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TRANSLATED FROM THE GERMAN

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SCOTT, GREENWOOD & SON
"THE OIL AND COLOUR TRADES JOURNAL" OFFICES
8 BROADWAY, LUDGATE HILL, E.C.
1908

D. VAN NOSTRAND COMPANY,
NEW YORK.

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PREFACE

THE term "Mineral Wax," though properly relating only to ozokerite and its product ceresine, as well as the more recently discovered wax from lignite tar, is often applied to paraffin as well, the latter having been the original substitute for beeswax, though comparatively unimportant in relation to the cheap vegetable waxes available.

Our knowledge of the mineral waxes is of comparatively recent date, for though ozokerite was known one hundred and fifty years ago, it was used to only a very small extent, if at all, and the true mineral wax industry dates only from the early seventies of the last century. Paraffin was known and in frequent use some forty years earlier, but its employment on a large scale practically coincided with the commencement of the ozokerite industry, at which period is formed the only product obtained from that material. For these reasons it is not difficult to understand why general knowledge should be scanty on the subject of ozokerite, ceresine, and paraffin, in comparison with other industrial products, and why the literature of the subject is small. Hence it seemed advisable to treat these important materials, so extensively used in many branches of technology, in a thorough manner, sketching their methods of preparation, properties, and adulterations, and also to bestow on their uses and applications the attention they so richly deserve. The preparation of paraffin, ceresine, and more recently mineral wax, ranks among the great industries, whilst the manufacture of their products can be carried on with simple appliances; and the Author consequently hopes that his labours will prove useful both to the former and to the numerous branches of industry concerned with the production of chemico-technical specialties.

THE AUTHOR.

1908.

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MINERAL WAXES

INTRODUCTION.

AT a comparatively early period, beeswax was used for a great variety of purposes,—we need only mention the writing tablets of the ancients, the wax paintings of the Greeks, application to modelling, etc.,—whilst it afterwards played an important part for centuries in various religious functions, and was used to an equal extent for lighting purposes. No other material known at the time possessed the admirable qualities of beeswax, combining the ease with which it could be worked and the number of uses to which it could be put; and it was not until the early part of the last century that beeswax met with a dangerous rival in stearine for the manufacture of lighting materials. On the other hand, the hardness and deficient elasticity of stearine unfitted it for many other purposes, and consequently, although the consumption of beeswax for candle-making declined considerably, the general industrial progress led to an extension of its use, so that the price continued to rise, despite importation from producing centres. Even the introduction of paraffin wax, produced on a manufacturing scale from bituminous substances, especially bituminous lignite, shale, etc., could not entirely displace beeswax; for although the candles of the new material had a high illuminating power, the softer grades of paraffin wax used at first became flexible at a lower temperature than either beeswax or stearine, the candles also exhibiting a tendency to gutter—a defect that has not even yet been entirely removed. Other uses were, however, discovered for paraffin wax, and its consumption has increased very largely, especially since it was discovered that this wax can be recovered from the distillation residues of petroleum (lubricating oils), as well as from the tars obtained by distilling bituminous coals, etc. These petroleum distillates contain varying proportions of paraffin wax, according to their origin, and this paraffin imparts consistency to the distillates, together with a tendency to solidify at low temperatures, whereby the lubricating oils containing it cease to fulfil their purpose and give rise to great inconvenience when used for oiling machinery, vehicles, etc. As soon as paraffin was recognised as the cause of this tendency to thicken, suitable measures were taken to secure its elimination, and the quantities so obtained form a very large proportion of the whole supply of paraffin. On account of its indifference to the action of acids and alkalis, paraffin is used for such a large number of purposes that it is impossible to specify them all. Although it plays an important part in the manufacture of candles,—the defects of which are accepted in view of their low price,—very considerable quantities are also consumed for insulating purposes in electrical work; in the match industry (for impregnating the match sticks); as a basis for ointments and unguents in pharmacy; for making waterproof fabrics and papers; as a dressing to impart lustre, fulness, and softness to textiles; for covering the mouths of bottles; in the manufacture of lubricants; making leather softening preparations (artificial vaseline) and other lubricating greases; floor and shoe polishes; as a protective coating against acids and alkalis; as an insulator for readily oxidisable substances; in the saturation plant in sugar-making; in glass-making; for cooling baths, etc. etc.

Ozokerite is a hydrocarbon like paraffin, and is chemically identical with the latter, so that mixtures of the two cannot be separated into their components, the only difference between them being that ozokerite is found native. It was first discovered in 1736 on the coast of Finland, but though it was afterwards found occasionally in other places, the earliest output on a large scale was only begun in 1860 at the Boryslaw mines in Galicia. No special industrial importance was acquired by ozokerite until, in 1870, means were found to purify the unsightly raw product and convert it into refined ceresine, which bears a considerable external resemblance to beeswax and also possesses a certain amount of plasticity. The following report was drawn up with reference to ozokerite and ceresine by Dr. Heinrich Schwarz, on the occasion of the Universal Exhibition at Vienna in 1873. "This material has recently come forward as a third powerful rival to beeswax (vegetable waxes were already well known). It occurs in pockets in the Salzthon formation, at Drohobycz and Boryslaw on the northern edge of the Carpathians in Galicia, and is won partly by open-cast working, partly by underground mining. Its occurrence is certainly connected with petroleum on the one hand and salt on the other. In the Galician section of the exhibition it was shown in the crude state, containing inclusions of fibrous gypsum and clear rock-salt crystals, and also in the condition in which it is obtained when freed from earthly impurities by melting and skimming. At an early period this dark brown, almost black, substance was used in Galicia for making candles, which gave a good light, although unsightly in appearance; but, when larger quantities were obtained, it was employed for making paraffin wax. Specimens of bleached ozokerite were shown at the 1873 Exhibition by a firm at Frankfort-on-Oder." At that time, according to Dr. Schwarz, the name ceresine was applied to a mixture of beeswax and more or less soft paraffin wax, exhibited by the Stockerau ceresine works, which firm afterwards manufactured ceresine in its present form. In this connection it may be mentioned that, although H. Ujhely's patent was dated 1879, the method of refining ozokerite with sulphuric acid was introduced by Dr. Berlinerblau early in the seventies. At any rate, Ujhely deserves the credit of having called into existence a great industry, which is not restricted to Austria, factories for the bleaching and refining of crude Galician ozokerite having also been established in Germany.

Since refined ozokerite resembles beeswax in appearance, and is also capable of replacing that substance in many of its uses, ozokerite not only finds extensive employment in the making of candles—which, by the way, lack the aroma of wax, especially on being extinguished — but is also used largely and for practically the same purposes as paraffin. Unfortunately, the popularity of ceresine has led to extensive adulteration, cheap brands, consisting of ceresine melted with paraffin and colophony, being placed on the market at prices far below those of the genuine article, which they consequently displace unless in exceptional cases.

Mountain or mineral wax is a new product, which has only been introduced within the last four or five years, and is obtained by extracting dry lignite with volatile hydrocarbons, which are then expelled by distillation. The crude article is of a blackish brown colour, and must be bleached in order to obtain a white product. It differs from paraffin and ozokerite in being composed of an acid that is completely saponified by alkali and of a hydrocarbon that is entirely carbonised by concentrated sulphuric acid. It can be put to the same uses as the hydrocarbons already mentioned.

CHAPTER I.

OZOKERITE AND CERESINE.

OZOKERITE, which has undoubtedly been known for a very lengthy period, is a product of the conversion of animal remains into hydrocarbons, and is in fact a solid and more or less plastic hydrocarbon, belonging to the group of bituminous substances—a collective name for solid fossil hydrocarbons. Numerous varieties are found in Nature, differing both in appearance and composition, but especially in point of purity and melting-point, the latter ranging from 50 to 100° C. It is mostly found associated with petroleum, and no doubt has been used for a very long time in a manner similar to beeswax, which it resembles in plasticity and combustibility, though no reliable data on this point are available. However, up to about the middle of the last century ozokerite remained a comparatively unimportant substance, the more so because the occurrence of the numerous deposits that would pay for working is chiefly restricted to two countries, Galicia and Moldavia. Ozokerite (Gr. odorous wax) is also known as mineral wax, mineral fat, mountain tallow, rock tallow, fossil wax, mineral adipocere, Citrisite (in Moldavia), Nestedigil (Caspian Sea), Baikerit (in Siberia). There are also a number of bitumins and resins closely allied to ozokerite, namely: Albertite or melanasphalt, Berengelite or Berengela resin, Branchite, Chrismatine or Chrismatite, Elaterite, Fichtelite, Könleinite or Könlite, Grahamite, Guayaquillite, Hartine (Xylorhetinite), Psathyrite, Hartite, Hatchettine, Hirein resin, Idrialine, Ixolyte, Middletonite, Posepnyte, Retene, Phylloretene (Retinasphalt), Scheerite, Tekoretine, Urpethite (according to Dr. Berlinerblau); but none of these has attained any importance, the quantities in which they occur being too small.

In his valuable work on the ozokerite mining industry in Boryslaw, Mück reviewed all the theories advanced as to the origin of ozokerite, and expressed his own views on the subject as follows: During the transformation of animal remains into petroleum, paraffin was formed at the same time and became dissolved in the oil. The heavy rock pressure in the depths caused this solution of paraffin to gradually rise in the deep rock fissures, filled with broken and pulverised material, the operation being attended with various changes, including a process of filtration between the petroleum and the paraffin. In consequence of this filtration, the petroleum ascended more rapidly, first impregnating the sandstone wherever possible, and the surplus ascending through the rock fissures up to the surface, where it was carried away by atmospheric agencies.

The residual paraffin in its upward course was subjected to additional filtration, resulting in the production of the very fatty grades of wax, as well as the dark kind. This also explains why the "lep" (wax, coating or impregnating the rock and sandstone) furnishes a dark greasy wax when boiled, since it contains the filter residues which have absorbed the dark resinous colouring matter. This concentration was accompanied by oxidation according to the amount of oxygen present, the process going on in two ways: on the one hand, by the direct absorption of oxygen to form the wax resins (colouring matter of the resulting ozokerite), or even as far as conversion into asphaltum; whilst on the other hand an elimination of the molecule of water may have taken place (according to Grabowski), which would account for the formation of more highly carbonaceous members of the methane series and separation of olefines. The final product of both these reactions is found in the case of marble wax.

During these processes the paraffin in its upward course was converted into pure ozokerite (ceresine), either as the result of prolonged pressure on the paraffin no longer in solution, or else from the amorphous, instead of crystalline, form of the paraffins resulting from the elimination of the elements of water. The aromatic constituents of the gas accompanying ozokerite are a remnant of the animal constituents of petroleum. How far the petroleum itself has sustained elimination of the elements of water cannot be ascertained, though probably to a far smaller extent than the more slowly ascending ozokerite. The fact that petroleum and kindebal are found in large quantities in higher strata affords proof of this, the deposition of this dark-coloured and highly contaminated paraffin proceeding in exactly the same way, as is observable any day in the pipe lines from the petroleum wells. Kindebal must therefore be regarded as an intermediate product of the conversion of paraffin into ozokerite.

Furthermore, oil sands are found only a few feet below the surface which were very rich in oil and were worked for a number of years in succession, the oil also containing paraffin. The percentage of paraffin in the oil from the deeper strata (down to about 1900 feet) is about 10 per cent., but the proportion contained in the upper oil is unknown, though old refiners, who formerly treated this oil exclusively, aver that it held much less paraffin than the oil now produced. This statement is also confirmed by Gasierowski. The oil from the higher horizon was darker and thicker, a condition attributable to more extensive oxidation. The migration of the ozokerite shows that its upward movement has not yet ceased.

With regard to the occurrence of ozokerite in the marine portion of the Neogenic formation (formerly known as the Miocene) in Galicia, and especially in and round Boryslaw, Mück says, in his above-mentioned work, that variable accumulations of this material are found in veins and fissures. Occasionally it would seem as though a smaller vein of wax were present inside the chief vein, and the distribution is so irregular, often disappearing altogether, that no one can foresee the yield of any working place from one day to another. Large accumulations of wax are generally found near the floor, the central portion of the vein being frequently barren and consisting of such a mass of apparently stratified clay shales, diversified by folds and streaks, that it is difficult to say whether it is standing rock or filling. In such cases ozokerite is more plentiful near the roof. The rock near the floor is frequently softer and crushed fine, thus forming a suitable opportunity for the ascent and deposition of the wax. The thickness of these veins of wax varies considerably, from a small fraction of an inch to about 20 inches. Some of them may be traced for a long distance, but generally they come to an end suddenly. Where pockets and lumps of wax are formed, the productivity of a working place is often suddenly increased, but may vanish just as suddenly. Moreover, the whole of the material constituting the vein, and known locally as "lep," is impregnated more or less with ozokerite, sometimes with petroleum as well, and almost invariably with gas, and must be treated for the recovery of the ozokerite it contains. Even apparently quite barren, dense hard clay shale will show a few hundredths per cent. of solid and gaseous hydrocarbons on analysis.

In the south-east and western parts of Boryslaw the veins diminish in size and are more of the nature of fissures, which in many cases are filled with ozokerite alone. The narrower the fissures the harder the wax, as a rule. In connection with these are beds of porous, fissured sandstone, containing large quantities of wax, mostly in the form of intrusive matter forced in between the bedding planes of the stone. Sometimes the corresponding fissure is absent, having been reclosed by rock pressure after the wax had been slowly pressed out. The largest accumulations of wax are found at the intersection of two veins; and wax is also found almost everywhere in the form of thin flakes between the layers of the hard clay shale. Owing to this circumstance, coupled with the occurrence of the beds and ignorance of the character of the wax veins, it was formerly believed that the wax occurred in seams, as it could be found all over the wax zone, though in variable quantities. A highly important matter for the ozokerite mining industry is the percentage of wax in the total mass raised from the mine, and long experience has shown that this quantity diminishes as the depth increases. The appearance of the so-called "matkas" accords with this phenomenon. When a fissure filled with soft wax is attacked with the pick, the wax is forced out into the shaft or heading by the pressure of the rock, and can be easily collected. The miners term such an exudation a "blower." It has occasionally happened that the soft wax (in this case kindebal) has been forced up to the shaft kerb. Formerly, when the horizons near the surface were being worked, these matkas were of frequent occurrence, but became rarer and smaller with the increasing depth, and now for several years past have ceased entirely. Another correlated phenomenon is the migration of the wax, fresh quantities of which have frequently been found exuding in abandoned workings. Mück examined one of these workings, and found, close beside residual fragments of hard yellow wax, others quite green and soft, that had apparently been forced up from below into the vacant space after the working had been discontinued. This migration upward has not been generally appreciated. In addition to the fluctuations in the amount of wax found in the deposits, there are also considerable differences in point of quality, the wax being generally harder in the upper horizons, and softer and of lower melting-point in the depths. Marble wax or

Boryslawite was often found down to about 110 yards, but seldom below that level and practically not at all below about 220 yards. The hardness and melting-point diminish progressively as the depth increases, and this inferior consistency of the wax facilitates its migration upward and explains the smaller percentage found in the lower workings. Kindebal, a soft, dark, plastic mass, chiefly paraffin, is evidently an intermediate product between petroleum and ozokerite. In many petroleum wells the pipes get stopped up by deposits of this product, a paraffin-like mass of pale yellow colour and undistinguishable from pale yellow ozokerite. Mück regards kindebal as an impure paraffin.

