream and in small quantities at a time. About $\frac{1}{10}$ part of the solvent is added in the beginning and half that quantity each time afterwards, sampling after each addition. So long as the liquid maintains a certain viscosity on cooling and thickens rapidly, the addition of spirits of turpentine is continued. But when the consistency diminishes greatly on the addition of a small quantity of spirits, the limit has been reached and no more must be added, otherwise the quality of the varnish will be injured.

The temperature of the process may vary between 190° and 140° C., according to the quantities operated upon. If the temperature be too high there is considerable loss of spirits of turpentine by volatilisation, but, on the other hand, if the temperature be too low the incorporation is effected badly or not at all. The essence is generally heated so as to avoid too sudden cooling of the mixture, and in this case the mixture of resin and oil may be brought to a lower temperature than when the spirits of turpentine is added in the cold, thus greatly diminishing the disengagement of vapour on the addition of the latter. With ordinary good working the loss of spirits of turpentine is about 5 per cent., but it may rise as high as 10 per cent. if the temperature be too high.

(2) *Addition of Resin to the Oil.*—This process, due to Tingry and Andres, requires the adoption of special plant, the work-

ing of which has already been described in the chapter devoted to the influence of heat upon the solubility of resin. The melted resin falls drop by drop into a vessel containing the oil, heated to a suitable temperature; solution takes place as soon as it drops into the hot oil, producing a homogeneous product. But this process presents many difficulties, the principal being the risk of fire from the inflammation of the vapours.[1]

Mixed Process.—The manufacture of oil varnish in certain English factories is based upon the two previously described processes. Nine parts of oil are heated in a pan almost to boiling, and $\frac{3}{5}$ of it are taken out and put into flagons, each containing $\frac{1}{5}$. These flagons are placed on the plate of the furnace and kept at a good heat. On the other hand, 1 part of copal is fused in the gum pot in the ordinary manner, and when the copal is completely melted and runs like limpid oil the contents of one of these flagons of oil is added, *i.e.*, $\frac{1}{5}$ of the total oil. When the mixture is complete the whole is run into the boiling pan which contains the rest of the hot oil, kept at a sufficiently high temperature.

The melting pot is cleaned with a brush or a cloth rag previously dipped in spirits of turpentine, and 1 part of copal is again melted in it, and at the proper moment added to the contents of one of the two remaining flagons of oil; the mixture is run into the boiling pot, which has already received the mixture resulting from the first fusion. The same operation is gone through with another part of copal, to which is added the contents of the third flagon of oil. Working in this way, after having first mixed the oil with the resin and then added the resultant mixture to the oil in the boiling pan, there will in the end be in the latter 3 parts of copal and 9 parts of oil, which is placed over a good fire and maintained at a rather high temperature, taking care, however, to remove the pan from the fire if at any time it

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[1] See Fig. 6, p. 164.

threatens to boil over. The driers are now added in successive small quantities, with continuous stirring. The time necessary to bring the three successive "runs" to the desired condition varies from three and a half to four hours, when a sample is taken and placed on a glass plate. It ought to give a clear, homogeneous, transparent mass, which strings into elongated filaments when pressed between the finger and thumb, and these afterwards extended away from each other. If it does not string the boiling must be continued. The pot and contents are withdrawn from the fire, and when sufficiently cool the spirits of turpentine are added, the quantity varying from 13 to 17 parts, according to the hardness of the copal and the success which has attended the fusion. The operation finished, the boiling pan is emptied and cleaned as before with the spirits of turpentine which had already served to clean the melting pot.

FIG. 27.—MANUFACTURE OF SUPER BLACK JAPAN.

This process, which would appear to give very good results, is applicable to large quantities, certain factories working in the melting pot a mixture of oil and copal of about 70 lbs. in weight. Further, sometimes the contents of ten of these pots are mixed in the boiling pot, which is mounted on a movable truck (Fig. 27) and placed on a furnace. Seven hundred lbs. of oil and copal are thus boiled at one operation; manganese driers are used to the exclusion of lead; ¼ lb. of borate of manganese is used for every 100 lbs. of oil. The whole is boiled for half an hour.

Fig. 27, illustrating the manufacture of black japan, will give some idea of modern English methods. The vapours, it will be observed, ascend through the hood and pass along the conduit to the chimney shaft.

SOLUTION IN CLOSED VESSELS OF THE PREVIOUSLY FUSED RESIN.

Attempts have been made to secure better results by dissolving the previously fused resin in a closed (hermetically sealed) vessel.

Bartky's Process.—Bartky works at the lowest possible temperature, thus avoiding loss of raw material and risk of fire.

He accordingly breaks up the previously fused resin and heats it with the oil, at first to 80° and then to 150° C. The pan is then covered with a lid which hermetically seals it. Through the lid there passes an agitator, which is kept in motion for about an hour, the temperature is allowed to cool down to 100° C., the agitation being still kept up; it is then reheated to 130° C., then allowed to fall to 110° C., and by an opening inserted in the lid the necessary quantity of spirits of turpentine is introduced.

Andres' Process.—Andres works in rather a different manner, but based upon the same principle, *viz.*, solution in a closed vessel at a low temperature. The boiler which he uses is placed in a water bath and is furnished with a lid which dips into a hollow flange or circular gutter affixed to the circumference; by filling this hollow with oil the boiler is hermetically sealed, except so far as to allow of the escape of any small quantity of vapour which may be engenerated. In the interior of the boiler and about two-thirds from the bottom there is placed a ring shelf, upon which rests a metal box with a perforated bottom in which the previously fused resin is placed.

The boiler being filled with oil and the water bath started, there is added to the oil one-fifth of the copal to be used to which its own weight of oil of copal produced during the fusion of the resin has been added. The remainder of the copal is then placed in the interior receptacle and the necessary quantity of spirits of turpentine added, taking care that the copal is immersed for about 4 or 5 inches.

The lid being placed on the boiler all that has to be done is to heat the whole at 100° C. for three or four hours to effect complete solution. When the operation is finished, the contents of the pan are run off through a pipe in the bottom, taking care to place a cloth in front of the orifice so as to secure easy filtration of the hot varnish.

The principal advantages claimed by Andres for this method are economy in fuel and working expenses, as the work is done without stirring. Moreover, there is no risk of fire, no loss of spirits of turpentine, and finally, there is obtained a very pale varnish due to the low temperature at which the process has been conducted by heating all the ingredients together in closed vessels.

SIMULTANEOUS SOLUTION OF RAW RESIN OIL AND SOLVENT IN CLOSED VESSELS.

So far back as 1846 Schutzenberger suggested the heating of the three ingredients (solvent, unchanged resin and oil) in a closed vessel. The operation lasted two hours.

In 1856 Violette took the matter up and showed that perfectly pale varnishes could be obtained by this method and without loss of raw material.

Although this process does not appear to be adopted in actual practice, it is as well to bear it in mind, because it is probable that owing to the great improvements and developments which have been effected upon plant working under pressure, it may be generally adopted in the near future.

Certain methods of solution have also been suggested, based upon an observation of Violette, *viz.*, that amber and copal which melt at 350° to 400° C. melt without loss in weight at 100° C. in closed vessels, in consequence of a new grouping of molecules.

Experiments made with finely pulverised resins in hermetically sealed apparatus have afforded interesting results, but up to the present this mode of working has not been adopted on the large scale. Moreover, foolhardy and inexperienced persons run great risk in working or experimenting with plant of this description.

SOLUTION IN THE COLD OF THE PREVIOUSLY FUSED RESIN.

Attempts have been made to simplify the operation of varnish-making by using previously fused resins and dissolving them directly in the cold in a mixture of oil and spirits of turpentine. The resin may be dissolved in the ordinary manner or in a closed vessel.

When solution is effected in closed vessels, the product is not darkened in any way, and there is no loss if a suitable temperature be used. Now Violette has shown that hard resins, like copal and amber, after fusion in closed vessels have the property of dissolving either in the hot or in the cold in the usual solvents, and particularly in spirits of turpentine and fatty oils.