According to Dr. Berlinerblau, ozokerite occurs in Hungary, on the slope of a hill near the village of Pecora, on the river Iza, half-way between Dragomic and Szelistye. The conditions are said to be precisely the same as at Boryslaw. Ozokerite is frequently found in this district, and lumps have often been discovered in sand or the soil, as well as in the Miocene sandstone at Szascul near Nagybanya, a layer of sand containing ozokerite being found under a cover rock of sandstone.

Berlinerblau gives the following particulars of this product:—

The first ozokerite is black, soft, bituminous, with a smell like petroleum and a specific gravity of 0·900 and melts at 40·5° C. On distillation it furnishes—

10·12	per cent.	of light oils boiling at	90–150°	C.
9·04	,,	,, ,,	150–200°	,,
23·65	,,	heavy oils boiling at	200–250°	,,
42·16	,,	paraffin,		
11·80	,,	residuum,		
2·43	,,	gas and loss.		

Zsibo ozokerite is dark brown, soft, sticky, and gives off an aromatic smell when heated. Its melting-point is between 44 and 45° C. Dry distillation furnishes—

88 per cent. of oil and paraffin (containing 45·4 per cent. of pressed paraffin scale),
2·5 per cent. of water,
8·5 „ coke, and
0·7 „ gas.

With regard to Moldavian ozokerite from Slanik, the same authority gives the subjoined particulars: The product melts at 82° C. and, according to Magnus, consists of 84·61 per cent. of carbon and 15·305 per cent. of hydrogen. Glocke gives the specific gravity as 0·9555 (at 18° C.) to 0·970. Malagutti, on the other hand, gives the specific gravity of Zietriska as 0·945, the melting-point being 84° C. and the boiling-point 300° C. Moldavian ozokerite is very sparingly soluble in boiling alcohol and ether, readily so in oil of turpentine, naphtha, and fatty oils.

Russian ozokerite from the Caspian region is mentioned in the literature, under the names: nestgil, naphthyil, nestegil, naphthadil, nestetachil, and nestedegil, and these products were known at an even earlier period than Galician ozokerite. Apscheron ozokerite was known in early ages, whereas the deposits in the island of Tscheleken, on the eastern shore of the Caspian Sea, were first mentioned in 1838. Dr. Berlinerblau says about this material that, according to Herrmann, nestedegil is chocolate brown in colour, with a smell like petroleum, a splintery fracture, is plastic, and melts at 81° C. to an oily liquid, which apparently crystallises when cooled slowly. The specific gravity is 0·956. When provided with a wick it burns with a very bright, smokeless flame, and it has long been used for candle-making in China and Bokhara.

One hundred parts of nestedegil, when treated with boiling alcohol, furnish—

66·28	parts of	substance insoluble in alcohol,
17·77	,,	substance soluble in alcohol,
13·33	,,	resin soluble in alcohol,
2·62	,,	earthy impurities.

Oil of turpentine and petroleum dissolve nestedegil completely, whereas the greater part is insoluble in boiling ether. Distillation furnishes—

87·74	per cent. of a	buttery distillate,
4·28	,,	gas,
5·01	,,	carbon,
2·62	,,	earthy impurities.

The solid substance forming 37 per cent. of the buttery distillate was named kerone by Herrmann, and this substance, when freed from the oil by pressing, and recrystallised from boiling alcohol, furnishes a white, silky, crystalline, bituminous mass, changing to a crumbling plastic substance when melted and recooled. Kerone has the specific gravity 0·893, melts at 67° C., and is soluble in the proportion 1 : 0·4 in boiling alcohol. Concentrated sulphuric acid decomposes it in the warm, with liberation of sulphur dioxide and the formation of carbon, no paraffin distilling over. Beilstein and Wiegand isolated lekene from Caucasian ozokerite, together with an oil soluble in ether. According to these workers, Caucasian ozokerite is a brownish black sticky mass, almost completely soluble in hot benzol, from which mass almost the entire content of hydrocarbons is precipitated by treatment with alcohol.

A substance very similar to nestedegil, but somewhat softer and darker, is the ozokerite known as Baikerite or Baikerinite, occurring in large quantities in clefts in the rocks about Lake Baikal. In the cold it is as hard as wax, but ceases to crumble at 12° C., and is then plastic and greasy to the touch. It has a faint smell, which is different from the petroleum odour of nestedegil. The specific gravity is 0·92, the melting-point 52° C. It is completely soluble in boiling water, petroleum, and oil of turpentine, and also, except for 7·02 per cent. of residue, in boiling alcohol. A waxy substance separates out again on cooling. It distils over, with the exception of a small residue, when raised to a high temperature, and burns with a bright flame when provided with a wick. Herrmann's examination gave the following result:—

7·02	per cent. of	resin insoluble in alcohol,
60·18	,,	wax-like resin, soluble in alcohol,
32·14	,,	thick resin,
0·39	,,	earthy impurities.

The quantity soluble in alcohol is appreciably larger than with Galician ozokerite. The soluble waxy substance has the specific gravity 0·89 and melts at 74° C. The resin from Baikerite is as thick as tar at 15° C., and has a penetrating balsamic smell and sharp taste resembling wood-tar. At 10° C. it deposits crystals and has the consistency of honey. The distillate from Baikerite is buttery, and consists of kerone and burning oils.

On distillation, 100 parts of Baikerite give—

59·95	per cent. of	kerone,
35·33	,,	burning oil,
5·36	,,	gypsum,
5·36	,,	carbon.

The product is said to form an approved remedy for rheumatic pains (as is the case with ichthyol).

In Northern Finland rich deposits of ozokerite are said to have been discovered in 1901 by a Russian engineer on the shores of the Kemi-Joki (Torma district), the material being reported as of favourable composition and containing 6 per cent. of various resinous substances.

At Inhambane (Portuguese East Africa) a spongy form of ozokerite is said to have been recently discovered, and to furnish petroleum and the usual by-products. An analysis made in Johannesburg gave the following results:—

3·41	per cent. of	naphtha,
20·00	,,	kerosene (lamp oil),
12·50	,,	intermediate oils,
18·08	,,	lubricating oil,
4·16	,,	hard paraffin scale,
2·00	,,	soft paraffin scale,

a total of 60·64 per cent. of useful substances. A syndicate —"The Inhambane Standard Oil Syndicate"—was formed at Johannesburg in July 1904 to work the deposit, and founded "The Transvaal Inhambane Oil Wells Limited" at the beginning of December of the same year. The necessary machinery is said to have been shipped to Inhambane already, *viâ* Delagoa Bay.

In the United States, a deposit of peculiar ozokerite has been known to exist in Utah for the last twenty years, but its importance was not properly appreciated until quite recently. Three workable portions of the deposit have been discovered at the northern extremity of the Wisatch plateau, in the vicinity of Denver and of the Rio Grande railway. The chief centres are: (1) near Midway station, three miles west of Soldier Hill and at the source of Soldier Creek; (2) near Colton, seven miles south of Soldier Hill; and (3) near the railway crossing at Soldier Hill. The ozokerite is found in beds of sandstone and limestone, at the foot of the Tertiary formation of the Wassatch group, and the mineral has been proved for an extent of 500 feet. The strata are of various colours—greyish green, green, blue, and red; the sandstone is fairly soft, and light drab to brown in colour, whereas the masses of limestone containing the ozokerite are thin, and range in colour from white to yellow and blue. At the places mentioned, the rock is partially fissured and is traversed by parallel cracks in which the ozokerite is stored in the form of thin plates and occasionally in lumps an inch thick. The cracks are nearly vertical, and dip 10 degrees north-west in the vicinity of Colton and Soldier Hill. Half a mile west of Midway station the fissures containing the ozokerite are nearly vertical. The samples of ozokerite from the various centres have been thoroughly examined, and found to exhibit the same general physical characteristics, though deviations are not infrequent. In some places the ozokerite is smooth, but is generally somewhat brittle and readily crumbles, without being compressible between the fingers; and it can be cut with a knife, like hard cheese, without sticking to the knife. The colour is mostly black, with a yellow, resinous skin, and in some parts of the Midway deposit the mass is traversed by yellow streaks as though intermixed with some other substance. This ozokerite melts at 64° C. and is soluble in boiling ether; the smell resembles that of petroleum. The Colton ozokerite melts at 70° C., is inodorous, and only imperfectly soluble in boiling ether, whilst that obtained a mile and a half farther north is perfectly black, smells strongly of petroleum, can be kneaded between the fingers, melts at 54° C., and is completely soluble in boiling ether. It also contains more asphaltum than that from the other deposits. When heated, all three products behave like paraffin. According to Kroupa, who has investigated Utah ozokerite for the past twenty years, there are five mines in work and three sets of plant for separating the ozokerite from the associated rock. Borings have been carried to a depth of 740 feet, tapping several beds of ozokerite, occasionally as much as 3 feet thick. The recovery of the ozokerite itself is a very simple process, the whole plant consisting of a boiler, a mortar (or stamps), and steam-heated troughs. The ozokerite and rock are crushed, and the mass is turned into long troughs with conical bottoms, where it is heated with an addition of water. The ozokerite melts, separating from the crushed gangue (which falls to the bottom), and is run off, at a temperature of 54–70° C., into cooling vats, where it is kept hot and fluid for some time, to allow the contained water to evaporate. The treatment of the ozokerite rock does not pay unless it contains at least 10 per cent. of recoverable crude ozokerite.

Newberry found Utah ozokerite to contain 86·15 per cent. of carbon and 13·75 per cent. of hydrogen. Reports on the melting-point differ, Newberry giving it as 61·5° C., Seal as 53–55° C., Würz as 66° C., whilst the specific gravity is given as 0·9205 (Seal) to 0·934 (Würz). Berlinerblau states that Utah ozokerite is readily soluble in warm benzol, petroleum spirit, ether (?), and carbon disulphide; the diluted solutions exhibit strong fluorescence. Alcohol precipitates solid substance from the solution in benzol; and when the solution has been effected by successive portions, the bulk of the colouring matter is thrown down with the first fraction, leaving the solution more or less pale.

When Utah ozokerite is treated with concentrated sulphuric acid, at temperatures above the melting-point, an immediate liberation of sulphur dioxide occurs.

Ozokerite has also been found in the province of Mendoza (Argentinia), and Adolfo Fader of Mendoza gives the following particulars of this material, the quantity of which is small:—

Total distillate	80·32	per cent.
Residue (earth and coke)	12·72	,,
Loss (gas)	6·96	,,

Fraction No. 1 (100–150° C.), referred to total distillate	3·20	per cent.
,, 2 (150–300° C.), sp. gr. 0·853,	12·75	,,
,, 3 (up to red heat)	70·00	,,
Residue and loss	14·05	,,

The crude paraffin melts at 54° C.

The most important of all ozokerite deposits is that of Galicia, in the districts of Drohobycz (comprising Boryslaw, Wolanka, and Truskawiec) and Stanislau (Dwiniacz, Straunia, Wolotkow, and Niebylów). Of these, Boryslaw is the most important, the mining of ozokerite in the others being either of merely limited extent, prohibited altogether (as is the case at the watering-place of Truskawiec), or not pursued at all. Ozokerite is also found in Western Galicia (Klecany, near Neu-Sandec, and at Limanow in Modarka); at Sloboda Rungurska to the south-east of the valley of the Pruth; at Gorlice (unprofitable), and at Dolina (merely traces). The history of ozokerite mining at Boryslaw is a very interesting one, and has been attended by many unfortunate incidents. In 1821 and 1843 mining licences were granted for working "mountain tar"; and the first large discovery of ozokerite was made in 1854. The native peasantry confined themselves at first to collecting petroleum, which was sold in a thick state as "ropa" (cart grease), and in a more fluid condition as "kipiaczka" (leather grease). This oil was allowed to collect in shallow pits, and it was not until 1856 that a commencement was made with the sinking of shafts. About 1860, soft, plastic masses were found at a depth of 50–65 feet, but these were simply thrown aside in heaps. Soon the quantity of this substance forced out into the shafts by the pressure of the rock became so considerable as to prevent further sinking. The first use of the once discarded ozokerite was for making candles for the peasantry, but in 1862 it was successfully distilled for the production of paraffin. The discovery of this sphere of utility led to indiscriminate working, and very soon the whole district round Boryslaw was covered with shafts, some 1500 in number, and often only a few feet apart. The ozokerite was raised by the aid of simple winches, and the whole field was crowded with a throng of miners, sellers, and buyers. In 1865 Jicinsky estimated the number of shafts in work at 5000–6000, but only 165 of these were productive, the total output being fixed at 252 wagons (2250 tons). In 1881 the output for the second half-year was as much as 5000 tons, the total for the year being between 9000 and 10,500 tons. In consequence of legislative measures, the number of enterprises began to decline from 1885, the number of shafts in work being 820 for ozokerite and 530 for petroleum, within an area of barely 240 acres. On the introduction of Pilz and Ujhely's method of recovering ceresine from ozokerite, greater attention was devoted to ozokerite mining up to 1885, in which year the maximum output of 1230 wagons of melted ozokerite was attained, this level being approximately maintained until 1891, when another decline set in. In 1897 there were 270 shafts, but in 1900 only 18 in work. One chief reason for this diminution in the shafts was the greater working depth and the consequently increased capital required, the yield of ozokerite being also smaller and the profits less; besides which, many of the mine owners lacked the requisite knowledge to carry on the work systematically. The Mining Police Regulations of 1886 were followed by an Ordinance from the Department of Mines, which not only prescribed a minimum distance of 66 yards between adjoining shafts, but also insisted on properly qualified managers and other officers, thus putting the mining operations on a firm basis. One peculiarity of the Boryslaw ozokerite mining industry was the organised theft of ozokerite—a state of things that is said to exist at the present time to a small extent. The miners made a practice of hammering ozokerite out into flat cakes, which they could readily conceal beneath their clothing; and these cakes of so-called "Pazuchy" wax were accepted in payment at all the public-houses in the vicinity. Numbers of girls and children also walked about all day long seeking for lumps of wax between the shafts or on the waste heaps, and burglarious attempts on a large scale were by no means infrequent, the booty being disposed of to receivers, who melted it down and mixed it with ozokerite of undeterminable origin (*e.g.* from wax pickers) for sale. It may be taken as certain that within the last few years (up to 1903) at least two wagon-loads of ozokerite of doubtful origin were offered for sale every month; and formerly, when the whole circumstances practically invited theft, double the above quantity would be a moderate estimate; and it may be taken for granted that the evil was still more prevalent in earlier and more lawless times. In fact, it is estimated that, between the years 1862 and 1890, at least 1200 wagon-loads of wax, to the value of 6 million kronen (£240,000 sterling) were stolen and sold.

Merz, in his work on the technology of fats, etc., gives the following data on the specific gravity and melting-point of Boryslaw ozokerite:—

Olive green .	sp. gr. 0·9236,	melting-point 60·5° C.
Dark yellow .	,, 0·930	
Dark brown .	,, 0·930	

The results of distillation are given variously, as under:—

According to Merz—

Boryslaw olive green ozokerite furnishes—

6·25	per cent.	of light oils boiling below 150° C.
35·10	,,	heavy oils, with paraffin, boiling below 300° C.
49·73	,,	paraffin, boiling above 300° C.
8·92	,,	residue in the retort and loss.
100·00		

Dark yellow Boryslaw—

4·32	per cent. of	benzine, sp. gr. 0·710–0·750
25·65	,,	lamp oil, sp. gr. 0·783–0·820
7·61	,,	lubricating oils, sp. gr. 0·895
56·64	,,	paraffin, etc.
2·85	,,	coke
2·93	,,	loss
100·00		

Dark brown Boryslaw—

3·50	per cent. of	benzine, sp. gr. 0·710–0·750
27·83	,,	lamp oil, sp. gr. 0·780–0·820
6·95	,,	lubricating oils, sp. gr. 0·895
52·27	,,	paraffin, etc.
4·63	,,	coke
4·82	,,	loss
100·00		

Perutz obtained the following products from Boryslaw ozokerite :—

	(1) Per cent.	(2) Per cent.
Water	0·33	2·13
Benzine, sp. gr. 0·700–0·745	3·67	11·00
Benzine spirit	...	0·27
Petroleum, sp. gr. 0·750–0·820	5·67	...
Paraffin for crystallising	82·33	75·03
Pyrene, chrysene	2·05	...
Lubricant paraffin	...	3·29
Residue (coke), gas, loss	5·95	8·28

Pawlewski examined several grades of Truskawiec ozokerite. The commercial product is brown to black in colour, contains sulphur, and consists of: carbon, 84·46 per cent.; hydrogen, 14·00 per cent.; oxygen (by difference), 0·34 per cent. The following particulars are also given:—

	Yellow crude.	Green crude.	Commercial crude.
Specific gravity	0·9235	0·9237	0·9260
Melting-point .	64–65° C.	65–66° C.	65–66° C.
Ash . .	0·172 p. c.	0·37 p. c.	0·86 p. c.
Sulphur . .	trace	trace	0·37 „

Distillation furnished—

Water	1·70	per cent.
Paraffin at 150° C.. . . .	7·20	„
Oils at 150–320° C. . . .	30·00	„
Paraffin mass.	52·00	„
Coke and residue	4·80	„
Loss and gas	4·30	„

Burgmann reports as follows on the composition of Boryslaw ozokerite. Dark green Boryslaw ozokerite, free from kenderbal:—

	(1) Per cent.	(2) Per cent.
Water	0·33	2·13
Benzine spirit	...	0·27
Benzine, 0·700–0·745 . . .	5·67	11·00
Petroleum, 0·750–0·820 . . .	3·67	...
Paraffin mass (crystallisable) . .	82·33	75·03
Pyrene, chrysene	2·05	...
Lubricant paraffin	...	3·29
Coke and gas	5·95	8·28

As previously remarked, ozokerite varies considerably in appearance, and it is no exaggeration to say that to find two samples exactly alike from different mines is quite the exception. The colour varies to an extraordinary extent between pale yellow, dark yellow, more or less pure to dirty, brown and green of all shades, and black. In the Boryslaw field the following chief kinds of ozokerite are found:—

1. Marble wax. This is scarce and very hard; the fundamental colour is pale yellow, with greenish, brownish, or blackish inclusions, so that when a lump is ground, smoothed, and then polished, it exhibits markings like those of marble and in different shades. It furnishes about 30–40 per cent. of refined ceresine. In the melting process the darker portions dissolve in the yellow mass, and pale crude ozokerite can be obtained by careful sorting. Marble wax free from moisture has now become so rare that its only claim to consideration is as a mineral for the collector's cabinet.

2. Hard wax, darker in colour than marble wax, with coarsely granular, shaly fracture.

3. Fibrous wax is hard wax with fibrous fracture, and the structure is probably due to unilateral pressure.

4. Bagga is a very dark, almost black product, largely intermixed with lep wax. It has a low melting-point, and serves for the production of second-grade ozokerite.

5. Kindebal (kinderball, kenderbal, kunderball) is a soft black mass of low melting-point, mixed with oil and earthy particles, and forms an intermediate stage between petroleum and ozokerite. A similar product is found as a deposit in the pipes of the petroleum wells at Boryslaw, Schodnica, etc., and is also obtained by exposing petroleum containing paraffin to the air for several months at a temperature of 18° C.

6. Blower wax (matka) is a pale yellow ozokerite which is pressed out with considerable force by the rock pressure when the miners strike a fissure or vein. This kind has also become rare.

7. Lep is ozokerite mixed and greatly contaminated with gangue.

Apart from marble wax and blower wax, which, when occurring in any quantity, can be easily separated from the rest of the material, the mined ozokerite has necessarily to be raised along with the gangue with which it is contaminated, and is then subjected to further treatment. The pure fragments of ozokerite (known to the miners as "scale" wax) are separated from the rock in the mine and raised separately, the rest being brought up in the mixed condition and then separated by hand, no mechanical process having yet been found suitable for the task. In this treatment the scale wax is freed from adherent particles of rock, and small fragments of wax are picked out, the last traces of wax adhering to the rock being scraped off with knives and forming picked wax. The resulting products are: wax, pit smalls (washing lep), sandstone, and waste, these latter being cast on the waste heap, where they are occasionally sorted over again. The washing lep forms that portion of the mineral in which the ozokerite is present as lumps, nests, or thick veins, embedded in the rock, and it therefore contains small splinters and pieces of ozokerite. It is placed in large wooden vats, filled with water, and is worked about with large wooden paddles until all the fine powder or ash-like particles have been disseminated in the water. During this treatment the specifically light ozokerite rises to the surface of the water and is removed by means of a skimmer, to be washed over again for the removal of the earthy particles it contains. The contents of the vat are stirred up once more, and the floating wax skimmed off as before. An important part is played by the saline matters in the gangue, these increasing the density of the washing water and facilitating the ascent of the wax. In this manner about 0·4–0·5 per cent. of wax is extracted in the first washing, and 0·08 per cent. in the second washing, out of a total of

2 per cent or more in the lep. The recovered wax is next dried, and is melted to separate it from the numerous impurities—gangue, wood, etc.—it contains. The washing treatment is not suitable for all kinds of lep, especially those in which the gangue is either impregnated with the ozokerite, or contains the latter in the form of a thin film on the surface of the fragments; and in such cases boiling has to be resorted to. The lep is placed in a large pan into which hot water is then poured, and the whole is heated, without stirring, until the melted ozokerite rises to the surface of the water and can be skimmed off with ladles. When only a little ozokerite remains on the water, the contents of the pan are stirred for a time, and then left at rest, so that the ozokerite can rise without carrying with it more of the very fine gangue than can be avoided. Nevertheless, the wax obtained in this way still contains about 60 per cent. of finely divided mineral matter. Since it is essential to recover as much of the total ozokerite as possible, the wax obtained by scraping the lumps of rock on the waste heap, and also the waste (solójka) from hand-picking the scale wax, must be melted with hot water. After this treatment, the following products are available for further manipulation: (1) Scale wax, obtained by hand-picking; (2) more or less pure wax, either raised as such or in the form of scale wax separated in the mine; (3) washing wax obtained from the cold washing process; (4) wax extracted from the lep by hot washing.

Attempts to extract the ozokerite from these prepared materials by the aid of liquid hydrocarbons do not seem to have found application in practice, though it is not clear why such should not be the case, in view of the success attending the extraction of mineral wax from bituminous lignite. Mück obtained the following results in the laboratory by extracting ozokerite-bearing material from Boryslaw with benzine:—

		Soluble in Benzine.	
1.	Polojka from the scale-wax picking house	10·21	per cent.
2.	Rich lep from gangue	1·35	,,
3.	Poor lep ,, ,,	1·31	,,
4.	Apparently barren clay shale (krydowania)	0·03	,,
5.	Average sample of lep from the lep melt	0·82	,,
6.	,, ,, ,, washery	0·30	,,
7.	Residue from the lep melt	0·17	,,
8.	,, ,, upper washery	0·32	,,
9.	,, ,, lower washery	0·22	,,
10.	,, ,, wax melt	6·50	,,

It may be that a profitable method of extraction will yet be discovered, though it is evident that large quantities of solvent would be required for the treatment.

The further treatment of the prepared material consists in melting, either dry or with water, the former being preferable with pure materials and the latter when impure products have to be dealt with. Dry melting must be carried on at such a temperature that the ozokerite is not darkened in colour or decomposed, and its object is to keep the wax in a liquid condition long enough to allow the finely powdered gangue time to settle down, so that the liquid wax drawn off shall be fairly pure. In the wet-melting process the same object is in view, and the wax floating on the surface of the water is transferred to special vats in which the water and mineral matters can subside. From these vats the ozokerite is poured into the sheet metal moulds in which the ozokerite is marketed. According to Mück, the following brands are now put on the market as the result of careful grading and mixing the various melts :—

1. Prime special, pale green, dry, with merely a slight tendency to stick to the fingers, and melting at 68° C.

2. Prime, somewhat darker in colour, with a greenish sheen

on fresh surfaces of fracture, more difficult to detach from the fingers, *i.e.* somewhat sticky, and melting at 68° C.

3. Standard grade, dark brown to black, dry, easily rubbed between the thumb and finger, melting at 66° C.

4. Seconds, dark and greasy, melting at 52–56 or 60° C.

The above melting temperatures are not the actual melting points, but those at which the ozokerite solidifies, this change being more easily and accurately determined than the converse one.

The moulded ozokerite forms the raw material for ceresine manufacture, as also (occasionally) for the production of paraffin for the purposes of electrical insulation, and as an impregnating agent for waterproofing.

According to the most recent information, there were in Galicia in 1905 sixteen ozokerite mines, in Boryslaw, Kruskawiec, Dzwiniacz, and Starunia, finding employment for 2950 hands. The output of ozokerite in the same year was 2860 tons, value 4,348,000 kronen (£174,000), as compared with 2980 tons, value 4,584,000 kronen (£183,000), in 1904. Owing to the Mining Regulations of 1904, the working of the mines is said to be conducted on quite up-to-date lines; but the development of the industry is seriously hampered by subdivision of the land among a large number of small proprietors, thus rendering extensions and the establishment of new mining enterprises well-nigh impossible. The recovery of ozokerite and the trade in this product will therefore remain at about the existing level.

The exports of ozokerite from Galicia in 1905 totalled 1614 tons—

To Russia	137·8 tons
,, France	80 ,,
,, America, direct	121·2 ,,
,, Germany	1315 ,,

About 200 tons were shipped to the warehouses in Hamburg and stored there, the remainder being sent to Bremen, Düsseldorf, Hamburg, Cologne, Munich, Rixdorf, Stettin, and Geisslingen. The actual quantity disposed of was 1125 tons, and about 30 per cent. of this was shipped to Great Britain and America.

PROPERTIES OF OZOKERITE.

Ceresine, or refined ozokerite, forms white amorphous masses, in contrast to crystalline paraffin—greatly resembling beeswax in appearance. In the cold, ceresine has no appreciable odour, but smells like petroleum on being heated, the liquid at the same time having a faint bluish sheen. The specific gravity of ceresine is 0·915–0·925 (that of beeswax is 0·963–0·969), melting-point, 60–80° C. (beeswax, 62–64° C.), and setting-point 2–3° C. lower on the average. Ceresine with a melting-point of 75° C. has the same fracture and structure as beeswax. On exposure to the air, pure white ceresine darkens in colour.

Yellow grades of ceresine are either imperfectly decolorised or else are prepared by colouring white ceresine with vegetable or coal-tar dye-stuffs. The higher melting-point of ceresine causes it to be very hard, so that it cannot be kneaded and moulded so easily as beeswax. Ceresine can be melted along with vegetable and animal fats and oils, mineral oils, colophony and other resins, to form products, which are hard or soft according to the proportions taken, and find extensive application. It is soluble in oil of turpentine, volatile distillates from coal, lignite, or petroleum, in the warm, and forms salve-like masses, the consistency of which depends on the amount of solvent used. If treated in a suitable way, it can also be emulsified in alcohol. Concentrated or dilute acids have no action on ceresine in the cold, nor is it saponified by caustic alkalis or their carbonates, though it can be emulsified by special means.

The specific gravity of ceresine at various temperatures is given in the following table:—

0·790	at 73° C.
0·772	,, 88° ,,
0·769	,, 106° ,,
0·763	,, 112° ,,
0·750	,, 120° ,,
0·750	,, 130° ,,
0·735	,, 170° ,,

ADULTERATION OF CERESINE; TESTING.

As in the case of many products of the chemical industry, ceresine is often found to be mixed with extraneous substances, which, however, are not always added with fraudulent intent, but to satisfy the demand of consumers for cheaper grades, because pure ceresine at present is about double the price it was a few years ago. To comply with this demand, paraffin is mixed with the ceresine in varying quantities up to as much as 90 per cent., so that in judging the quality of ceresine samples with regard to the percentage of paraffin, the price of the article must always be taken into consideration.

Since, owing to the identical chemical composition of ceresine and paraffin, there is no reagent capable of distinguishing between them, it is a very difficult matter to determine the percentage composition of mixtures, and the only criteria that can be adduced in proof are the melting-point, specific gravity, and the behaviour when rubbed between the fingers. Of these, however, the specific gravity gives no reliable information to go upon, because the mixture may contain ceresine of low melting-point and hard-scale paraffin, so that only large additions of paraffin can be revealed by the specific gravity, the result being inappreciably affected by even 25 per cent. of that substance.

The mechanical test by manipulating the material with the fingers necessitates skill—the product being generally more slippery and less fragile than pure ceresine, also more translucent and lustrous.

According to Dr. Berlinerblau, mixtures of ceresine and paraffin have no well-defined melting-point, and, by applying heat carefully, a large number of melt fractions can be obtained, each of which may be examined separately in respect of its physical properties (specific gravity, melting-point, tensile strength, crystalline habit, etc.).

TABLE OF MELTING AND SETTING POINTS AND SPECIFIC GRAVITY OF MIXTURES OF CERESINE AND PARAFFIN (BERLINERBLAU).

Proportion of Ceresine in Mixture.	Proportion of Paraffin in Mixture.	Melting-Point.	Setting-Point.	Specific Gravity at 15° C.	Difference.	Specific Gravity at 83–85° C.	Specific Gravity at 95° C.
		° C.	° C.				
10	0	70–73	69·5	0·921		0·7835	0·774
9.5	0·5	69–73	68·5	0·919	0·002	...	...
9	1	68–72	66·5	0·9175	0·0015	0·7800	...
8	2	66–71·5	65	0·914	0·0035	0·7775	...
7	3	64·5–70	63	0·910	0·004	0·7750	...
6	4	62–69	62	0·907	0·003	...	...
5	5	58·5–67	60	0·904	0·004	...	...
4	6	56·5–65	59	0·900	0·003	...	...
3	7	54·5–62	57	0·897	0·003	...	...
2	8	52·5–58·5	54	0·894	0·003	...	...
1	9	49·5–54·5	49	0·892	0·002	...	...
0	10	47–52	47	0·889	0·003	0·7655	0·756

Far more important, but more readily detected, are the adulterations of ceresine with other substances, especially with heavy mineral oils, crude oil, colophony, saponifiable fats (tallow, stearine, palmitine, Japan wax), and with additions of carnauba wax (to increase the hardness) or beeswax (to impart the characteristic smell, but not as an adulteration; for though one may speak of the adulteration of beeswax with ceresine, the converse does not apply), and finally the clumsy adulterations with mineral substances (gypsum, barytes, kaolin), talc, and other minerals.