Violette wrought with silverplated copper tubes capable of being hermetically sealed. In this tube, which measured about 20 inches in length, $2\frac{1}{3}$ inches in width, and about $\frac{3}{8}$ of an inch thick, he heated about $2\frac{1}{4}$ lbs. of resin.

With regard to colour, the temperature varies according to the resin used. Calcutta copal melts below 315° C., the hardest copals at 350° C., and certain ambers at 400° C The tube should be made in such a manner that when opened the small quantity of gas disengaged during the melting may escape without danger. *No inexperienced person should attempt to work with sealed tubes. The risk is great, and explosions not only during the time the tubes are being heated but also on opening them are much to be feared.*

Solution in the cold sometimes yields a turbid varnish, but this is simply due to the presence of water, which may be allowed to escape before sealing the tube, or may be driven off by heating the varnish to 100° C.

This cold process ought to attract the attention of manufacturers. It is easily wrought and there is no loss of spirits of turpentine. Besides, if previously fused resins be bought all risk of fire is done away with, and the process has the further advantage of producing very pale, quick-drying varnishes with great fluidity if an oil the drying properties of which have been stimulated by being subjected to one of the cold processes previously described. In particular, if we employ an oil treated in the cold with precipitated lead and a salt of manganese, the proportion of spirits of turpentine may be largely diminished, this oil having preserved its original fluidity even whilst acquiring intense drying powers, and the varnish into which it enters after drying will have great elasticity.

(*B.*) VARNISHES, THE RESINS OF WHICH DISSOLVE DIRECTLY IN THE SOLVENT WITHOUT PREVIOUS FUSION.

The manufacture of varnish becomes very simple if the resin dissolves directly in oil. Solution is effected on the water bath, and the product is afterwards thinned down by adding the requisite quantity of spirits of turpentine. The resin ought to be ground as finely as possible and beaten up with a little cold oil; this mixture is added to the bulk of the already heated oil, the whole is stirred continually and the heating continued until complete solution is effected.

If the resins do not dissolve easily in oil, or if it be desired to accomplish solution more rapidly, an intermediate solvent may be used; thus shellac, sandarach, resin, etc., or such like substances as india-rubber and gutta-percha may be dissolved in an appropriate solvent such as benzol, spirits of turpentine, ether, etc., and the resultant solution added to the oil or to an oil varnish made from a hard resin.

This addition may be done in the hot or in the cold according to circumstances, and the whole may afterwards be thinned down with spirits of turpentine. This process may even be employed with certain hard copals.

When it is desired to make varnish from a mixture of hard resins and resins directly soluble, these latter may be added to the copal or amber in the melting pot and the whole poured into hot linseed oil, thinning down afterwards with spirits of turpentine. Finally, certain difficultly soluble resins may be coaxed to dissolve by adding to them certain resins which dissolve easily.

This is the case for example with kauri to which dammar has been added. We may recall the direct addition of liquid driers to linseed oil.

Filtration and Clarification.

Varnishes after manufacture and before being used ought to be filtered and clarified to separate the smallest impurities which would seriously depreciate its value by preventing it from drying with a uniform surface. The loss entailed by these operations may amount to 1 or 2 per cent. according to the quality of the resins used.

Filtration is effected by running the hot varnish through coarse linen or fine tulle. There is no advantage in using metallic sieves; they are dear, are easily pierced, and can only be cleaned with difficulty.

Clarification is effected by prolonged storing of the varnish in a warehouse kept at a moderate temperature and preferably in semi-darkness during a period of not less than eight days. Under these conditions all impurities, even those of extreme tenuity, are deposited. In England, high-class varnishes are allowed to deposit for several months.

The clarified varnish is decanted and the residue is filtered through hot filtering plant.

If the cooled varnish after standing appears to be too thick, it must not be diluted by the simple addition of spirits of turpentine, but be thinned by adding spirits of turpentine well mixed in the hot state with a certain quantity of the cold varnish. If, on the contrary, the varnish be too thin, it is reheated to drive off the excess of solvent or a thicker varnish is added to it.

THE DIFFERENT VARNISHES YIELDED BY DIFFERENT RESINS.

Varnishes may be divided into three classes: carriage, house decoration—interior and exterior—and varnishes for industrial purposes.

The different varnishes yielded by individual resins have now to receive attention, and the maximum and minimum proportions of the different ingredients formulated, leaving it to the manufacturer to choose the relative proportions which will best accomplish the object he has in view; the same remark applies to the amalgamation of different resins entering into the composition of mixed varnishes and introduced for the purpose of correcting any faulty characteristic of the predominating resin.

AMBER OIL VARNISHES.

These are the hardest varnishes known, but, on the other hand, they are wanting in elasticity; they are accordingly used to cover surfaces little subject to vibration or fluctuating movements; for instance, they answer remarkably well for such comparatively inflexible substances as glass. They are generally dark in colour owing to the high temperature at which amber melts and owing to the different pieces not melting at the same temperature.

The relative proportions of the ingredients usually taken in the preparation of this varnish are the following :—

Fused amber	1 part
Boiled linseed oil	0·8 to 3 ,,
Spirits of turpentine	1·0 to 2·5 ,,

The quantity of linseed varies from 0·8 if hardness be required, to 3 parts when elasticity is a desideratum. The quantity of spirits of turpentine is regulated by the degree of fluidity required. In certain cases, if hardness and transparency be required in preference to elasticity, and if at the same time a quick-drying varnish be desired, the proportion of amber is largely increased, and a very volatile solvent, such as benzene, is employed. We might take, for instance, fused amber :—

Fused amber	1 part
Linseed oil	$\frac{1}{4}$,,
Benzene	$\frac{1}{2}$,,

Finally, in practice it is often advantageous, whilst taking advantage of the hardness of amber and with the view of increasing its elasticity, to replace a part of the amber by copal. A hard, durable, elastic varnish is obtained by taking

Fused amber and copal, equal parts	1 part
Boiled linseed oil	1 ,,
Spirits of turpentine	2 ,,

In cheaper varnishes the whole of the copal in the above may be replaced by carefully selected higher-grade resin.

Golden oil varnish, into which amber enters, is made as follows :—

$\frac{4}{5}$ amber ($\frac{1}{5}$ seed lac)	1 part
Linseed oil	0·8 ,,
Spirits of turpentine	1·6 ,,

The coloration is imparted by a solution of dragon's blood, gamboge, annatto and saffron, in spirits of turpentine.

For 200 lbs. amber the solvents may be varied as follows, according to the body, etc., desired in the resultant varnish :—

Raw Linseed Oil. lbs.	Spirits of Turpentine lbs.
190	390
150	370
100	300
Boiled Oil.	
100	290
70	270
40	250

FUSED AMBER (PYRO-SUCCIN) VARNISHES.

For every 500 lbs. pyro-succin—fused amber—the driers and solvents may be varied as follows :—

	Quality of Amber.	Double Boiled Oil. lbs.	Black Oxide of Manganese. lbs.	Spirits of Turpentine. lbs.
Coach varnish	1	750	17½	1200
Coach iron work	2	750	17½	1200
Furniture I.	1	130	10	570
Furniture II.	3	130	10	750
Final coat	1	170	10	780
			Mn. Borate.	
Table (I.)	0	120	7½	700-730
Table (II.)	3	240	7½	1600-1700
Tinware	Prima	180	7½	850
Floor I.	—	150	10	780
Floor II.	—	120	7½	700-750

ROSIN AND AMBER VARNISH.

	Parts.
Fused amber	81
Boiled oil	100
Rosin—window glass	16
Spirits of turpentine	100

COPAL : OIL VARNISH.

Oil varnishes made from hard copal have a remarkable lustre, very great transparency, and are distinguished for their elasticity. They are specially adapted for prized articles decorated with fine delicate oil colours : upon white polished surfaces they look like glass.

The beautiful hard copals of the east and west coast of Africa yield very bright pale varnishes because they contain very little foreign organic matter susceptible of carbonising during fusion.

The oil varnishes from the semi-hard copals are more brilliant than the preceding, a fact due to their melting-point being lower; they are generally but slightly coloured except in the case of certain Asiatic copals, which contain a notable proportion of foreign organic matters which carbonise on fusion; they are less durable and resistant.