The adulteration with heavy mineral oils furnishes a product in which the presence of 10 per cent. of the added matter is inappreciable, though 20 per cent. is revealed by the increased softness and greasiness. Moreover, this form of adulteration is restricted to crude ozokerite, and is now rarely practised. Ceresine is frequently adulterated with colophony, which can be detected by the smell, stickiness when rubbed between the fingers, by the melting test, and by chewing. Ceresine being insoluble in alcohol, the colophony can be extracted with this solvent (hot) and precipitated by the addition of water. Colophony is also saponifiable by boiling with caustic potash or soda, and also with carbonate of potash, and can be determined with comparative ease, the quantitative determination of this adulterant being always an advisable test to perform, since large quantities are often present in cheap brands of ceresine. According to Berlinerblau, the chief fatty adulterants are tallow, stearic acid, and palmitic acid. The methods of determination are based on the acid, ether, and saponification values, the ratio of acid to ether value furnishing information on the composition of the added fat. The same methods are also used for determining additions of carnauba wax, beeswax, Japan wax, and allied substances.

Inorganic admixtures are preferably determined by melting 100 grms. of the material under examination, and leaving it to stand awhile in the liquefied state, the liquid being decanted from the dense sediment, which is then treated repeatedly with benzine, dried, and tested by the usual methods.

For the detection of small quantities of ceresine in paraffin, Dr. E. Graefe recommends the following method: 1 grm. of the substance is dissolved in 10 c.c. of carbon disulphide at 20° C. If the mixture contain more than 10 per cent. of ceresine, the solution is turbid at the above temperature and exhibits a silky sheen when shaken up. 1 c.c. of this solution is treated, also at 20° C., with a mixture of 5 c.c. of ether and 5 c.c. of 96 per cent. alcohol in a test-glass. No precipitation occurs in the case of pure paraffin melting at about 54° C., though a deposit is produced if ceresine be present, the precipitate being flocculent (like alumina thrown down from dilute solution by ammonia), and varying according to the amount of ceresine. By this means as little as 1 per cent. of ceresine can be detected in the mixture.

Should any doubt exist in the case of such, or smaller, additions (which, however, will only happen when the operator is applying the test for the first time), it may easily be resolved by repeating the test with pure paraffin of the same melting-point as the mixture under examination, and comparing the two results side by side. Harder paraffin scale than that of melting-point 54° C. gives a deposit on the addition of the ether-alcohol mixture, because of its low solubility therein, though the precipitate is quite different in appearance from that given by ceresine. In such event the test-glass is slightly warmed in the hand until all has dissolved, whereupon it is laid aside to cool. With pure paraffin the subsequent deposit is merely crystalline, whereas the appearance of flocculent matter—chiefly collecting on the surface—indicates the presence of ceresine.

Mixtures of paraffin and mineral wax, which also do not form clear solutions in carbon disulphide, differ in a marked manner from those of paraffin and ceresine: (1) by their milky appearance; (2) by the cloudy solution clearing, instead of giving a deposit, on treatment with the ether-alcohol mixture; and (3) by the acid value, 1 grm. of mineral wax requiring 17 c.c. of decinormal alcoholic caustic soda for titration, whilst mixtures of paraffin and ceresine are free from acid. The above-mentioned melting-points of paraffin were determined in the Shukoff apparatus.

Tests performed by Dr. F. Sommer gave results that differed considerably from those of Graefe. The former worker found that only American and German paraffin remained in solution when treated with ether-alcohol, whereas Scotch and Galician paraffin, though melting below 54° C., and the more refractory Java paraffin, gave considerable precipitates—often resembling those from ceresine—which disappeared only at higher temperatures. Hence the test should be applied at a somewhat higher temperature, *e.g.* 25° C., or preferably by adding the ether-alcohol mixture at 20° C., and then shaking and warming the whole a little. If ceresine be present, there will then remain a proportionate flocculent precipitate, of the kind described by Graefe, whilst pure paraffin will usually pass completely into solution, or leave merely fine drops undissolved. Nevertheless, according to Sommer, the accuracy of the method is considerably impaired.

DETECTION OF CERESINE AND PARAFFIN IN BEESWAX.

The method pursued by A. v. d. Haar is as follows: 14 grms. of the wax are saponified with an excess of alcoholic caustic potash, and when the operation is completed the surplus alcohol is expelled by heating. The crude soap is dissolved in hot water, and the solution cooled, whereupon all the unsaponifiable matters (the myricyl alcohol, the hydrocarbons of the wax, and the ceresine or paraffin) separate out in a solid form, and must be repeatedly boiled in water to recover any occluded soap they may contain. The mass, which is collected again when the water cools, is next boiled with glacial acetic acid for 2½ hours under a reflux condenser, and is afterwards treated with sufficient acetic acid to prevent the reprecipitation of the myricyl acetate formed. When cold, the hydrocarbons will be obtained in a solid form floating on the surface of the acetic acid, from which they are removed and are heated at 110° C. for an hour to completely volatilise the acid. The next stage is to heat the mass on the water bath with 20 c.c. of sulphuric acid for 2½ hours, to destroy (carbonise) any residual myricyl alcohol. After recooling, the paraffin is freed from adherent sulphuric acid by repeated melting in hot water, and is then dried at 110° C., any carbonaceous particles present being finally removed by filtration. By this means about 45 per cent. of ceresine or paraffin was recovered from a sample of wax in which adulteration was suspected on the ground of Dieterich's table of specific gravities (0·934). At the same time v. d. Haar investigated the results of Schwald and Kraft, and found that perfectly pure beeswax contains about 5 per cent. of hydrocarbons, which, however, are carbonised when heated to 100° C. for some time in presence of sulphuric acid, whereas paraffins only commence to partly carbonise at 160° C.

When beeswax is to be tested for adulteration with ceresine or paraffin, the best method is to saponify the substance and throw down the hydrocarbons by adding water, in order to determine their melting-point. In the case of paraffin, only three grades come under consideration—namely, those melting at 40–42° C., 46–48° C., or 50–52° C. respectively. Paraffin melting at 36–38° C. is detected immediately by the smell, and grades melting at 60–62° C. or above are practically excluded on account of their high price. On the other hand, ceresine melting at 62° C., 64–66° C., or 70–72° C., has to be reckoned with. A good preliminary test consists in determining the refractive index of the substance with the Zeiss butyro-refractometer, which must be fitted with a thermometer graduated up to 75 or 80° C. The test should be performed at a minimum temperature of 67° C., since many waxes melt at 65–66° C., or even higher. The method recommended by Marpmann—dissolving the wax in peppermint oil and determining the refractive index at 40° C.—has much in its favour.

Sometimes it is also possible to settle the question of adulteration by melting the isolated hydrocarbons with an equal weight of pure wax and moulding the hot mass into a cake. If reticulated elevations appear on the surface when cold, ceresine is certainly present; but these must not be confounded with the more or less rounded depressions exhibited by nearly all waxes when moulded in an overheated condition. Wax adulterated with paraffin generally exhibits a shiny surface when cut, and smells of petroleum on being heated; whereas, if adulterated with ceresine, the cut surface is rough and the wax is white and crumbling when kneaded, instead of being smooth and glossy as is the case with paraffin.

The method of judging the presence of extraneous hydrocarbons in wax from the specific gravity is unreliable, owing to the fluctuating specific gravity of the wax itself and still more on account of the almost entirely incalculable density of ceresine and paraffin, for which reason this method has been abandoned by Berg. The sulphuric acid test also gives doubtful results in most cases. The methods of Büchner and Hager are both reliable when the presence of large quantities of extraneous hydrocarbons is in question, but where these additions are small—*e.g.* in many wax candles improved in appearance by the addition of 3–5 per cent. of paraffin—even these methods leave one in doubt, the small quantities of soft paraffin being soluble in strong alcoholic caustic potash and also in benzol.

THE PREPARATION OF CERESINE FROM OZOKERITE.

The most profitable use of remelted ozokerite is to convert it into ceresine, the excellent properties of which command a much higher price than could be obtained by working the raw material up into paraffin. One of the chief advantages of remelted ozokerite—namely, its great plasticity, which approximates to that of beeswax—is, however, partially destroyed in the bleaching process; and all attempts to restore this plasticity to the ceresine have been unsuccessful. As in the case of all petroleum products, the refining agent used is concentrated sulphuric acid, which destroys all the colouring matters (and also some of the other hydrocarbons) present in ozokerite. The process is carried on at different temperatures, and must generally be supplemented by treatment with bleaching agents, the colour of the refined ceresine being dark instead of yellow or white. This colour is removed by a bleaching and filtering process, a yellow product being obtained the first time and a more or less pure white ceresine on repeating the operation.

There are two methods of refining ozokerite with sulphuric acid—namely, the asphalting method and the boiling method. In the former the temperature is not allowed to exceed 120° C., whilst in the other it is maintained at 160° C., whereby the tar deposited at 120° C. is decomposed.

Since, owing to the method of melting pursued at the mines, all ozokerite contains a certain amount of water, it must be freed from this latter by heat in order to prevent the undue weakening of the acid by dilution. The water content should not exceed 3 per cent., and this amount is got rid of by maintaining the ozokerite at a temperature of about 120° C. until it ceases to throw up bubbles and froth, and is therefore free from water. This operation may be performed with or without the condensation of the steam, which always contains oily particles from the ozokerite.

The two methods of refining are thus described by Berlinerblau:—

In the asphalting method the dried wax is mixed with sulphuric acid (30 per cent. at most, there being no advantage in using more) at 120° C., whereupon a viscous black tar is formed, which sinks to the bottom, owing to the presence of combined and free sulphuric acid. This completes the operation for obtaining ceresine, though when a paler or white product is required, the mass must be treated repeatedly with fresh sulphuric acid. The resulting asphaltic mass is treated with milk of lime, to neutralise the acid, and is finally boiled with water for the recovery of any contained ceresine.

The boiling process is the same in the early stages, but instead of stopping the operation to allow the tar to subside, the whole is heated to 160° C., which produces decomposition manifested by the liberation of a large quantity of sulphur dioxide and water vapour. This stage is a very important one, and must be carefully followed, the temperature being raised (but not above 200° C.), when the decomposition is approaching completion. The powerful oxidising action of the sulphuric acid is not thoroughly exerted below 160° C., at which point the sulpho-compounds are decomposed and the hydrocarbons regenerated, whereas at 120° C. a large proportion of the added acid enters into combination (sulpho-acids?) and the oxidising effect is slight. At the higher temperature the deposition of tar is superseded by the separation of small granules of carbon and asphaltum.

According to Lach, the amount of sulphuric acid originally used in the asphalting treatment was only 2–5 per cent. The asphaltum was distilled, and gave the following products, according to the character of the ozokerite :—

Petroleum	5–10	per cent.
Paraffin oils	10–20	,,
Paraffin scale	60–90	,,
Coke	20–25	,,
Loss	5	,,

Calculated to ozokerite, the yield of asphaltum varies from 5 to 10 per cent. Of late, the asphalting treatment has been restored to favour, because the residue, when melted with colophony and other residual substances, forms an excellent insulating material for electric cables. Lach states that the amount of acid used in the boiling process ranges as follows, according to the extent of decoloration in view:—

	Per cent.	
Natural or yellow . . .	15–20	English commercial sulphuric acid (66° B.).
Thirds or semi-white . .	20–25	
Seconds or white . . .	25–30	
Prime or FF . . .	30–35	

the acid being added all at once or (preferably) by degrees, with stirring, at a working temperature of 100–130° C.

When all the acid has been run in, the temperature is raised by degrees to 170–210° C., and is maintained at that level until all the free acid has been expelled as sulphur dioxide. When this stage has been reached, and the boiling mass gives off a sweetish smell, the contents of the vessel are left to cool, and the bleaching agent is stirred in. The practice of leaving a portion of the free acid in the mass, and neutralising it with the bleaching powder, has been found undesirable. It is advisable to make an intermediate product by using only 5–10 per cent. of acid at first, and to treat this with a further quantity of acid after it has been pressed. Neutralising any residual acid with alkalis has also proved unsatisfactory.

Von Boyen ascribes considerable importance to the composition of the sulphuric acid. The larger the proportion of anhydride present, the greater the decolorising power, but, on the other hand, the greater the loss of ceresine. Of course this may be neglected when it is desired to obtain a ceresine capable of resisting the action of fuming sulphuric acid (ordinary ceresine prepared with bleaching agents alone being more plastic and being partially carbonised by this form of acid). This product, however, is seldom required. The use of acid containing too much anhydride is, however, undesirable on other grounds as well, for, apart from its high price rendering it unprofitable, it retards the deposition of the asphaltum in the same way as an over-rapid rise of temperature does. Acid of density 83° B. and over causes the mass to thicken as soon as the critical temperature is reached, separation then usually ensuing when the temperature rises farther, though even less satisfactorily than is the case when heat has been applied too quickly, the mass refusing to clarify even with an excess of clarifying powder, and cannot be pressed or filtered.

When the final traces of acid are not expelled by evaporation, they are removed by means of a suitable quantity of grey neutralising powder, added gradually after the contents of the vessel have been cooled down to about 160° C. To facilitate clarification, the powder should be in a moist condition, and if added all at once, would cause the mass to froth over. In fact, the mass must be carefully washed, even when the powder is added in small quantities at a time. When all the powder is in and the frothing has subsided, the vessel is covered up and left at rest for twenty-four hours, whereupon the pure wax is drawn off for treatment with the bleaching agent. The residue is pressed for the recovery of any ceresine still present, or else is cooled with continued stirring, and afterwards reduced to powder.

The chief bleaching agent still employed is the lixiviated residues from the manufacture of ferrocyanides. Both the grey and black powders are used, their efficacy differing according to the content of carbon. The black powder is in a finely divided state and is a better decolorant than the grey, but is extremely difficult to separate again from the ceresine, even by filtration. The best decolorising powder contains up to 50 per cent. of water (seldom below 20 per cent.), and as this water hinders the process, the powder should first be dried at 110° C., taking care not to exceed that temperature owing to the risk of igniting the powder. To eliminate sand and other impurities, the powder should be levigated with water, left to settle until the greater part has subsided, and the portion in suspension should then be decanted and allowed to settle down, the water being afterwards poured off. This mass, pressed, dried at 110° C., and powdered, forms an excellent decolorising agent. The quantity of powder used varies from 5 to 12 per cent., but its mode of action has not yet been fully explained. Zaloziecki advances three hypotheses for explaining the decolorising action, two of them being on a chemical basis, and the third mechanical.

(1) Oxidation, owing to the power of the decolorising powder for occluding gases. (2) Neutralisation by the action of basic constituents. Carbon possesses in a high degree the property of absorbing oxygen from the air and condensing it into a form capable of powerfully oxidising various colouring matters and converting the same into colourless substances. This action, however, has been criticised by various workers, Scheibler, for instance, having long ago pointed out that the decolorising action of bone black (which is also said to occlude oxygen) is not impaired by heating it for some time in a current of hydrogen. No loss of power occurs when the black is heated in a current of carbon dioxide or freed from air under the air pump. The basic compounds (also the carbonates) in the decolorising powder are able to combine with acid colouring matters, precipitate them from solution, and carry them down to the bottom along with other impurities in suspension. (3) The third or mechanical action in decolorisation is based on the surface attraction exerted by the powder in consequence of its porosity. Possibly all three of these factors operate more or less in producing the result.