A good copal varnish ought to be thick, of a bright golden yellow colour; it ought to spread easily and without striæ, and to dry completely in six or seven hours.

In treating of the manufacture of oil varnishes in general, different processes for making copal varnish have been described, and it is therefore unnecessary to refer further to them here except to indicate a very special process, due to Andres, for the manufacture of a colourless varnish.

ANDRES' COLOURLESS COPAL (OIL) VARNISH.

Andres pulverises Indian copal very finely and dries in an air bath at 120° C. for several hours. To this powder there is then added its own weight of ground glass or very dry quartzose sand. Enough chloroform or petroleum ether is poured on the still hot mixture to cover it, and the whole allowed to stand in a well-closed flask for twelve hours. In contact with the chloroform the copal swells greatly, and becomes more apt to dissolve in other solvents. The contents of the flask are then run into a pan closed by a lid provided with an agitator, heated on the water bath and furnished with a delivery tube connected with a serpentine condenser (see Fig. 11, p. 180). The necessary quantity of spirits of turpentine is added and the water bath gently heated, so that the vapours of chloroform condensed in the serpentine fall back into the pan. After heating for half an hour to one hour the solution of the copal is sufficiently advanced, and the petroleum ether or chloroform is distilled off. They are recovered without loss in a pure state, as spirits of turpentine does not volatilise at this temperature. Heat is continued from thirty to forty minutes to cause the turpentine to boil and dissolve the copal.

On the other hand, raw linseed oil as pale as possible is heated on the water bath to 100° C., and rendered drying by borate of manganese. The previous solution of copal in spirits of turpentine, cooled to 60° to 70° C., is ladled into the oil, stirring after each addition. The heat is withdrawn, agitation is kept up for twenty minutes, and then the varnish is run into large flasks. It is very pale and bright, and soon clarifies completely.

This varnish is colourless when dry, for the oil when well made bleaches completely as it oxidises. Moreover, there is no tendency to pinhole, etc., but it takes about twenty-four hours to dry.

Varnishes by Violette's Process.

Very pale and very durable varnishes from hard fused copals may be made by Violette's process by replacing the boiling process by simply heating by means of a steam coil in which water at 100° C. circulates, the mixture of oil and spirits of turpentine into which the copal dips by means of an arrangement previously described when treating of dissolving resins in closed vessels.

Relative Proportion of Different Ingredients.

The proportion of the ingredients of copal varnish vary according to the purposes for which they are to be employed and the nature of the copal used. The greater the quantity of oil, the greater will be the elasticity, but the oil added ought to be in itself very elastic after oxidation, and the best results are obtained, as in the case of certain English varnishes, by boiling slowly for a very long time, if need be for several days, without the addition of drier and at such a temperature that they do not darken; this process has, however, been described whilst treating of the boiling of linseed oil. In this case it is only when the varnish is manufactured that a small quantity of liquid drier is added.

The proportions for copal oil varnish vary between the following limits:—

	Outside Decoration. Parts.	Interior Decoration. Parts.	Carriage. Parts.
Hard copal - - - -	100	100	100
Raw linseed oil - - -	30 to 80	25 to 50	150 to 200
Spirits of turpentine - -	130 to 240	250 to 300	100 to 150

Very elastic varnishes prepared by the mixed process:—

	Parts.
Hard copal - - - - - - -	100
Linseed oil - - - - - - -	250 to 350
Spirits of turpentine - - - - -	130 to 300

These very elastic varnishes are particularly adapted for japanned work which has to be wrought after being stoved.

Less durable oil varnishes made from semi-hard copal are principally used for interior house decoration.

	Parts.
Copal (semi-hard) - - - - - -	100
Linseed oil - - - - - - -	16 to 80
Spirits of turpentine - - - - -	230 to 250

The proportion of linseed oil varies with the hardness of the copal used.

ZANZIBAR COPAL (OIL) VARNISHES.

For 100 lbs. of resin the solvents may be varied, according to the body and consistency of the varnish desired, as follows:—

Copal.		Old Tanked Oil. lbs.	Spirits of Turpentine. lbs.
W. A.	1.	200	350
,,	2.	150	375
,,	3.	75	300
W.	4.	150	350
,,	5.	120	410
,,	6.	100	300

WEST AFRICAN COPALS.

Solvents for 100 lbs. of resin :—

Copal.	Old Tanked Oil. lbs.	Spirits of Turpentine lbs.
Angola - - -	200	350
	150	300
	110	280
Benguela - - - -	190	360
	140	300
	100	270
Pebble - - - -	180	360
	150	310
	100	270
Glass - - - -	190	360
	140	290
	100	270
Sierra Leone - - -	180	340
	130	280
	110	280
White Angola and Benin -	170	330
	130	280
	100	270
Akra - - - -	170	340
	140	290
	100	270

Working with "boiled" oil and dissolving the fused resin therein the solvents may be varied as follows for red Angola, Benguela, Pebble, Sierra Leone and Benin copals :—

Boiled Oil.	Spirits of Turpentine.
80	250
60	240
40	240

KAURI COPAL OIL VARNISHES.

For every 200 lbs. of gum the linseed oil and spirits of turpentine may be varied as follows :—

Raw Linseed Oil lbs.	Spirits of Turpentine. lbs.
200	500
150	400
100	350
"Boiled" Oil.	
80	280
70	270
40	240

MANILLA COPAL AND BORNEO COPAL OIL VARNISHES.

According to the nature of the varnish desired, for every 200 lbs. of resin the linseed oil and spirits of turpentine may be varied in quantity as follows:—

	Raw Linseed Oil. lbs.	Spirits of Turpentine. lbs.
1.	250	420
2.	225	400
3.	200	400
4.	150	350
5.	100	325
6.	70	300
	"Boiled" Oil.	
7.	75	300
8.	60	290
9.	40	250

In formula 1, hard Manilla alone is prescribed but is excluded in 2, whilst Borneo alone is used in 7. In the others either hard or soft Manilla or Borneo may be used.

REBS' STOCK VARNISHES.

Rebs uses Zanzibar copal for *very* hard varnishes, Akra, Angola and Benguela for *medium* hard, and Sierra Leone, Kauri and Manilla for *ordinary* hard varnishes.

From these resins he makes six classes of "mother" varnishes:—

No. 1 from Zanzibar or Madagascar.
,, 2 ,, Akra, Benguela or Angola.
,, 3 ,, Sierra Leone, Kauri or Manilla.
,, 4 ,, *débris* from No. 2.
,, 5 ,, *débris* from No. 3.
,, 6 ,, dust from 1 and 3.

The different ingredients are prepared as follows:—

Linseed Oil.—This is heated without driers until it deposits mucilage and clarifies, after which it is tanked for three to four weeks before use.

Manganese Boiled Oil.—One hundred parts of oil are slowly heated to 180° C. and 2½ parts of manganese borate dissolved therein for slow and 5 parts for quick drying oil, after which it is tanked for use.

Litharge Boiled Oil.—One hundred parts of linseed oil are slowly heated to 180° C., and at this temperature there is added in small quantities at a time 5 parts of litharge. The litharge dissolves in the oil between 180° and 190° C., and must be kept from settling at the bottom by constant agitation.

The oil is finally heated to 220° C., and when cold tanked for use.

"Mother" Varnish I.

	Parts.
Copal No. 1 - - - -	1
Linseed oil - - - -	3 or
Manganese boiled oil -	3
Spirits of turpentine - -	1 to 2

"Mother" Varnish II.

Copal No. 2 - - - -	1
Linseed oil - - - -	1
Manganese boiled oil -	2
Spirits of turpentine - -	1 to 2

"Mother" Varnish III.

Copal No. 3 - - - -	1
Manganese boiled oil -	1
Linseed oil - - - -	⅓
Spirits of turpentine - -	1⅓

"Mother" Varnish IV.

	Parts.
Copal No. 4 - - - - -	10
Manganese boiled oil - -	6
Litharge boiled oil - - -	5
Spirits of turpentine - -	20
Siccative - - - - -	½

"Mother" Varnish V.

Copal No. 5 - - - - -	10
Litharge boiled oil - -	6
Spirits of turpentine - -	20
Siccative - - - - -	½

"Mother" Varnish VI.

Copal No. 6 - - - - -	10
Litharge boiled oil - -	6
Spirits of turpentine - -	2

Mixed Varnishes.

These are generally made by adding a certain quantity of Venice turpentine to hard copal.

Polychrome Varnishes.

Very often different colours are imparted to varnishes for metals, particulary tin, by aniline dyes. But these having a very feeble resistance, they are advantageously replaced by metallic compounds, principally green copper-salts.

According to Lohmann, 30 parts of finely pulverised acetate of copper are heated on the water bath until a bright, brown powder remains; this powder is mixed with double its weight of spirits of turpentine and heated to 75° C. There is then added 100 parts of good copal varnish. If the acetate of copper has been pulverised finely enough, it rapidly and almost completely dissolves. After standing, a deep green varnish is obtained.

To impart to tinned objects a beautiful green colour four or five coats of this varnish are laid on. If only two coats be applied, and if the articles be placed in a stove, there is obtained, according to the temperature, different shades of colour varying from golden yellow to deep yellow and from greenish yellow to orange yellow. The same golden colour may be obtained upon glass.

DAMMAR OIL VARNISH.

Varnishes made from dammar or soft copal (copal tendre) are soft, very flexible and transparent, but dry rather slowly. They are employed for inside work or upon metals, or on tissue paper, which they render transparent. These varnishes have a bright appearance and a faint pale yellow coloration. The colour may be varied from golden yellow to yellowish brown by gamboge, dragon's blood and asphaltum of Judæa, and coloured in this way they enter into the composition of certain English varnishes used for metallic boxes and cases.

The proportion of the different elements vary between :—

	Parts.
Dammar - - - - - - - -	100
Boiled linseed oil - - - - - -	50 to 120
Spirits of turpentine - - - - -	200 to 500

The dammar is pulverised and dissolved in the hot in spirits of turpentine, and boiled linseed oil or linseed oil to which a liquid drier has been added is run into the solution.

If the harder kinds of dammar be used, they may be rendered more soluble in linseed oil by the addition of a little (about $\frac{1}{10}$th) of very soft dammar.

SHELLAC: OIL VARNISHES.

These varnishes are very brilliant, but they scale off very easily, hence their use is very limited. They are prepared by boiling linseed oil to which has been added for every 100 parts of oil 1 part of shellac, and as a drier a mixture of 4 parts of litharge, 4 parts of red lead, 2 parts of sugar of lead, 2 parts of acetate of lead, and 1 part of sulphate of zinc. After cooling the whole is thinned down with 80 parts of spirits of turpentine. The varnish thus prepared is generally applied by dipping.[1]

A varnish of better quality is prepared as follows:—

10 parts garnet shellac.
3 parts balsam of copaiba.
3 parts boiled linseed oil.

The mixing is done in the hot; after cooling the whole is dissolved in 100 to 150 parts of spirits of turpentine according to the consistency desired.

Blackboard varnish is also made from shellac, but the spirits of turpentine is replaced by alcohol and linseed oil by a liquid drier. It is in fact more of a spirit varnish with an oil drier. The following formula gives good results:—

	Parts.
Shellac	80
Paris black	80
Paris blue	5
Burnt umber	40
Liquid drier	100
Alcohol	700

This varnish scales off easily and is sometimes too brilliant. This may be remedied by the addition of ground pumice, white lead and a little Venice turpentine.

MASTIC, SANDARACH AND ELEMI OIL VARNISHES.

These different resins are only used in mixed varnishes, generally in those with a copal or amber base.

However, oil varnishes are met with which only contain mastic, and employed principally for retouching pictures. Their composition is as follows:—

Mastic	1 part
Walnut or poppy-seed oil	9 parts

Spirits of turpentine *quem sir* according to fluidity desired.

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[1] This is nothing more or less than a japanner's gold size.

The following varnish may be used for application upon wood:—

Mixture of	Parts.
Sandarach - Mastic and Rosin	100
Boiled oil	200
Spirits of turpentine	150

Coloration is imparted by dragon's blood or umber. When these varnishes are added to oil varnishes, such as copal or amber, the following are the proportions used:—

	Parts.		
Amber	100		
Mastic	30		
		Elemi	20
Or Sandarach	10	Or Mastic	10
		Venice turpentine	10
Boiled oil	50 to 70		
Spirits of turpentine	90 to 150		

Rosin: Oil-Varnishes.

On account of its cheapness and the ease with which it dissolves, rosin enters into the composition of many common varnishes. In the preceding formula it often replaces a certain part of gum resins.

Very beautiful colourless varnishes may be made by using rosin only, provided choice qualities are used, as pale as can be obtained, and by effecting solution in the cold. It is also advantageous to use linseed oil which has been boiled for a sufficiently long time without the addition of driers.

The proportions generally used are:—

	Parts.
Rosin	100
Linseed oil	25 to 30
Spirits of turpentine	125 to 180

A certain portion of the spirits of turpentine or the whole of it is sometimes replaced by naphtha. For certain small articles, such as boxes for confectionery, snuff-boxes, etc., a simple solution of resin in linseed oil is used; several successive coats are supplied, placing the article in the stove after each coat. The proportions are:—

Rosin	1 part
Linseed oil previously boiled	9 to 8 parts

Plaster casts are hardened and rendered unalterable by atmospheric agents by this last composition. The casts are well dried and heated to 80° to 90° C., are dipped into this solution, dried in the air, again dipped, and finally dried completely in the air.

Mixed Varnish.—A little more elasticity may be imparted to resin varnishes by replacing a part of the rosin by Venice turpentine or mastic, and by increasing a little the quantity of oil, the proportions in such cases being as follows:—

	Parts.
⅔ Rosin + ⅓ Venice turpentine	100
Linseed oil	60
Spirits of turpentine	120

ANTIFOULING COMPOSITION.

A quick-drying varnish suitable for antifouling compositions may be made by dissolving rosin in half its weight of shale naphtha. To this is added an equal bulk of japanners' gold size, in which spirits of turpentine has been replaced by shale naphtha. Finally, to the above mixture, an equal bulk of a solution of resinate of lime or resinate of copper or an admixture of both dissolved in their own weight of shale naphtha is added, and finally the requisite amount of colour added along with the toxic principles if any.

ROSIN VARNISHES.

	I. Parts.
Rosin	220
Litharge boiled oil	70
Spirits of turpentine	140

The rosin is first melted and then the 7 parts of quick-drying litharge boiled oil added, and the whole "boiled" for an hour at 190° C., when the mass is cooled and thinned down with the spirits of turpentine which may be replaced by shale or petroleum naphtha.

	II. Parts.	III. Parts.
Rosin	220	260
Shellac	10	5
Black oxide of manganese	10	20
Litharge boiled oil	60	30
Spirits of turpentine	140	180

The black oxide of manganese is ground and made into a paste with the litharge boiled oil, which is gradually added to the fused mixture of shellac and rosin, and the whole boiled for 1½ hours at a temperature of 190° C., after which it is cooled and thinned with the spirits of turpentine or by benzene.

	IV. Parts.	V. Parts.
Rosin	220	100
Dammar	—	100
Boiled oil	70	50
Naphtha	200	180

COPAL VARNISH SUBSTITUTE.

One hundred and twenty-five parts of "good strained" rosin are heated to 180° to 190° C., and 5 parts of either litharge or manganese borate added thereto, and the whole heated until the temperature rises to about 230° C. and a drop hardens on a glass slab so much that it can scarcely be removed therefrom. There is then introduced into it 20 parts of either litharge or manganese boiled oil containing 5 per cent. of either drier, and the heat continued for another hour, after which it is taken from the fire and thinned down with 100 parts of spirits of turpentine and filtered, and then ½ to 1 per cent. of copal oil added. This varnish dries quickly, yielding a very hard, smooth, highly polished coat.

GALIPOT VARNISH.

Three parts of galipot are dried at 160° C. and dissolved in 1 part of warm spirits of turpentine and ½ per cent. (of the weight of the galipot) of borate of manganese added. To every 100 parts of the finished varnish 4 parts of manganese boiled oil are added.

A sample of the varnish is cooled in a test tube for fifteen minutes, and if it remains clear the varnish may then be stored without further treatment.