Bone-black is also said to be a suitable bleaching agent for ozokerite, whereas the natural silicates, such as aluminium-magnesium hydrosilicate, are not so well adapted, and, according to some reports, are useless for this purpose. Other porous substances, like wood charcoal, coke, pumice, quicklime, calcined magnesia, brick-dust or firebrick dust, and the like, have no decolorising effect at all on ozokerite; and the same applies to the oxides of the alkaline earths and of the iron-aluminium group, though patents have been granted for their use in this respect. Hence the residues of ferrocyanide manufacture still continue the most advantageous decolorising agents used.

The acidified and reneutralised ozokerite is intimately mixed with the bleaching powder, and maintained for some time at the melting temperature of the wax, two or three hours being usually sufficient for the reaction. The further treatment relates to the pressing of the total mass in—

1. Filter presses,
2. Pressure filters,
3. Hydraulic presses.

The filter presses do the work most quickly and reliably, whereas the pressure filters are only suitable for semi-refined grades, owing to the difficulty experienced in getting the acid-refined product to pass through the filters. The oldest and best-known process is that of the hydraulic press, working at pressures of 300 atmospheres and over. The wax is either packed in cloths or moulded into cakes and placed in the press, which is suitably heated, and the ceresine expressed by the influence of heat and pressure. The product is obtained in the form of thin cakes, whilst the press residue is mixed with some absorbent material, such as sawdust, chopped straw, rice husks, or the like, and then extracted in the usual manner with benzine, carbon disulphide, or carbon tetrachloride. The residues left in the filters are also treated in the same way. The pressed or filtered ceresine, though of the desired colour, still contains small particles of residue, and is therefore not perfectly white, but more of a dirty grey. It must therefore be put through an additional treatment, which, according to Lach, may consist of clarification, filtering over filter tables or filtering in a press.

The clarification process has never been much used. About 2 tons of ceresine are placed in a vat of suitable dimensions, and treated with an addition of $\frac{1}{2}$ lb. of stearic acid, a similar amount of colophony being added in the case of yellow ceresine. The mass is boiled up overnight by means of a steam coil and treated with about 2 oz. of caustic soda in the form of a solution of specific gravity 10° B. and boiled for an hour. The resulting stearine or resin soap is in the form of a fine emulsion, which surrounds the mechanical impurities present and gradually carries them down to the bottom, whereupon the clarified ceresine may be carefully drawn off. This method requires a rather long practical experience to ensure success.

The method of filtering over tables is in general use, the filters being box-shaped and divided into two compartments, each of which contains five wedge-shaped sheet zinc moulds, with slits at the bottom to enable the filtered ceresine to drain away into a collector. The moulds are heated by steam coils laid under the bottom, and are lined with filter paper. When the apparatus is made ready and heated, the melted ceresine is poured in from a can with a spout, or introduced through a suitable pipe. The collector discharges into a pipe leading to the cooling vats. The method is slow, entails hand labour, and the consumption of filter paper is large.

The simplest and quickest method is to filter through presses lined with cotton filter cloths, since large quantities of ceresine can be treated and made perfectly white in a few minutes.

The clear filtered ceresine is next stirred, in order to dispel its transparency and make it look whiter, and also, in the case of yellow ceresine, to make the colour paler and impart a wax-like appearance. This operation, though slow and troublesome, is necessary, because the consumers desire the ceresine to resemble beeswax as closely as possible. The stirring may be performed by hand or with mechanical stirrers. In the former case, hardwood vats holding about 4 cwt. are used, and the melted ceresine is ladled backwards and forwards from one vat into another, until it cools and begins to solidify, whereupon it is worked about with wooden paddles until the desired character is obtained, this being judged by the temperature, though chiefly by the eye, since different qualities of ceresine differ in their melting or setting point. The pappy ceresine is then ladled with a tin-plate scoop into buckets and poured thence into moulds. A useful form of mechanical stirrer consists of a tee-shaped wrought or cast iron standard, the base of which is let into the ground, the ends of the cross piece being provided with a couple of superimposed bearings for the support of vertical sleeves, each of which is provided with a bevel pinion and a thickened upper rim cut in the form of teeth. Through these sleeves are passed shafts, which can be raised or lowered by means of swivel hooks and counter-weights suspended on rollers attached to the ceiling of the workroom. When lowered to a certain distance, a toothed ring on the shaft engages with the toothed rim of the sleeve and thus couples up the shaft, so that when motion is transmitted to the cross piece from a parallel shafting fitted with bevel pinions, the vertical shafts are caused to rotate. The bottom end of each shaft is provided with slanting cross vanes. When the stirring apparatus has been hauled up and thrown out of gear, a vat filled with ceresine is placed underneath, and the stirrers being then lowered and thrown into gear again, the vanes set the ceresine in motion at a rate corresponding to their own velocity. Over the top of each vat is laid a strong wooden frame, carrying on either side a pair of vertically adjustable arms supporting a paddle-wheel like a toy waterwheel. These wheels dip half-way into the ceresine, and are caused to rotate by the whirling motion of the latter, which they cool by contact with the air. At first these paddle-wheels run at a high speed, but slow down in proportion as the ceresine solidifies and its resistance increases. Finally, when the mass has become so thick that the paddles will no longer turn, the stirrers are removed and the operation is completed by hand. This final hand-stirring is unavoidable and cannot be replaced by any mechanical device, because the ceresine must be kept stirred until the last drop has been transferred to the buckets, after the vat has been taken away from the stirrers and tilted for pouring.

The operation of pouring the moulds, for making the blocks and cakes of ceresine for sale, must be performed as quickly as possible, in order that the mass may be uniform in colour and appearance. The moulds are of different kinds, mostly of tinned iron, round or rectangular in shape, and holding $\frac{1}{2}$, 1, 2, 5, or 10–11 lb.; and 2 oz. cakes are also made. The moulds are laid on the floor in rows close together and are kept in motion during the pouring, so as to ensure uniform filling with the thick, pappy ceresine. At the end of 2–3 hours the moulds will be cool enough to be safely laid in cold water, or out of doors in the winter. The water troughs, at least two in number, and varying in size according to the daily output of the factory, are made of cement, 20 inches in depth, and are fitted with a controllable water-supply and one or more overflows. The bottom has a 2-inch slope towards the one end, where a closed draw-off is provided, through which the water can be run off for cleaning the troughs. The overflow water passes through a small separator to catch any small particles of ceresine, the lukewarm water being utilised for feeding the boiler or in any other way. To protect the cement bottom from injury by the full moulds, it is covered with a wooden grating held down by lateral wedges. The sides of the troughs are topped with strong wooden kerbs, faced at the top with short iron plates against which the ceresine can be knocked out of the moulds. The outdoor cooling places are made in the form of compartments built of strong wooden spars, between which the loaded moulds can be piled up in rows. They must be carefully levelled with a spirit level, to prevent any warping of the warm blocks, and screens of strong canvas must be erected on the windward side to exclude dust. Before removal from the moulds the blocks must have cooled sufficiently to drop out under the slight shock produced by knocking the moulds against the iron plates or wooden blocks provided for that purpose. In winter this task is easy, but more difficult in summer; and it is therefore highly important to have a sufficiency of cooling water and large cooling tanks at disposal in the warm season.

The colouring of ceresine will be dealt with later on, and all that need be mentioned here is that the moulded blocks must be cooled by air alone and without the aid of water. When the blocks are to be stamped with the factory brand or quality marks, suitably engraved or cast zinc plates are inserted in the moulds, or else the design is stamped on tin-plate, the under side of which is provided with a soldered-on piece to keep the whole level in the mould.

The cooled blocks are transferred to a special room, where they are wiped over with rags, trimmed up if necessary, packed in paper, and sent to the storeroom.

UJHELY'S METHOD OF REFINING OZOKERITE.

The ozokerite is brought into solution, by the warm or cold process, either in crude or distilled petroleum, or in coal-tar distillate or other liquid hydrocarbon, a third class of solvent consisting of carbon disulphide. The specific gravity of these solvents is immaterial. The resulting solution is digested with bone-black, the residue from ferrocyanide manufacture, or with a mixture of both, and then filtered. An alternative method consists in passing the solution through a filter charged with any of the above bleaching agents. The filtrate, containing refined and bleached ozokerite, is then freed from the solvent, which is recovered either by evaporation *in vacuo* or by distillation, or preferably by exposing the filtrate to the influence of low temperatures, which cause the bleached ozokerite to solidify or crystallise out of solution.

PREPARATION BY THE AID OF SAPONIFIABLE FATS.

Ozokerite is melted, along with 2 per cent. of cocoanut oil or palm oil, in a steam-jacketed cast iron mixing cylinder fitted with stirrers, and is heated to 80° C. Next, 7–8 per cent. of fuming sulphuric acid is poured in, as a thin stream, through a funnel, with continued stirring, the whole being left for 2–3 hours in the warm, and the liquid then drawn off from the viscous tarry residue into a similar vessel. Here the warm acidified substance is washed with dilute caustic soda, containing a soap solution prepared from 2 per cent. of cocoanut oil and 2 per cent. of caustic soda, and is left to settle for another 2–3 hours. The soap carries down with it the finely divided particles of carbon, and clarifies the mass thoroughly, whereupon the lye is drawn off and the mass washed with water. When the ozokerite is found to be neutral, it is returned to the first vessel, and the entire series of operations is repeated thrice. After each treatment the ozokerite will be found lighter in colour, so that the fourth acid treatment leaves it almost perfectly white. The final purification consists in bleaching it with finely granulated bone-black or the residue from the manufacture of ferrocyanide.

CHEMIN'S PROCESS OF REFINING.

Crude ozokerite is melted in water at 65–75° C. and is then placed in a retort which is heated direct and also by means of a jet of superheated steam. In this vessel the ozokerite is treated with 5–10 per cent. of flowers of sulphur, which substance is almost entirely converted into gaseous products, sulphuretted hydrogen, and organic sulphur compounds, and exerts a bleaching action on the ozokerite, which distils over in a bleached condition. A similar bleaching action is effected by melting the ozokerite and sulphur together without distillation. The more fluid constituents are removed from the distilled ozokerite by warm pressing, or by washing it, in a finely divided state, with amyl alcohol or hot water. The product is melted, mixed with 20 per cent. of amyl alcohol, cold pressed after cooling, and then purified with bone-black.

To facilitate these operations, the ozokerite is treated with 20–40 per cent. of petroleum residue before distillation. The ozokerite purified in this manner may be used to replace white wax, or be mixed with wax, resin, etc., as a substitute for inferior waxes.

PREPARATION OF CERESINE BY DECOLORISING OZOKERITE.

Nearly fifteen years ago, E. von Boyen stated that yellow and white ceresine could be prepared, without the aid of the acid process, by means of decolorising agents, mostly compositions, the chief effect of which is due to chemically precipitated silica or silicates. These substances have little action on melted ozokerite, which they decolorise by attracting colouring matters and particles of carbon, which are thus separated from the pure white solid hydrocarbons. The resulting ceresine is always more plastic than that obtained in the ordinary way, from which it can be distinguished by the fact that it is powerfully acted upon and partly carbonised by sulphuric acid.

Contemporaneously with von Boyen's discovery, Lach reported that ozokerite could be decolorised by repeated treatment with finely powdered bone-black, with hydrated (gelatinous) silica freshly prepared from sodium silicate and hydrochloric acid, and also with magnesium silicate, but that the method was not so successful on the large scale as in the laboratory, and was therefore unsuitable for practical application, apart from the excessive cost of the reagents (bone-black especially) and the extraction process. With gelatinous silica the decolorising can be effected by simple stirring, followed by filtration, provided the treatment be several times repeated. When ozokerite is treated with 10 per cent. of magnesium silicate—prepared by acting on sodium silicate with magnesium chloride, followed by drying and calcination —by stirring in the silicate at 120° C. and filtering, a yellow ceresine is obtained, the residual silicate acquiring a dark colour. On extracting the wax and colouring matter from the silicate by boiling or solvents, and treating the product with fresh magnesium silicate, a dark yellow wax is again obtained, and the wax residue left in the silicate is deep black. A few more repetitions of the process will concentrate the colouring matter to such an extent that it can no longer be bleached by magnesium silicate or even by sulphuric acid, the product forming a deep black greasy mass resembling kindebal, and furnishing distillation products, like the latter, which can only be purified by distillation. According to Lach, this proves that the dark colour of ozokerite is due to the presence of dark soft substances analogous to kindebal. More recently, the attempts to dispense with the acid process have been renewed, by treating mixtures of ozokerite and paraffin scale with magnesium hydrosilicate (Floridin brand) and black decolorising powder, large quantities of these reagents being necessary. The yield of ceresine is increased 15 per cent. by this treatment, but the product is rendered greasy by the presence of the constituents which are destroyed by sulphuric acid. The method is naturally restricted to inferior grades of ceresine containing large proportions of paraffin, but owing to the enormous rise in the price of ceresine, this method has increased in popularity, and rightly so, because of the good results it achieves when properly applied.

MIXING PARAFFIN AND CERESINE.

The cheapening of ceresine by admixtures of paraffin is extensively practised, and the product is placed on the market by ceresine manufacturers as second and third grade ceresine, etc. In these factories the Lach treatment precedes the acid process, or else the paraffin scale employed is bleached separately and added to the coloured ceresine — a more economical way. The amount of added paraffin varies from 10–80 per cent. according to the price of the finished article. If the scale be added to the unbleached ozokerite, a loss of up to 15 per cent. is sustained in the acid treatment, whereas with bleached scale the loss is only 5 per cent. or so, and the consumption of other material is small. The use of white paraffin scale is not advisable, apart from its cost, the melting-point being lower (47° C. as compared with 52° C.) than that of yellow scale, so that only a small quantity can be added to the ceresine. Refined Scotch or Thuringian paraffin, of higher or lower melting-point, though largely used, is less suitable.

Certain brands of ceresine are also mixed with up to 70 per cent. of colophony, especially for the export trade. The colophony is added by melting a weighed quantity of ceresine in a tinned or lined pan, and stirring in the colophony in proportion as it becomes incorporated with the mass, care being taken to prevent the temperature rising above the melting-point, or the product will smell strongly of turpentine. The same smell is noticeable when the colophony is melted beforehand and the ceresine is added to it for melting and mixing.

WAX PITCH, THE DISTILLATION RESIDUE FROM OZOKERITE.

On the comparatively rare occasions when ozokerite is treated for conversion into paraffin, the products include substances fluid at ordinary temperature, semi-solid paraffin butter (first runnings), slightly coloured paraffin as the middle fraction, a mixture of paraffin and empyreumatic resins as final runnings, and finally wax pitch, which is solid at the ordinary temperature. The paraffin butter consists of oils and paraffin, is liquid at 25° C., and is worked up into paraffin scale (crystalline paraffin) or vaseline, the latter, however, of inferior quality to natural vaseline.