VENICE TURPENTINE (OIL) VARNISH.

Solid turpentine plays an important part in the manufacture of oil varnishes, imparting elasticity to them, but is only used in conjunction with other resins. No oil varnishes properly so called are made with solid turpentine as the only basis; they would not have sufficient resistance. However, an undiluted oil varnish, with Venice turpentine as the sole base, is used to cover surfaces upon which real gold or silver leaf is afterwards to be applied. The formula is:—

Venice turpentine	1 part
Linseed oil	2 parts

In the following mixed, very elastic varnish the proportion of Venice turpentine predominates:—

	Parts.
½ Venice turpentine + ½ of a mixture of rosin and mastic	100
Linseed oil	30
Spirits of turpentine	270

ASPHALTUM AND PITCH (OIL) VARNISHES.

These varnishes are principally employed for metallic surfaces, leather and certain articles in ebony.

ASPHALTUM VARNISH.

When a thick varnish which will effectually protect the surface of the object is required, the following proportions are used:—

	Parts.
Asphaltum	1
Boiled linseed oil	3 to 6
Spirits of turpentine	2 to 4

The asphaltum is generally heated with $\frac{1}{20}$ of its weight of balsam of copaiba and the hot linseed oil added, and then, after suitably cooling, the spirits of turpentine.

According to Livache, when a beautiful black colour is desired, a coat of ivory black paint is first applied, then a coat of thin varnish made as follows:—

	Parts.
Asphaltum	1
Boiled linseed oil	300 to 400
Spirits of turpentine	2000

Mixed Asphaltum Varnishes.

The best are generally those with an amber or copal basis. Super-black Japan is such a varnish with a copal base.

When asphaltum is mixed with amber there are taken:—

	Parts.
Asphaltum	100
Amber	50
Boiled linseed oil	275
Spirits of turpentine	250

More durable varnishes are made by using the following proportions:—

	Parts.
Asphaltum	100
Copal or amber	100 to 300
Boiled linseed oil	100 to 300
Spirits of turpentine	600 to 1000

It is advisable to boil the asphaltum for a sufficiently long time, sometimes for several days, to drive off the volatile products, which might render it dull.

Black Varnish for Iron Work.

I.

	lbs.	lbs.
Fused amber, No. 5	12	
Syrian asphaltum	12	8
Double boiled oil	10	6
Litharge	1	$\frac{3}{4}$
Spirits of turpentine	48	22
American rosin		4

II.

		lbs.
Fuse	Copal	12
	Rosin	30
	Asphaltum	30
	Yellow Wax	5
	Venice Turpentine	6

add

	lbs.
Rosin oil	12
Boiled oil	30

and thin down with

	lbs.
Spirits of Turpentine	30
Naphtha	30-45

ASPHALTUM VARNISH FOR LOCKS.

	Parts.
Asphaltum	1
Linseed oil	25
Spirits of turpentine	50

But as they dry slowly it is preferred to make them from the following proportions:—

	Parts.
Asphaltum	100
Linseed oil	5 to 16
Spirits of turpentine	50 to 60

BLACK JAPAN.

	Parts.
Raw linseed oil	25
Prussian Blue	3
Litharge	1½
Fused amber No. 5	18
Syrian asphaltum	18
Spirits of turpentine	90

The raw linseed oil, Prussian blue and litharge are heated together in an enamelled iron vessel and boiled with constant stirring until the mass assumes a syrupy consistency. Care must be taken to remove the pan from the fire as soon as the contents begin to froth and to replace it as soon as they subside. After boiling for ten to twelve hours the whole is allowed to cool over night so that the litharge may settle out.

The bluish black mass is now transferred to a large enamelled pan and the fused amber and asphaltum added. The pan is placed on the fire and the contents heated until the amber and asphaltum are completely dissolved, after which it is allowed to cool and then thinned down with 90 parts of spirits of turpentine and filtered through cotton wool.

Coal-Tar Pitch Varnishes.

These varnishes, so generally used to coat iron and woodwork, may be referred to two distinct types, according to the rapidity of drying required and the number of coats proposed to be given. The proportions are :—

	Parts.
Coal-tar pitch	1
Linseed oil	8
Spirits of turpentine	5
or	
Coal-tar pitch	100
Linseed oil	50
Spirits of turpentine	150

In asphaltum and tar varnishes spirits of turpentine is often replaced by coal-tar naphtha, or by petroleum or shale naphtha.

There has recently been manufactured in Russia a varnish which protects both wood and iron from the action of moisture by employing the petroleum tars obtained by distilling with steam the tars coming from the acid residues from the refining of petroleum, neutralised by lime, or extracted directly from a petroleum bitumen extensively distributed throughout the Caucasus, in the government of Koutais.

The proportions are :—

	Parts.
Tar	100
Linseed oil	25
Naphtha	125

It is mixed in the cold, and during the mixing process a little white lead is added.

RESINATE (OIL) VARNISHES.

For toys and tin boxes, etc., cheap and quick-drying varnishes are required. Now-a-days there have been substituted for the resins—even ordinary resins—resinates which are easily soluble in linseed oil, and of which some, the resinate of zinc in particular, is very durable.

These resinate varnishes are made by dissolving the resinate in linseed oil heated to about 120° C. and diluting with the necessary quantity of spirits of turpentine.

The resinates most usually employed are those of lead, zinc, manganese and lime.

A great number of aniline colours being soluble in certain resinates, various coloured varnishes may be made.

CAOUTCHOUC VARNISHES.

These are employed especially to render articles waterproof. The rubber should be dry, so that solution may be effected under good conditions. The rubber is cut into thin strips and placed in a store, where it is kept at 40° to 50° C. for several days. Moreover, the operation is generally started by "swelling" the rubber with one-half part of ether.

The proportions used are:—

Caoutchouc	1 part
Linseed oil	1 ,,
Spirits of turpentine	1 ,,

The rubber, which has been left in contact with ether for several days, swells and gives a fluid mass when heated on the water bath so as to drive off the ether; this whilst still hot is mixed with linseed oil and finally with spirits of turpentine.

Hancock dissolved the rubber in a cylindrical iron vessel capable of standing pressure, the lid being fixed by bolts and nuts. The vapour of the solvent passes into this vessel through a pipe leading from the still in which it is generated.

Mixed Varnishes.—Oil varnish may also be added to rubber so as to impart both durability and hardness. In these cases the proportions of oil and spirits of turpentine are increased, thus to 12 parts of copal varnish there is added a caoutchouc varnish consisting of:—

Caoutchouc	1 part
Linseed oil	6 parts
Spirits of turpentine	4 ,,

Miller's Rubber Varnish.

Caoutchouc	5 parts
Carbon disulphide	2½ ,,
Spirits of turpentine	5 ,,

The rubber is cut into very small pieces and digested with the carbon disulphide in a glass retort heated in a sand bath until complete solution is effected. The linseed oil is then heated, and whilst still hot added to the above solution, which after cooling is thinned down with the spirits of turpentine.

Winkler's Rubber Varnish.

Caoutchouc	6 parts
Rectified spirits of turpentine	12½ ,,
Rectified shale naphtha	15 ,,

The rubber is dissolved in the mixed solvents in a retort heated on a sand bath, and the recently warmed copal varnish is then added.

Champagnet's Rubber Varnish.

Caoutchouc	6 parts
Spirits of turpentine	50 ,,

The rubber is digested with a portion of the spirits of turpentine for two days without stirring, after which it is well stirred with a wooden spatula. When all the turpentine has been taken up the remainder is added. The digestion and stirring are continued until the whole is dissolved. Then a varnish is made as follows:—

Above solution	75 parts
Pale boiled oil	75 ,,
Finest pale copal varnish	100 ,,

These are well mixed and incorporated together by agitation and then digested on the sand bath until complete amalgamation and solution is effected.

Caoutchouc oil varnishes, with a certain brilliancy and harder, are made by adding resin, say :—

Caoutchouc	1 part
Resin	2 parts
Linseed oil	2 ,,

Gutta-Percha Varnishes.

These varnishes are used to waterproof paper or skins. As with rubber, the gutta-percha ought to be dried with care. The following proportions may be used :—

Gutta-percha	1 part
Linseed oil	9 to 12 parts

The solution is made on the water bath and filtered through linen.

Mixed Varnish.—A leather varnish is made by generally adding to the preceding a certain quantity of copal varnish, and the necessary fluidity imparted by the addition of naphtha, say :—

Gutta-percha	1 part
Boiled linseed oil	8 parts
Copal oil varnish	1 part
Naphtha	1 ,,

Characteristics of Good Varnish.

1. A varnish ought to be as pale in colour as possible, so that the tint or tone of the object to which it is applied be not appreciably altered.

2. It ought to be perfectly clear and transparent, and present no turbidity nor dulness.

3. Its consistency should be such as to allow of its being easily, smoothly and uniformly laid on with the brush at the ordinary temperature (15° C.). If the varnish be too viscous, coatings of unequal thickness are produced, and consequently the dried coat is not uniform. On the other hand, if it be too

limpid, it runs or flows too easily, and the too thin layer does not present the desired brilliancy.

4. One of the most important points connected with varnish is its drying properties. As soon as the varnish is applied to any surface, the volatile solvent evaporates, leaving a viscous layer of oil and varnish ; at this point care must be taken to prevent the deposition of dust upon the varnish, because the latter will fix it permanently. Little by little the oil oxidises and becomes converted into solid linoxin, which is incapable of fixing dust. However, the varnish is not yet completely dry, for it sticks to the hand when placed against it. By-and-by the layers underneath absorb oxygen at the expense of the more highly oxidised layers on the surface, and so on until the whole of the oil has attained its maximum point of oxidation and become perfectly dry, and no longer tacky to the touch even when the warm hand is applied thereto for some considerable time. The varnish is then said to have dried.

According to Andés, good quality varnish ought not to fix dust after four hours, and should dry completely in twelve hours. As regards varnish for outdoor work, the time is respectively six and sixteen to twenty-four hours. On the other hand, carriage varnishes must be allowed ten to twelve hours and two to five days respectively, according to the quality of the varnish.

A coat of varnish applied to an object ought to have the appearance of glass present, without bloom, bubbles or blowholes ; in a word, it should show no signs whatever of rapid deterioration.

Good quality varnish ought to be capable of being detached, even several months after its application, in the form of a thin, flexible, elastic pellicle. Should the layer, on the contrary, disintegrate into scales and dust, the varnish is of bad quality, too rich in gum resin, and the operator may be convinced that it will be rapidly attacked and destroyed under the action of all those extraneous influences included under the term "weather".

GENERAL RULES INFLUENCING THE QUALITY OF VARNISH.

In order to obtain an article exhibiting all the desirable qualities and characteristics just enumerated, it is necessary to pass in review all the predisposing causes which may act upon either the ingredients of the varnish or the manufactured article, so as to profit by those which tend to improve, and to eliminate those which have an injurious tendency.

The colour should be as pale as possible.—In the manufacture of varnishes from amber and copal as already indicated, care must be taken to use select parcels, and to "run" the resins with care and avoid as far as practicable all darkening of the resin. The oil ought likewise to be pale, and it is advisable to use oil which has been "boiled" for a long time at a low temperature, and as far as possible without driers.

Finally, the admixture of different ingredients ought to be effected with all necessary care, and at such a temperature that none of them char.

The varnish ought to be perfectly clear and transparent.—This important quality is obtained by filtration and clarification by aging; moreover it is evident that the nearer varnish is to the top of the tank in which the clarification by deposition is going on the brighter and better will be the quality, because the freer it will be from suspended impurities.

The presence of a large quantity of driers, even although originally soluble, in the varnish is one of the prevailing causes which militate against its transparency, because the excess of driers is often deposited as the varnish ages.

The varnish ought to have such a consistency as to be easily applied with the brush.—The desired result may be obtained by using the proper portion of solvent; nevertheless the greater the duration and regularity of the boiling of the mixture of resin and oil the more freely will the resultant varnish flow under the brush, and the more regular will the coating be.

Varnish dries in a shorter or greater length of time according to the nature of the object or article to which it is applied and the ingredients entering into its composition.

In the first place regard must be had to the consistency of the varnish; if in fact the desired consistency has been obtained with *too small a proportion of solvent*, there will be obtained, after evaporation thereof, a thick layer consisting of a mixture of resin and oil, through which the oxygen indispensable to the thorough drying of the varnish can only penetrate with difficulty, especially to the very bottom layers of the coating. If, on the other hand, *too much solvent* be used, the resultant layer will be too thin and will only very imperfectly cover and protect the article or object to which it is applied.

But the most important factor is the relative proportion of resin and oil; if the proportion of resin be too great, it will, as the varnish dries, very soon exclude the air, and the apparent quick drying of the varnish will result in the incomplete drying of the layers underneath. Accordingly too quick drying is not always a sure index to a superior quality varnish, the more so as an excess of resin, as will be seen in the sequel, diminishes the elasticity.

Lustre—The varnish when dried ought to have the appearance of glass.—The ingredient resin imparts this quality. The brilliancy depends upon the nature of the resin and the proportion thereof, and also to a certain extent upon the purity of the oil.

It ought to be flexible and elastic.—Flexibility is imparted by the use of certain very soft resins, but, over and above, this flexibility and elasticity depend to the greatest extent upon the drying oil, and the greater the purity of the oil and the more skilfully it has been "boiled" the longer will the varnish maintain its flexibility and elasticity unimpaired. Thus it is that copal varnishes, made from very hard copal, are made by the process known as the mixed process, and by the addition of a large proportion of drying oil. The greater length of time taken in drying is more than compensated for by the elasticity of the coating obtained. Moreover, this elasticity will be more permanent the less combined linoxic acid present, that is to say, the smaller the proportion of drier used in the "boiling"—a prominent feature of the practice followed in the mixed process.

The dried varnish should show no defects.—The chief defects presented by dried varnish are due either to faults in their manufacture or to their being applied under unfavourable conditions.

Too much Oil—Sweating.—Amongst manufacturing faults mention must be made of too large a proportion of oil; in such a case, some time after the application of the varnish, the surface presents a greasy aspect and the varnish is said to sweat. The same effect is produced when the surface on which the varnish is applied is not perfectly dry. This defect may be remedied by allowing the varnish to dry as completely as possible, so that it may sweat to its fullest extent. It is then rubbed down with an old soft rag or with very finely ground pumice and then a fresh coat of varnish laid on. Owing to the large quantity of oil in the harder and more durable varnishes they are most subject to this fault.

Too little Oil.—If the proportion of oil be not sufficient the varnish shells off, breaking into pieces of variable dimensions. This effect is due to the unequal expansion of the successive coats and to the want of elasticity in the varnish as well as to any excess of driers.

When consistency has been obtained by using too little solvent, the layer remaining after its evaporation is too thick and the surface yields a skin too readily, whilst the layers underneath still remain viscous, bend under their own weight, forming folds and wrinkles.

Too much Driers.—If too great a quantity of driers had been used in the manufacture of the varnish, and if it had not been allowed to clarify by aging sufficiently long so as to deposit all suspended particles, then each particle becomes the centre of an air bell, thus giving rise to striæ.

Mixed Varnishes.—If a mixed varnish be used consisting of an admixture of varnishes of different quality and manufacture, owing to their not being homogeneous the layer in drying pinholes, etc.

Manner of Application.—The conditions and circumstances under which the varnish is applied may also affect it. Without dwelling upon this point, altogether foreign to the principles which underlie the manufacture of good quality varnish, it is, however, advisable to indicate some of the most frequently recurring conditions which exert an injurious effect upon varnish as it is being applied and before it has time to dry.

Too low a Temperature.—Supposing, for instance, the varnish is applied at too low a temperature, then it spots owing to the oil congealing. The varnish is then said to *chill* or *freeze*. Again, on the other hand, should the surface upon which the varnish is to be applied not be properly prepared to receive the varnish, or if it had been soiled or greased by contact with the hands, an unequal coating results, and the varnish is said to draw or pull. Moreover, under the influence of heat or the sun's rays, the fatty matter expands and thereby prises up the superimposed layers. The varnish *blows* or blisters.