The residue in the still is brownish black to black in colour, occasionally with greenish fluorescence, and of such consistency that it can be kneaded without crumbling; the specific gravity is 0·92–0·96; and in good qualities the fracture is smooth and conchoid, the appearance recalling that of crude ozokerite, but darker and harder. According to Berlinerblau, wax pitch is in good request for making shoemakers' wax, and still more so for the production of insulating material for electrical purposes. To make shoemakers' wax, the pitch is melted and treated with about 2 per cent. of concentrated sulphuric acid, being then heated, whereby it darkens in colour and acquires greater hardness. A little carnaüba wax is added to harden the mass, and lampblack to deepen the colour, the whole being well mixed and poured into small round or square metal moulds bearing an impressed mark.

In the electrical industry, wax pitch is chiefly used in making cable insulation, and since Edison gives the resistance of crude ozokerite as 450 million megohms per centimetre, whilst that of paraffin is only 110 million megohms, the insulating capacity of the paraffin-free residue must be far higher, and in fact it has proved to be very satisfactory, whilst the power of resisting heat is also considerable.

THE ARRANGEMENT OF CERESINE WORKS.

Plans of a Ceresine Works are given in Figs. 1 to 3, and in these, I., II. indicate the living rooms, III. the weighing house,

IV. the store, V. the factory, VI. the cooling room or ice cellar, VII. the gas holder, VIII. the bone-black regenerating plant.

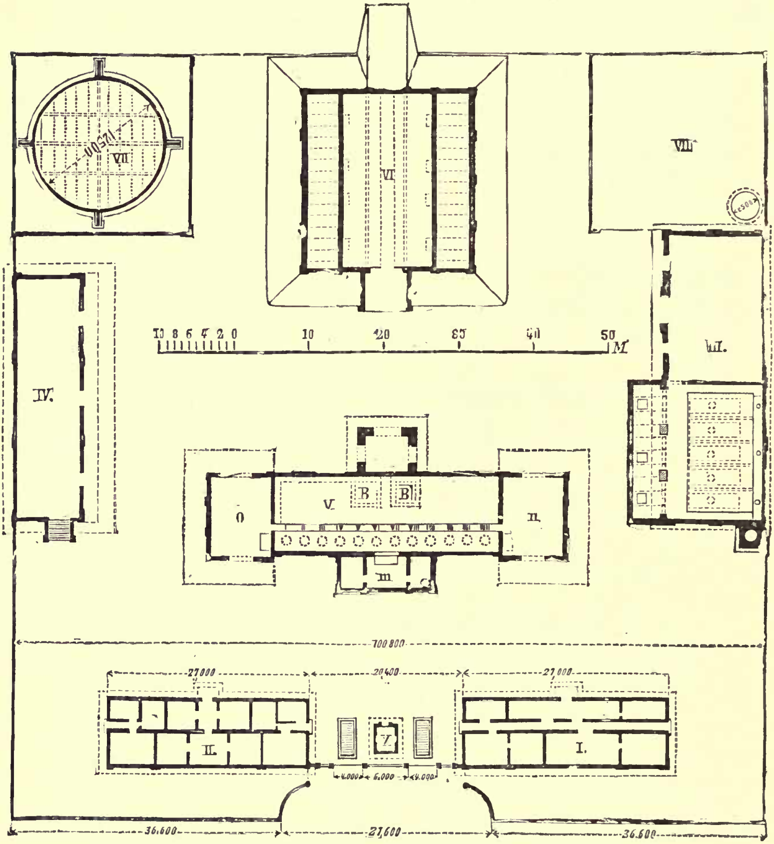


FIG. 1.—Ceresine Works (Ground Plan).

The ground plan of the works shows the position of the filter battery for the dissolved ozokerite and the dissolving pan. Close to the central tower is the still, B, mounted on four iron pillars and communicating with the filter battery by means of the gallery against the wall. Level with the top of the battery is a rail track, so that the battery can be charged from above by means of trucks. The emptying and cleaning out are effected through lateral doors near the bottom. All the valves controlling the apparatus can be operated from a central stand by means of hand wheels.

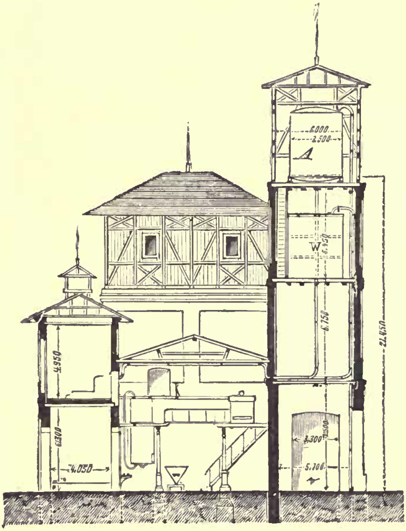


FIG. 2.—Ceresine Works (Cross Section).

The battery is represented by the twelve circles marked on the plan, and the first four units are charged with ozokerite, the next with bone-black. The solvent is run down from the tank A in the tower under a pressure of 2 atmospheres, and passes through the first unit, from below upward, thence through the second and third in the same way, and then into the filter. The current flows thence to the evaporator, the vapours from which are led to the condenser, whilst the filtered residue goes into a collector. When all the soluble matters have been removed from the first dissolver, the first filter being also exhausted, having been doing most of the work, the current is diverted, and petroleum spirit for cleansing the first filter is run into the latter from above and thence into the next dissolver. As soon as the first filter is freed from fat, the first dissolver is recharged with ozokerite, and the emptying of the filter is begun, compressed air being admitted for this purpose. The first filter is now entirely disconnected, and the second one is treated in the same manner to empty it in accordance with the direction of the current, whereupon steam at a pressure of 7 atmospheres is admitted from below. In order to recover all the dissolved matter, a separate condenser is set up behind the battery, the vapours being condensed therein and the solids collected. This pro-

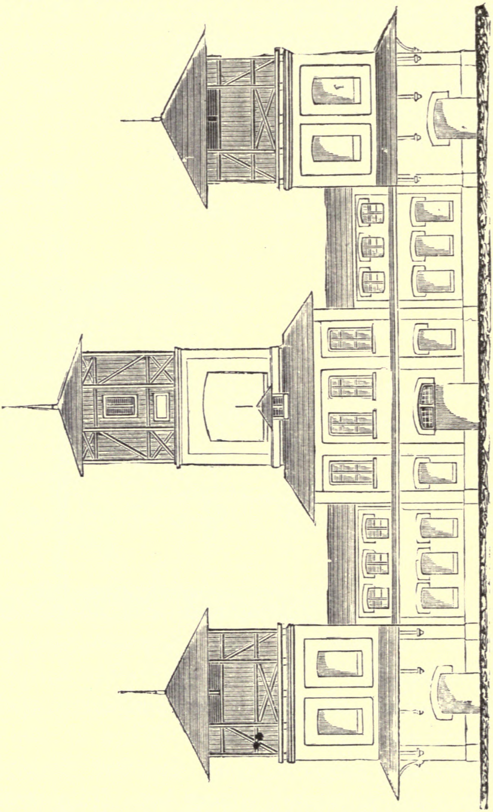


FIG. 3.—Ceresine Works (Front Elevation).

cedure is repeated right through, each unit being treated twice, so that an uninterrupted cycle is maintained. A separate gas holder is provided for collecting the vaporised benzine. To ensure saturation of the solution and increase the efficiency of the filter, the whole plant is kept under a pressure of 2 atmospheres and is connected up with the heating plant. A detachable dissolver is charged with water from below, in order to remove the solvent in the direction taken by the circulation. The rectangular evaporators are fitted with heating appliances, and are connected together by pipes. The condensers for the solvent are cooled by means of ice water. The receivers are under a pressure of 3 atmospheres and are of cylindrical form, the joints at the covers being made tight by shellac and glycerine. The ceresine is packed in tins and carried on trucks to the cooling room. A bridge for conveying the ice is arranged on a level with the top of the ice chamber. The solidified ceresine is returned to the factory, where it is centrifugalised, filtered through filter paper, and washed with a spray of petroleum spirit. The high velocity causes an extraction of heat, accompanied by evaporation, so that the washed ceresine is hardened.

CHAPTER II.

PARAFFIN.

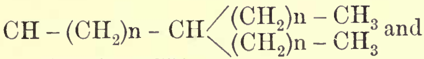
DERIVATION, COMPOSITION, PROPERTIES, AND MANUFACTURE.

THE substance known as paraffin, and more closely resembling stearine than wax in outward appearance, was first discovered by Reichenbach in 1835 in beechwood tar, but at the time attracted no particular attention, the raw material furnishing only small quantities of the product. It was not until about fifteen years later, when the technical utilisation of bituminous lignites and shales was commenced, by treating them for the recovery of illuminating oils, etc., that paraffin began to be made on a large scale, first in Scotland and afterwards in Germany, and an extensive industry was founded, the further development of which was connected with that of petroleum.

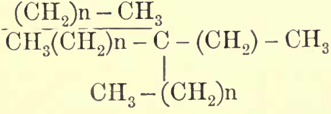
Paraffin is a hydrocarbon of the so-called paraffin series, the general formula of which is C_nH_{2n+2}, and whose members occur in nature as gaseous, liquid, and solid bodies, and can also be produced by the dry distillation of resins, bitumen, etc. The hydrocarbons of the paraffin series are as shown in the table on opposite page.

The first four members of the series are gaseous, the next eleven liquid, and the still higher ones solid, the melting and boiling points increasing with the carbon content. With regard to the classification of the paraffins, Dr. Berlinerblau says that they are divided into normal paraffins and isoparaffins. The structural formula of the former represents a straight chain with simple primary bonds, in which, therefore, one carbon atom is combined with one carbon group, or at most two, *e.g.* $CH_3 - (CH)n - CH_3$. In the isoparaffins, on the other hand, one carbon atom is combined with three or four carbon groups (ternary or quaternary combination): *e.g.*

Name.	Formula.	Carbon.	Hydrogen.	Boiling-Point. ° C.	Specific Gravity.	Density ° B.
Methane (marsh gas)	CH_4	75·00	25·00	Gas	0·559	...
Ethane . . .	C_2H_4	80·00	20·00	,,	1·036	...
Propane . . .	C_3H_8	81·81	18·19	,,	...	...
Butane . . .	C_4H_{10}	82·80	17·20	1	0·600	106
Pentane . . .	C_5H_{12}	83·33	16·67	30	0·629	98
Hexane . . .	C_6H_{14}	83·72	16·28	69	0·668	84
Heptane . . .	C_7H_{16}	84·00	16·00	75	0·699	72
Octane . . .	C_8H_{18}	84·21	15·69	125	0·703	71
Nonane . . .	C_9H_{20}	84·38	15·62	136	0·741	60·5
Decane . . .	$C_{10}H_{22}$	84·51	15·49	158	0·760	53
Undecane . .	$C_{11}H_{24}$	84·61	15·39	182	0·765	54·5
Dodecane . .	$C_{12}H_{26}$	84·70	15·30	198	0·776	52
Tridecane . .	$C_{13}H_{28}$	84·78	15·22	206	0·792	48
Tetradecane . .	$C_{14}H_{30}$	85·55	15·15	238	...	...
Pentadecane . .	$C_{15}H_{32}$	84·90	15·10	238	...	...
...	$C_{18}H_{38}$	85·04	14·96	...	...	...
...	$C_{20}H_{42}$	85·11	14·89	...	...	...
...	$C_{23}H_{48}$	85·18	14·82	...	...	...
...	$C_{25}H_{52}$	85·23	14·77	...	...	...
Paraffin . . .	$C_{27}H_{56}$	85·26	14·74	...	...	...
Paraffin . . .	$C_{30}H_{62}$	85·31	14·69	370	...	...



$$CH - (CH_2)n - CH\left\langle\begin{array}{l}(CH_2)n - CH_3\\(CH_2)n - CH_3\end{array}\right.\text{and}$$



$$\begin{array}{c}(CH_2)n - CH_3\\ CH_3(CH_2)n - C - (CH_2) - CH_3\\ |\\ CH_3 - (CH_2)n\end{array}$$

Whilst the normal paraffins cannot form any isomers, the isoparaffins do so to an extent corresponding with the increase in the carbon content. Cayly has calculated the number of possible isomers as 799, a figure corrected to 802 by Herrmann. To facilitate comprehension of what will follow, it is necessary here to mention that paraffins can be obtained, not only from beechwood and bituminous lignites, but also from petroleums, ozokerite, peat, shale, and other bituminous materials. According to Krafft, the paraffins from lignite tar are normal paraffins, which are distinguishable from the isomers in formative tendency and stability; and as a rule the paraffins obtained by distillation assume the more stable normal form. The case may, however, be different with the paraffins occurring native, as in petroleum or ozokerite, or obtained in other ways than by distillation. Many points tend to show that the native paraffins are not completely identical with the distilled paraffins, and the only question is whether ozokerite and petroleum represent mixtures of normal paraffins and their isomers, or whether the normal paraffins are only formed from the latter in the process of distillation. Engler in particular has shown that the colloidal, vaseline-like mixture of hydrocarbons, left behind as residuum on the distillation of petroleum, becomes crystalline by distillation, from which observation one is inclined to assume that isoparaffins were originally present and have been converted into the normal form by the action of heat.

This hypothesis has also been made in connection with ozokerite. Nevertheless, both here and in the case of petroleum residuum, there is also a possibility that the capacity of the paraffins for crystallisation has been hindered by the presence of associated substances. The solution of this problem has also attracted the attention of Zaloziecki. Since the natural petroleum residues chiefly consist of two heterogeneous constituents —a solid and an oil—Zaloziecki sought to credit the oil with the power of restricting the crystalline faculty, but was obliged to admit his mistake. On separating the oil from the solid ingredients, and mixing it with crystalline paraffin in different proportions by the aid of heat, the resulting vaseline was found to differ considerably from natural vaseline, since it lacked the uniform ductility and also contained crystals, so that no tendency to hinder crystallisation can be attributed to the oil. If ozokerite be treated with amyl alcohol in the warm, it is found that one portion of the mass is more soluble than the rest, and that a crystalline component can be separated from the solution and the colloidal substance can be recovered subsequently from the mother liquor. The same behaviour can be observed with bleached ozokerite (ceresine) and petroleum residue (vaseline). Hence it is probable that colloidal paraffins are present in these products (as well as those of a crystalline nature) and are transformed into the crystalline variety by distillation. Zaloziecki now assumes that the property of preventing crystallisation belongs to these colloidal substances, and terms the natural paraffins (*e.g.* those present in ozokerite) protoparaffins, whilst he calls those obtained by distillation pyroparaffins. In his opinion, however, the crystalline constituents must be regarded as isoparaffins. Engler and Boehm, who studied the question in connection with the investigation of vaseline, were convinced that the transformation of the amorphous constituents into the crystalline ought to be considered as a change in the state of aggregation of one and the same substance. The distillation, *in vacuo*, of the solids of natural vaseline furnished them with an exclusively crystalline product, the closer examination of which showed it to be almost completely identical in composition with the original substance, although the slight diminution in the hydrocarbon content (0·2–0·27 per cent.) and increase (0·4–0·47 per cent.) in the hydrogen content indicated the probability of a small quantity of substance poor in hydrogen being left behind in the still. The melting-point of the original substance was 40° C., and that of the distillate 37° C., which slight decline justified the opinion that isomeric hydrocarbons had not been transformed into those of normal type, since the two kinds differ in the possession of a higher boiling-point and melting-point by the last named.

PROPERTIES.