Blooming.—Finally, if moisture condense upon the still fluid varnish, the surface becomes coloured with a velvety surface analogous to that encrusting certain fruits, *e.g.*, the *blue berry*. In other words, the varnish blooms or turns *blue*.

Noxious Gases.—In conclusion, certain noxious gases, generated locally and met with in the surrounding atmosphere, impart a yellow colour to varnish. In the case of black varnish faked up with blue, as is often done, the yellow thus generated unites with the blue and imparts a decided greenish cast to the black.[1]

Varnish made in winter, owing to the cold temperature at the date of manufacture, is too fluid in consistency when applied in summer, and thus gives a too thin and a duller coat.

AGING AND MATURING OF VARNISHES BY STORING.

Varnish ought to be stored or aged in an atmosphere more warm than cold. Comparative experiments have shown that varnish aged in a warm atmosphere dries even three times as quick as that which has been stored in a cold atmosphere.

Moreover, the length of time that has elapsed since the date of the manufacture plays a very important part in the production of good varnish, and ought therefore to be very seriously considered. According to certain practical varnish makers, no manufacturer ought to put on the market any varnish until after it has been stored and thus aged for several months, and not only so, but the good qualities of the varnish would be still more enhanced by further storage for a couple of years.

Working in this manner, a quick-drying, elastic, brilliant

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[1] The yellow colour itself may be generated from the blue by the action of ammoniacal vapours inducing partial decomposition of Prussian blue. Ivory black faked up with Prussian blue should never be used in coach painting. Neither should coaches be varnished in the neighbourhood of a stable.

varnish is produced with no tendency to "pinhole," etc. This is said to be the cause of the superiority of certain English varnishes, which are often kept for three years in the factory before being sent out, and afterwards, in addition thereto, for several years in their depôts or branches, making altogether a lapse of five years or more between the date of their manufacture and sale.

When a varnish is to be manufactured for immediate use, it ought to be made of greater consistency than when it is to be aged by storing.

TESTING OF VARNISH.

The testing of varnish to be of any real value ought to be done under conditions analogous to those to which the varnish will be subjected to in actual practice, that is, in the condition of a thin and perfectly dry layer.

Such a layer should be obtained by pouring the varnish on a glass slab. The glass absorbs nothing, and drying therefore goes on uniformly at every point. Nevertheless it is advisable to make tests upon wooden panels, painted or stained with colours which the varnish should protect from any wear or tear to which such panels may be subjected.

The coat of varnish after it is thoroughly dry ought to adhere firmly to the surface, and should not become detached after several repeated shocks or vibrations. When a piece is detached by a knife it should be flexible and elastic. As the coat ages, it should not become dull, but preserve its primitive brilliancy without cracking or shrinking, shrivelling or blistering. Moreover, it should not turn yellow.

JARHNS' METHOD.

Lohmann describes a method originated by Jarhns, by which comparative data may be obtained, based upon exact measurements.

To a very smooth glass slab is fixed a framework of well-sized glazed paper, having exactly the same thickness as that of the coat of varnish which it is desired to obtain. The glass slab having been fixed perfectly horizontally, a quantity of varnish is run into the space confined by the paper, sufficient to cover that space. Another smooth glass slab, with carefully filed edges, is cautiously slid over the surface of the paper framework so as to eliminate the excess of varnish. When the edge of the slab touches the fourth side of the frame, the excess of varnish is sucked up by filter paper so as to prevent it running back. There is thus obtained a coat of varnish, enclosed within the paper framework, of uniform thickness and precisely equal to that of the paper employed for the purpose. The glass slab used to remove the excess of varnish is now removed and the varnish allowed to dry, after which, within the framework, it may be comparatively tested for brilliancy, transparency and hardness, resistance to the thumbnail, etc.

Testing Action of Weather Variations.

It is often useful to ascertain the action of variations of the weather upon varnishes. This may be easily done in the following way: The glass slab used in the preceding experiment is thoroughly dried and then heated for 48 hours in a hot air or water bath at 100° C., and during the interval successive currents of air are caused to pass through the bath to assist oxidation, and thereby cause as complete drying of the coating as it is possible to obtain. On the coat of varnish, treated in this manner, fresh determinations of colour, elasticity, etc., are made.

Testing Hardness and Elasticity.

Again the varnish may be tested as to its resistance and elasticity by the use of special apparatus. This consists of a small cylinder capable of being rolled over the preceding glass slab. The weight of the cylinder may be varied by placing in its interior small bars of lead. To the circumference of the cylinder are fixed two blunt knife edges. It will be easily seen that as the cylinder is rolled over the layer of varnish it will leave tracks corresponding to the knife edges, which will vary with the weight of the cylinder and the hardness and elasticity of the varnish. If the weight of the cylinder be such as to cause the knife to pierce completely through the layer of varnish, they will then roll upon the glass and at the same time afford valuable data as to the force with which the varnish adheres to the glass. The microscopical examination of the cracks or striæ thus produced, will, with a little practice, enable the operator to decide exactly the quality of the varnish submitted to the test. It is equally interesting, however, to know the composition of different varnishes, so as to recognise the variations in properties which may result by the use of different proportions of the ingredients of which they are composed. Here it is that chemical analysis comes in.

DETERMINATION OF SPIRITS OF TURPENTINE.

The proportion of spirits of turpentine cannot be determined by direct distillation. There always remains a viscous residue, due to oxidation, even if the varnish be distilled off *in vacuo*, or if after the addition of water it be distilled in contact with air.

MacIlheny's Process.—The most exact process is that of MacIlheny, which consists in distilling 25 grammes of the varnish in a 400 c.c. flask after adding 100 c.c. of water and some granulated tin to stop bumping. From 90 to 95 c.c. of the distillate are collected, and the spirits of turpentine after separation are measured. A correction must be made for the essence of turpentine retained by the water of 0·35 c.c. in volume or 0·3 grammes by weight for 90 c.c. of the distillate at 15° C. If in place of spirits of turpentine another solvent be used it is detected and determined either by direct distillation or by the preceding method.

If the solvent consists of a mixture of spirits of turpentine and benzol, the process is also conducted as before, and after the water is separated the spirits of turpentine is converted by means of nitric acid into substances soluble in water. The unchanged benzol is separated and weighed.

DETERMINATION OF OIL AND RESIN.

Attempts have been made to identify the oil and the resin by the ordinary methods of oil analyses such as iodine and bromine absorptions, but only very unsatisfactory and uncertain results have been obtained.

The simplest and most exact process consists in placing on a glass slab a certain quantity of varnish and when it is quite dry to break it up with an appropriate solvent. Working in this manner, linoxin being insoluble in all solvents, it is easy to dissolve the resin, and after evaporation to weigh exactly the two ingredients, linoxin and resin. From the weight of the linoxin the proportion of linseed oil may be deduced, calculating upon, and allowing for, an absorption of 15 to 16 per cent. of oxygen. The different properties of the separated resin may be studied by following the scheme given for the analysis of spirit varnishes, but it must be borne in mind that these properties may be modified to a certain extent as regards hardness, transparency, etc., by the presence of infinitesimal quantities of the incompletely volatilised solvent or by certain greasy products arising from the incomplete oxidation of the oil into linoxin.

The nature of the driers used is determined by analysing the ash.

Amongst the general chemical tests are the following:—

1. When agitated with water the varnish should show no acid reaction which would indicate the presence of fatty acids.

2. The varnish should be completely saponifiable by alcoholic potash, if otherwise, the varnish contains rosin oil or mineral oil.

3. The ash should be infinitesimal.

In some cases it may be of service to ascertain the degree or extent of oxidation which a varnish may have undergone in the interval between its manufacture and application. With this object in view Dr. W. Fahrion has described a process based upon the fact that the non-saturated fatty acids yield on oxidation oxyacids insoluble in petroleum ether and which can thus be separated from the saturated fatty acids or their oxidation products.

GENERAL OBSERVATIONS ON THE MANUFACTURE OF OIL VARNISHES. ENGLISH PRACTICE COMPARED WITH CONTINENTAL AND AMERICAN.