In the refined state, paraffin is a colourless, more or less pure white, waxy or laminated crystalline mass, semi-transparent and somewhat resembling alabaster, though less opaque than the latter. It also resembles spermaceti, without its fibrous structure. In the cold it is odourless and tasteless. It is slippery to the touch, but not greasy, is softened by the warmth of the hand, and can be kneaded like wax. Paraffins with high melting-point are less transparent; and lignite paraffins give out a resonant note when the cakes are struck by the finger.

All grades of paraffin are insoluble in water, and as they do not impart either taste or smell to that liquid, they are used for impregnating wooden vessels for the storage of food stuffs and delicacies, as also in the preparation of air-tight packing materials, etc. When melted they form water-white liquids, which emit a faint odour. At ordinary temperature the paraffins show a high power of resistance to acids, both concentrated and dilute, and they are not saponifiable by caustic alkalis or alkali carbonates. Only at high temperatures is any action noticeable—for example, on the part of nitric acid and other oxidising agents, this action being manifested in the form of gradual saponification, resulting in the formation of carbon dioxide and water, with by-products in the form of fatty acids, oxalic acid, and succinic acid in small quantities. On prolonged exposure to the air, molten paraffin slowly absorbs oxygen, and to an increased extent when heated to high temperatures. According to Lippmann and Hawliczek, commercial paraffin always contains oxygen, which can be extracted by heating the substance in presence of metallic sodium. Paraffin cannot withstand the action of chlorine, and forms with this element chlorinated paraffins, the most highly saturated member of which has been named chloraffin (Balley). Paraffin is a non-conductor of electricity, its insulating resistance being given by Edison as 110 million megohms per centimetre. The refractive index was determined by Maassmann as 1·480, but no attempt seems to have been made to ascertain the refractive index of paraffins of different origin and melting-point. Reichenbach found, by microscopic examination, that paraffin crystallises in several forms: (*a*) In extremely delicate rounded laminæ; (*b*) in small infundibular aggregations, superficially resembling needles, formed by the coalescence of the laminæ; (*c*) in a granular condition. This observation was confirmed by Hofstädter, with the addition that the acicular form separates first from solutions, and is followed by the angular and finally by the laminated crystals. Recently Zaloziecki examined the crystalline habit of various paraffins from ceresine, petroleum, ozokerite, and lignite tar, and found that the matrix consists of lustrous, superimposed, stratified laminæ, with uniform dark contours. He failed to obtain the acicular crystals from alcoholic solutions, which, however, furnished rhombic or hexagonal plates or laminæ, more or less well developed according to the purity of the substance, the finest being obtained in paraffins recrystallised from ceresine. Paraffin distilled from ozokerite manifests a decided tendency to crystallise, and the slowly cooled paraffin cakes have a laminar-crystalline fracture. Ceresine prepared from ozokerite by extraction or by treatment with sulphuric acid is non-crystalline, and has a waxy granular fracture. It will, however, furnish crystalline paraffin when treated with suitable solvents. As a rule the paraffins obtained from fossil materials without the aid of high temperatures exhibit a more colloidal character, whereas by distillation they are transformed into readily crystallisable substances. Bearing in mind their crystalline character, Zaloziecki, as already mentioned, applies the name protoparaffin to the native paraffins, and calls those obtained by distillation pyroparaffins.

The solubility of paraffin was determined by Reichenbach as follows:—

100 parts by weight of absolute alcohol dissolve 3·45 parts of paraffin after boiling ¼ hour;

100 parts of 80 per cent. alcohol dissolve only 0·33 part of paraffin at 20° C.;

100 parts of ether dissolve 140 parts of paraffin at 25° C., but the solution sets to a pappy mass if the temperature be only slightly lowered.

Oil of turpentine and petroleum readily dissolve paraffin without the application of heat. Olive oil dissolves but little in the cold, but takes it up readily in the warm. Molten tallow and lard will mix with the solid paraffins, but separation occurs on recooling; and the same applies to camphor as well.

Stearine, spermaceti, beeswax, and resin are miscible with paraffins in all proportions in the warm, and do not separate out on cooling.

According to Pawlewski and Filomonowitz, 1 part of paraffin dissolves in—

7·6	parts of	carbon disulphide,
8·5	,,	ligroin, sp. gr. 0·7223, b. p. 75° C.
50·3	,,	benzol,
50·8	,,	ether,
10·86	,,	glacial acetic acid.

Zaloziecki determined the solubility of paraffin (specific gravity, 0·896 at 20° C., and melting-point, 56° C.) as—

1 part of paraffin is soluble in—

350,000	parts of	ethyl alcohol (75° T.) at 16–18° C.
370	,,	amyl alcohol at 16–18° C.
1,060	,,	amyl alcohol at 2–4° C.
12,000	,,	a mixture of alcohol (75° T.) and amyl alcohol at 16–18° C.
42,500	,,	the same mixture at 2–4° C.

The ready solubility of paraffin in oil of turpentine and the volatile hydrocarbons distilled from coal, lignite, and petroleum, and its miscibility with fats, waxes, and resins, mineral, vegetable, and animal oils, forms the basis of a number of technical applications of that substance. The specific gravity and melting-point of commercial paraffins vary, both according to the origin and method of recovery. The grades melting at 48–56° C. are known as soft paraffins, and those melting at 56–76° C. as hard paraffins. The price of the article increases with the melting-point. The following are the chief commercial varieties:—

Melting-point, 38–40° C.
,, 41–42° ,,
,, 43–45° ,,
,, 46–48° ,,
,, 48–50° ,,
Melting-point, 54–56° C.
,, 56–58° ,,
,, 58–60° ,,
,, 60–62° ,,
,, 74–76° ,,

The specific gravity of solid and molten paraffin was determined by Albrecht, the following values being obtained:—

	Melting-Point. ° C.	Specific Gravity at 17° C.	Specific Gravity at 55° C.	Specific Gravity at 60–65° C.
Solar oil paraffin .	38	0·872	0·779	...
Seconds ,, . .	43	0·883	0·785	...
Press ,, (2) .	43	0·887	0·788	...
Seconds ,, . .	46	0·889	...	0·781
Press ,, (1) .	47	0·900	...	0·775
,, ,, (1) .	51	0·908	...	0·775
Hard ,, . .	56	0·912	...	0·777

According to Sauerlandt, Galician ozokerite paraffins (distilled from ozokerite) have the following characteristics at 20° C.:—

Melting-point, 56° C.	Specific gravity, 0·912
,, 61° ,,	,, 0·922
,, 67° ,,	,, 0·927
,, 72° ,,	,, 0·935
,, 76° ,,	,, 0·939
,, 82° ,,	,, 0·943

Galletly obtained the following values for Scottish paraffins from boghead coal:—

Melting-point, 32° C.	Specific gravity, 0·8236
,, 39° ,,	,, 0·8480
,, 40·5° ,,	,, 0·8520
,, 53·3° ,,	,, 0·9090
,, 55·6° ,,	,, 0·9110
,, 58° ,,	,, 0·9243
,, 59° ,,	,, 0·9248
,, 80° ,,	,, 0·9400

No scientific differentiation exists as yet between paraffin and ozokerite; and Ragosine claims that even their specific gravity has not yet been accurately determined, because there is no doubt that commercial ozokerite and paraffins are not homogeneous substances with definite properties, but represent petroleum distillates, a series of hydrocarbons of different composition.

The only way of distinguishing between paraffin and ozokerite in practice is that the former gives long, spindle-shaped crystals, whereas ozokerite is not crystalline at all. When ozokerite and paraffin are melted together, the latter loses its crystalline faculty, wholly or in part, and it is only by accident that a spindle-shaped crystal can be discovered, here and there, under the microscope, and even these are always short, looking as if broken. Even with a sample of ozokerite in its natural form, one cannot be certain whether the same is perfectly free from paraffin; and refined commercial ozokerite or ceresine is always a mixture, since paraffin is cheaper and the purification of ceresine without an addition of paraffin is attended with considerable loss.

A partial distinction can be drawn from the melting-point, the latter being higher in proportion as the amount of paraffin present is smaller; but, owing to the difficulty of ascertaining whether a sample of natural ozokerite is really pure, the true melting-point cannot be determined. The only source of ozokerite thoroughly free from paraffin is Baku petroleum.

According to Ragosine, if paraffin be distilled at high temperatures, it is transformed into light distillates: solar oils, petroleum, and benzine, according to the temperature employed.

In the opinion of Dr. Neustadtl, the transparency of paraffin depends on the melting-points of the various sorts present in the commercial article. If the range of melting-points be low, the product is transparent; but if they vary considerably, the paraffin will be more or less milky.

With regard to transparent and milky paraffins, Mittler and Lichtenstern state that it does not seem to be generally known how milky paraffins are prepared, and on what their higher evaluation is really based. These workers applied Holde's test to perfectly milky paraffin, and found, by working at − 15 to − 18° C., that it contained only 94–96 per cent. of paraffin, the solution in ether-alcohol leaving behind, on distillation, a liquid oil corresponding approximately with the difference, after making allowance for the errors of experiment (0·2–0·3 per cent.). To check these results, a number of paraffins of different melting-points were treated with various liquid additions, the following results being obtained :—

Paraffin used.	Addition.		Appearance after Addition.
Transparent paraffin melting at 5·7° C.	Refined petroleum	1 per cent.	Speckled
	Compressor oil	0·5 ,,	,,
	Refined vaseline oil	3 ,,	Milky, with transparent edges
	,, ,,	1 ,,	Speckled
	,, ,,	0·5 ,,	Slightly speckled
	,, ,,	0·25 ,,	Transparent

Similar results were obtained with paraffins of lower melting-point, it being noticeable in all cases that an addition of 0·30 to 0·3 per cent. of special vaseline oil produced very little change, if any, in the appearance of the paraffin, more particularly as the melting-point of the paraffin receded. To ascertain the correctness of the recently expressed opinion that transparency depends on the range of melting-points of the constituent hydrocarbons, a number of paraffins of different melting-points were mixed together in equal quantities—for example, one melting at 42° C. with another melting at 57° C.; but no great alteration of the appearance, or even milkiness, was found to ensue. In some instances a strong bluish tinge was observed, but the cause could not be elucidated. The melting-point determinations were made throughout in the Shukoff apparatus, and the mixtures always furnished the calculated melting-point. On the other hand, the cooling curves plotted failed to reveal the steep curve attributed to the transparent paraffins in the treatise cited by the authors, this being manifested by those of high melting-point only. They consequently ascribe the varying transparency or milkiness of paraffins primarily to the oil content, though a limit exists below which the presence of oil has little or no effect. Purchasers should therefore only pay low prices for milky paraffin, in order to avoid buying cheap vaseline at paraffin rates, and for this purpose should have the samples analysed, or, if sufficiently experienced, judge the degree of transparency for themselves. The result of these researches is that transparent paraffin can be obtained at will, and at the same time the hardness and melting-point of the product can be controlled by a suitable selection of the paraffin scale for the melting or pressing process, without having to depend on chance for the result.

RAW MATERIALS FOR PARAFFIN.

Beechwood, the material from which paraffin was originally recovered by distillation of the tar, is too expensive, on account of its technical uses and fuel value, to serve as the material for a large paraffin industry, and also because the quantity of paraffin contained in the tar is too small to be obtained cheaply enough to permit of its extensive application. As far back as 1809, Fuchs is said to have observed paraffin in petroleum from the Tegernsee. This product was recovered from the oil by Büchner in 1819 under the name of "mineral fat," and its identicity with Reichenbach's beech-tar paraffin was established by Kobell in 1835. Almost contemporaneously with Reichenbach's discovery (1835), Christisen found "petrolin" (paraffin) in Rangoon tar, and the same product had already been observed in petroleum. Mineral wax (paraffin) was obtained in 1839, along with illuminating oil and gas oil, from bituminous shales; and in 1847 illuminating oil was made in Germany from Scottish coal, and later (1849) from slaty coal as well, by dry distillation, this oil being apparently the sole product considered of value. At an earlier date Professor Runge had made paraffin candles from peat-tar, but the method employed was never demonstrated as suitable for working on a manufacturing scale, and the Irish paraffin industry, started in 1850 for the utilisation of peat-tar, made no headway.

Although a commencement was made in 1846 with the utilisation of bituminous lignites for this purpose, it was not until 1855 that paraffin and candles were prepared from that material at Aschersleben; and the first real development of the lignite-tar industry occurred early in the sixties, when K. A. Riebeck opened a small works at Halle, which proved successful and was soon followed by others.

In England, as far back as 1850, James Young recovered large quantities of paraffin from cannel, boghead, and parrot coal, and laid the foundation of the existing manufacture of illuminants from bituminous materials. Young's first paraffin works were followed in the same year by the establishment of a factory at Bruel, on the Rhine, and this in turn by others.

Within a comparatively short time the lignite-tar industry in Saxon Thuringia attained considerable dimensions. In 1861 about 750 tons of paraffin and 3200 tons of solar oil were produced in the province of Saxony, and the output for 1871 was estimated as 15,000 tons of paraffin and 10,000 tons of solar oil, quantities which were then considered very large. Scheithauer gives the following figures as the output of ten works in Saxon Thuringia:—

1884 . .	7787 tons	1889 . .	7812 tons
1885 . .	7428 ,,	1890 . .	7401 ,,
1886 . .	8089 ,,	1891 . .	8025 ,,
1887 . .	7527 ,,	1892 . .	7963 ,,
1888 . .	7982 ,,	1893 . .	8126 ,,

The Scottish works turn out 20,000 to 30,000 tons per annum.

Later on, a very important factor in connection with paraffin arose in the form of the petroleum paraffin, recovered in large quantities from the residues from the distilled illuminating oils; and this competitor appeared likely to threaten the lignite-tar industry, just getting into a flourishing condition. These fears, however, proved groundless, because, on the one hand, boghead coal yielded 50 per cent. of tar very rich in oil, and on the other, the raw material used in the Thuringian industry was very rich in paraffin; so that only works dealing with certain low-grade shales and peat were obliged to succumb under the competition. The works originally manufacturing paraffin by the distillation of ozokerite were also nearly all shut down, because the extraction process gave much superior results.

Crude petroleum furnishes varying quantities of illuminating oil on distillation, according to its origin, American crude yielding 90 per cent., Galician over 80 per cent., Russian 100 per cent., and Sumatra 90 per cent. of saleable products. Russian oil, it is true, only gives 32 per cent. of marketable products, and 68 per cent. of residuum, because the "cracking process," by means of which light fractions are produced by the decomposition of portions of the crude oil at a high distillation temperature, has not been employed in that country until quite recently. The lubricating oils obtained by simple distillation, as well as those furnished by the cracking process, contain fairly large proportions of paraffin—according to the paraffin content of the crude product; and though it is often stated that Russian oils contain no paraffin at all, or that the residuum cannot be decomposed, Ragosin has shown that, by a suitable process of distillation, which will be referred to later on, a considerable amount of paraffin can be recovered from them.

Since all petroleums either contain ready formed paraffin, or the latter can be recovered from their residues by suitable treatment, these oils constitute a very valuable material for the production of paraffin, and large quantities of that product are actually obtained from them. The first question that arises in connection with the recovery of paraffin from bituminous material, whether coal, shale, petroleum or its residues, ozokerite or the like, peat, etc., is what quantity of paraffin do they contain or can be recovered from them, profitably, by suitable treatment? Thus, it has been pointed out already that paraffin is contained in the tar from beech and other deciduous trees, as well as in the coniferæ, but no one would think of treating wood for the recovery of paraffin. Consequently, it is highly important to examine bituminous materials very carefully for their paraffin content, and then study all the circumstances bearing on the profitable extraction of that product.