Varnishes of English manufacture have for a long time been regarded as superior to those made in France, Germany or Belgium. Moreover, at the present day, in spite of the improvements in manufacture adopted by certain French firms, many of their tradesmen rightly or wrongly prefer to use English varnish, in spite of its usual much higher price. It may therefore be interesting to ascertain what it was which, until the adoption of recent improvements, produced such differences in the quality of the manufactured products.

The raw materials used, resins, oil, driers, spirits of turpentine, would evidently be obtained under identical conditions in the different countries of manufacture. Consequently, the differences in the quality of the manufactured article must arise from different methods of selection of the raw material, their relative proportions, and the methods of manufacture adopted.

In the third place, the proportion of gum resin being relatively small, it will be readily perceived that the English manufacturer can, at the same cost, use such resins as Zanzibar and Sierra Leone copals in preference to Manilla, which is so often employed in the manufacture of French varnish, in consequence of its low price and because the best qualities only take their own weight of oil, a circumstance which dovetails in exactly with the French method of manufacture. By the use of the best grade of resins English varnish gains in durability and brilliancy—qualities due to the resins used.

Again, English varnish manufactories differ greatly from those of France and Germany from the point of view of the large output and the great amount of invested capital. It will therefore be readily understood that under such circumstances the highest class of raw materials may be used.

Linseed oil, for instance, is only used after being purchased and stored for a long time, and having therefore improved greatly in quality since purchased. Moreover, the gum resins, even after they have been carefully washed, garbled and assorted, either before exportation or by the gum washer or merchant who supplies them, may again, owing to the large quantity turned over, undergo fresh assortment, in which the lumps may be apportioned for any given quality of varnish.

Besides, the age of varnish from the date of its manufacture to the time of its use is a factor of prime importance. Now, English varnish makers, having capital and space at their disposal, can store their varnishes for several years before putting them on the market, a practice which can hardly be followed in the case of a small factory with but moderate capital and limited space.

The truth of these remarks in regard to the advantage of working on a large scale and with plenty of capital is vindicated by the fact that American factories established comparatively recently and who have adopted English methods, have already produced varnishes which will stand comparison with the most renowned English brands. This is further demonstrated by the fact that in recent years some French manufacturers have so far improved their processes as to successfully compete with varnish manufactured abroad.

The chief difference between the English and French method of working, consists in the relative proportions of the raw materials.

In France, oil varnishes are made with a very high proportion of resin to the quantity of oil used, after the latter has been strongly heated in presence of driers.

In England, a much smaller proportion of resin is used, but, on the other hand, English varnish contains a much larger proportion of oil. Further, the manufacture, which has already been described under the designation of mixed process, consists in adding to the resin raw oil, or oil which has only been moderately heated without driers.

Several advantages accrue from this method of working.

In the first place, linseed oil being much cheaper relatively than high quality resins, it will be easily understood that the cost of production of English varnishes will be proportionately less.

Secondly, oil being added to varnishes to impart elasticity after drying, English varnishes will have less tendency to crack and scale off. Besides, the oil being added in a perfectly fluid condition, the mixture is more homogeneous, and the method of boiling yields a product of great fluidity.

APPENDIX.

USEFUL EXERCISES.

1.—Verify the solubility of each resin and its varieties in the different menstrua given in table of solubilities and tabulate your results for reference, especially noting any very decided departure from the solubility given. Try the effect of adding a little camphor to the solvent, also the effect of mixed solvents.

2.—Make up a set of solutions of salt in water of such density that at 15° C. each resin floats in its respective solution and no more, the slightest addition of distilled or rain water causing it to sink. Take the density at 15° C. of each solution by Twaddell's hydrometer for liquids heavier than water Nos. 1 or 2, and table your result for each solution against its respective resin. Preserve each solution for future use in stoppered bottles and label distinctly.

3.—Take the density at 15° C. of all the oils and solvents used in varnish-making and also liquor ammoniæ by the hydrometer for liquids lighter than water with direct indications and tabulate your results. Note especially the difference between raw and boiled oil.

4.—Take the density at 15° C. of pure sulphuric, nitric, hydrochloric and acetic acids with Twaddell's hydrometer for liquids heavier than water. Repeat the experiments on dilute solutions. Convert the degrees Twaddell in each case into actual gravity by multiplying by 5, adding 1000, striking off the first three figures to the left and inserting the decimal point thus: 50° Tw. $= 50 \times 5 = 250$; $250 + 1000 = 1{\cdot}250$ specific gravity. Find the percentage of real acid from the gravity by means of the usual tables.

5.—Take the density as in 4 of alkaline solutions, such as solutions of caustic soda, soda crystals, caustic potash, carbonate of potash, and determine the percentage of real alkali from the specific gravity by means of the tables in use for the purpose and note results.

6.—Take the density at 15° C. of solutions of such salts as sulphate of manganese, acetate of lead, green vitriol, bichromate of potash, sulphate of copper, and from the density find the percentage of each salt present in its respective solution.

7.—Apply the Elaidin test to olive oil and all the oils at your disposal and admixtures of each with olive oil and tabulate your results.

8.—Apply the sulphuric acid spot to each individual oil and tabulate your results.

9.—Apply the perbromide of tin test to rosin oil and admixtures thereof with vegetable and animal oils and note results.

10.—Make up a solution of alcoholic potash, 80 grammes to the litre. Make a mixture of 10 parts rosin oil or mineral oil and 90 parts linseed oil. Weigh out 5 grammes of the mixture and transfer to capacious porcelain basin, add 25 c.c. of the alcoholic potash solution, evaporate to dryness with constant stirring on the water bath, dissolve in water, transfer to separating funnel, extract repeatedly with ether, distil ether extracts and weigh residue, which should equal as near as may be 0·5 grammes. This residue in the case of an unknown sample when multiplied by 20 gives the percentage of unsaponifiable oil in the sample. Test the residue with glacial acetic acid; if it dissolves it consists of rosin oil, if it remains insoluble, of mineral oil.

11.—Blow air through linseed oil both in the hot and the cold, with and without the aid of driers, using the ordinary laboratory foot blower.

12.—Repeat 11 on spirits of turpentine in the cold and without the addition of extraneous substances.

13.—Repeat 11 on a mixture of spirits of turpentine and raw linseed oil in the cold.

14.—Pass chlorine through linseed oil and note changes of colour.

15.—Pass dry hydrochloric acid gas through spirits of turpentine to saturation.

16.—Pass dry sulphurous acid through spirits of turpentine.

17.—Place filter paper moistened with spirits of turpentine in an atmosphere of chlorine. Note smoky flame and abundant deposition of carbon.

18.—Distil rosin and collect the distillate in a suitable receiver. Note the products, water, acetic acid, rosin spirit, rosin oil and residual pitch.

19.—Repeat experiment 18 on all the varnish resins at your disposal and register your results.

20.—Distil bone oil, collect and rectify the distilled naphtha and determine the solubility of the resultant pitch in various menstrua and compare the japan it yields with that obtained from other pitches.

21.—Prepare samples of each of the metallic resinates, linoleates, borates, acetates, etc., given on p. 303 *et seq.*

22.—Make litharge from metallic lead, red lead from litharge, orange lead from white lead, lead acetate from acetic acid and litharge, and basic acetate of lead from litharge and acetate of lead, and verify the chemical properties attributed to each.

23.—Make a sample of printers' ink.

24.—Make a sample of vulcanised oil, rubber substitute.

25.—Make samples of spirit varnish according to formula given under each resin and note merits and defects.

26.—Make samples of liquid driers, japanners' gold size, super black japan, Brunswick black, carriage varnish, oak varnish, etc.

27.—Make a sample of soluble pyroxyline from formula given, p. 74, and from the product you obtain prepare samples of collodion and celluloid varnishes as per formula on p. 208.

28.—Determine the iodine absorptions of linseed oil and spirits of turpentine, and also those of all the varnish resins and drying oils, as per instructions on p. 220.

29.—Boil linseed oil with a known quantity of lead and manganese driers. Determine the percentage dissolved by burning the oil, weighing and analysing the ash.

30.—Estimate the percentage of spirits of turpentine in an oil varnish by McIlheny's method, p. 382.

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