The following particulars relate to the results obtained by distilling a number of important raw materials for the production of paraffin.

The yield of tar from different kinds of lignite is—

Oranian	5·37–5·88 per cent.
Nassau	3·18–5·61 ,,
Tscheitsch (Moravia)	4·70 ,,
Eger (Bohemia)	7·50 ,,
Herbitz (Bohemia)	5·70 ,,
Frankenhausen	5·57 ,,
Bensberg (near Cologne)	4·85 ,,
Stockheim (near Düren)	6·24 ,,
Aschersleben	12·00 ,,

that is to say, it differs considerably, so that when the distillation of a new kind is in question, very complete and accurate tests are necessary to see whether its treatment will be profitable. The various products pass over at the following temperatures:—

At 130–170° C., lignite benzine—specific gravity 0·710–0·750;

At 170–220° C., light lignite-tar oils (photogene)—specific gravity 0·750–0·820 ;

At 220–290° C., heavy lignite-tar oils (solar oil)—specific gravity 0·820–0·880 ;

At 290–320° C., lubricating oil—specific gravity 0·880–0·900 ;

At 320° C., paraffin—specific gravity 0·890–0·910.

The mean percentage yield is—

Lignite-tar benzine	2– 6 per cent.
Photogene	8–30 ,,
Solar oil	12–50 ,,
Lubricating oil	15–30 ,,
Paraffin	15–12 ,,
Residue (asphaltum)	14–20 ,,
Gases	3–10 ,,
Water	5–20 ,,

H. Vohl compiled the following table showing the percentage of various products from different kinds of lignite tar (100 parts by weight) :—

Number of Sample.	Light Oil. Specific Gravity 0·82.	Lubricating Oil. Specific Gravity 0·87.	Paraffin.	Asphaltum.	Carbolic Acid, and Loss.
1	33·40	40·00	6·70	17·30	2·40
2	33·50	40·00	3·30	18·10	5·60
3	17·50	26·60	3·20	16·90	36·70
4	16·40	27·10	4·30	14·30	37·80
5	17·50	26·20	5·00	18·60	32·50
6	17·70	26·60	4·40	17·50	33·70
7	15·50	11·10	3·40	22·20	47·50
8	16·60	18·00	4·40	11·10	49·70
9	16·30	19·50	3·40	13·10	47·70
10	10·60	19·30	1·20	16·90	51·80

These figures reveal very considerable differences in the amount of the several distillates, more especially in the column referring to "carbolic acid and loss," the proportions of which range between 2·40 and 51·80 per cent., the increase indicating the relative unsuitability of the lignite for the recovery of tar.

With regard to the tar from Bohemian lignite, Hodurek states that it has the same chemical composition as that from Saxon lignite, and can be treated for the recovery of the same marketable products. The benzol, toluol, and xylol fractions are unsuited for nitration purposes on account of the impurities they contain, but find extensive employment as motor spirit, as solvents, and for carburetting, etc. The disagreeable smell possessed by all crude fractions from lignite can be removed by suitable refining processes, so as to furnish products not inferior in this respect to those from coal-tar, and suitable for replacing the latter as extractive and carburetting agents. After suitable purification the middle fractions give excellent water-white burning oils (solar oil), that can be disposed of profitably, whilst the resinous oils and tar acids from the refining process can be used as impregnating materials. The tar acids may also be worked up into crystalline carbolic acid, characterised by faint smell, pure white colour, and above all,—in contrast to the product from coal-tar,—by the fact that it is not liable to become discoloured on prolonged storage. The small quantity of tar bases present can be used for denaturing alcohol. The final runnings of oil may be used as impregnating oils, either in the crude state or, preferably, after chemical purification. They are also suitable for gas-making, though they do not yield quite so well as the corresponding petroleum fractions. The crude paraffin obtained by pressing the heavy oils yields a product of sufficiently high melting-point for candle-making when refined with sulphuric acid and caustic soda. The yield of paraffin is generally inferior to that from Saxon lignite; and therefore, especially in the case of small works, it is sometimes preferable to sell it in the crude state to a petroleum refinery instead of treating it on the premises.

EXAMINATION OF VARIOUS RAW MATERIALS FOR PARAFFIN AND DISTILLATES.

Scottish Boghead Coal.—This coal is brown to black in colour, and cleaves easily in the bedding planes, but is difficult to break transversely, and has the specific gravity 1·15–1·26. It is easily inflammable, and continues to burn with a bright flame. The 18–27 per cent. of ash mainly consists of alumina and silica. Like the tar-bearing lignites, it is very rich in hydrogen, containing up to 11 per cent. calculated to the dry coal, free from ash. It distils easily, without fusing, and yields 31–35 per cent. of tar, of specific gravity 0·860, which seldom contains more than 5–6 per cent. of fine bluish white but soft paraffin, melting at about 45–46° C. In comparison with the low paraffin content, the yield of oil is greater than that from lignite tar.

Russian Boghead Coal from Räsan.—The tar from this coal is of specific gravity 0·910, which leads to the conclusion that no large quantity of light oils can be present. The oils are water-white and consist of—

Naphtha	0·780	specific gravity.
Kerosene (solar oil) (1) .	0·815	,,
,, ,, (2) .	0·820	,,
Yellow engine oil (1) .	0·880	,,
,, ,, (2) .	0·890	,,

Paraffin is recovered in the form of scale cakes.

In France the beginning of the shale-oil industry dates from 1830. On account of the very keen competition to which this industry was afterwards exposed by petroleum, the French Government in 1890 offered a premium on every hectolitre of shale oil produced; and this caused a great revival of the industry, which was also able to take advantage of the great improvements effected in the allied Scottish industry. In the vicinity of Autun and Buxières there are nineteen large and small distilling plants in operation. The bituminous shales, occurring at a depth of 150–300 feet, are raised by means of shafts near the distilleries. The retorts used are of cast iron, parallelopiped in form and set in strong brick-work. The gases mostly condensed during the distillation of the shale are utilised as fuel, and the spent shale is let down, through sliding doors, into the furnace, where it is immediately ignited, by its own inherent heat and that of the walls, and assists in distilling the succeeding charge.

A ton of shale will furnish—

9–15 gallons of crude oil,
11–14 gallons of ammonia water, and
880–1235 cubic feet of permanent gas.

In the Scottish shale-oil industry considerable attention is devoted to minimising the production of permanent gases, by suitably controlling the distillation temperature to about 320–430° C. At the Clippens Oil Works, five miles south of Edinburgh, about 4000 tons of shale are distilled per week, and the average yield per ton includes—

25 gallons of crude oil,
2½ gallons of highly volatile distillate, and
22 lb. of sulphate of ammonia.

The illuminating oil (sunlight oil) has a flashing-point of about 95° F. and gives about 10 per cent. more light than Russian petroleum (flashing at 84° F.), or 50 per cent. more than that of imported American petroleum (flashing-point 70° F.). The other products made at this works comprise: naphtha for outdoor lighting and use in linoleum manufacture, gas oil for making the gas used in lighting railway carriages, lubricating oils, paraffin for candle-making (over 60 tons a week being refined), coke from the crude oil stills, and sulphate of ammonia.

The following table (from the *Colliery Guardian*) shows the progress made by the industry in the course of twenty years :—

	1871.	1880.	1890.
Number of Works	51	18	13
Tons of shale treated . . .	800,000	850,000	1,869,300
Production—			
Crude oil (gallons) . . .	25,100,000	29,000,000	52,876,700
Illuminating oil (gallons) .	11,250,000	11,400,000	21,680,000
Lubricating oil (gallons) .	2,500,000	5,000,000	9,000,000
Paraffin (tons)	5,800	9,200	22,846
Sulphate of ammonia (tons) .	2,320	4,750	18,488
Capital invested	...	£1,300,000	£2,583,982
Number of miners employed .	...	2,200	3,500
Annual coal consumption (tons).	...	350,000	600,000

The American paraffin industry is a very important one, and the bulk of the output is consigned to England, which country is unlikely to be rivalled by any other as a consumer. During the first nine months of 1906 the shipments of American paraffin to England totalled 70·8 million lb., whereas Italy, the second largest consumer, took only 9·85 million lb., and Germany 9·2 million lb., Japan being not very much behind with 9·15 million lb. The total exports of paraffin from the United States for the period mentioned were 126·5 million lb., or about 15 million lb. in excess of the previous year, whilst in the first nine months of 1904 the exports were about equal to those of 1906.

According to Wagenmann's experiments, laminaceous shales of the kind occurring in England, Hesse, Siebengebirge, and

Rhenish Prussia, furnish varying quantities of distillates yielding 5–20 per cent. of tar, from which can be recovered—

0·10– 6·00 per cent. of crude spirit,
3·00–12·00 „ crude oil (sp. gr. 0·850–0·900),
0.25– 1·00 „ paraffin.

The following particulars were obtained by Vohl :—

Rhenish Shale, so-called Paper Coal—

Photogene	32·50	per cent.
Solar oil	0·33	„
Paraffin mass	51·25	„
Coke	8·92	„
Loss	1·00	„

Westphalian Shale—

Light oil	27·50	„
Lubricating oil	13·65	„
Paraffin	1·12	„
Asphaltum	12·50	„
Carbolic acid and loss	45·30	„

Oeding Shale—

Light oil	18·33	„
Lubricating oil	38·33	„
Paraffin	5·00	„
Asphaltum	13·33	„
Carbolic acid and loss	25·80	„

Rhenish Shale No. 2—

Light oil	25·68	„
Lubricating oil	43·00	„
Paraffin	0·12	„
Asphaltum	10·00	„
Carbolic acid and loss	19·10	„

English Shale—

Light oil	24·28	per cent.
Lubricating oil	40·00	,,
Paraffin.	0·12	,,
Asphaltum	10·00	,,
Carbolic acid and loss . . .	25·29	,,

Büchner gives the following figures on the composition of tar from Bohemian bituminous shale:—

Liquid constituents . . .	20·94	per cent.
Coal, dust, and coke . . .	6·84	,,
Ash	74·00	,,

One hundred parts of the raw material furnishing—

6–10 per cent. of tar water,
4–12 per cent. of tar, with 6–10 per cent. of light oils,
6–10 ,, heavy oils,
1– 2 ,, paraffin,
12–25 ,, gas, etc.

Schädler gives the following table of the distillates obtained from various bituminous shales:—

Shale from	Tar.	Tar Water.	Coke.	Gas, Loss.
	Per cent.	Per cent.	Per cent.	Per cent.
Westerwald . .	10·85	9·23	58·68	21·24
Bielefeld. . .	4·90	9·00	59·85	16·25
Province of Saxony.	9·00	6·53	70·27	13·44
Teplitz . . .	8·45	7·68	70·48	13·42
Meissen . . .	10·50	10·00	61·10	15·40

One hundred parts of tar, specific gravity 0·865–0·890, yield—

Shale from	Light Oils.	Heavy Oils.	Paraffin.	Residue.	Creosote, Gas, and Loss.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Westerwald	30·45	31·32	3·12	22·63	22·48
Bielefeld	26·55	36·46	4·00	15·67	17·32
Province of Saxony	22·46	38·32	2·85	16·91	19·46
Teplitz	18·50	38·50	3·50	19·50	20·00
Meissen	20·00	40·50	3·25	24·75	21·50

The tars from the various kinds of wood, especially beech, also contain paraffin. The amount of tar obtained by the dry distillation of wood depends on the time and temperature of the process, and also, to a considerable extent, on the kind of wood itself, resinous woods yielding more than the non-resinous kinds. By slow distillation the former give 9–14 per cent. of tar, the latter 5–11 per cent. The more rapid the distillation, the larger the yield of tar and gas and the smaller the amount of acetic acid. At temperatures above 300° C. the tar furnishes on distillation: benzol, toluol, xylol, cumol, naphthalene, paraffin, phenic acid, cresyl-phenyl, and phlorylic acid.

Thenius obtained the following products by distilling wood-tar with superheated steam :—

Acetic-acid water	5	per cent.
Light crude wood-tar oil	20	,,
Heavy ,, ,,	25	,,
Wood-tar oil containing paraffin	15	,,
Wood-tar pitch	30	,,
Loss in distillation	5	,,
	100	

At low temperatures the wood-tar obtained by distillation with superheated steam is of the consistency of ointment, chiefly due to its high paraffin content, and contains a relatively small amount of tar pitch. The light wood-tar oils are more easily refined to a nearly water-white condition than those from charcoal tar and gas tar.

Dr. Bersch reports that the hydrocarbons present in wood-tar and boiling between 360 and 400° C. are classed as paraffins, and vary in melting-point from 40 to 65° C., according to the material furnishing the tar.

The distillation of crude petroleum furnishes the following products :—

German Petroleum from Elsass and Hanover (Oelheim).

		Spirit and Light Oils.	Solar Oil.	Lubricating Oil.	Paraffin.	Loss and Residue.
		Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Elsass oil	No. 1	23·41	23·30	35·64	...	15·65
,,	,, 2	25·92	16·20	45·60	...	12·28
Hanover oil	,, 1	44·20	...	47·60	1·10	7·10
,,	,, 2	42·70	...	50·00	2·00	5·40
,,	,, 3	42·90	...	49·50	2·50	6·10

An oil, of specific gravity 0·908, from Oelheim gave—

Good petroleum	15 per cent.
Heavy ,, ...	23 ,,
Spindle or vaseline oil . . .	21 ,,
Finest engine oil	18 ,,
Hard pitch	7 ,,
Sludge and water	3 ,,
Loss in distillation	12 ,,

Wietze Petroleum (after Dr. Haepke, Bremen).

1. From the higher zone: thick, dark brown, specific gravity 0·935–0·950—

    0·5 per cent. of benzine, water-white, specific gravity 0·725,

6·00	per cent. of	I^a petroleum, water white, specific gravity 0·80,
6·00	,,	II^a petroleum, yellow tinge, specific gravity 0·840,
20·00	,,	spindle oil,
22·00	,,	engine oil No. 1,
22·00	,,	engine oil No. 2,
10·00	,,	tar, yielding 10 per cent. of oil containing paraffin and 30 per cent. of coke.

2. From the lower zone: dark green, specific gravity 0·805—

1·00	per cent. of	benzine, specific gravity 0·701 at 15° C.,
2–3·00	,,	ligroin, ,, 0·727–0·730,
28·01	,,	naphtha and petroleum,
47·00	,,	lubricating oil,
17·00	,,	of residuum, containing a little paraffin, and including 4–5 per cent. of loss.

Light Wietze light oil from the lower zone furnishes about 23 per cent. of lamp oil and benzine, and about 77 per cent. of residuum, complete distillation giving—

about	3	per cent. of	benzine,
,,	20	,,	petroleum (lamp oil),
,,	5·5	,,	solar oil (gas oil),
,,	49·5	,,	vaseline oil,
,,	17	,,	tar,
,,	5	,,	gas and loss.

The oil contains about 3·8 per cent. of paraffin.

Heavy Wietze oil from the upper zone, when distilled for vulcan oil, yields—

about	6	per cent. of	petroleum,
,,	14	,,	gas oil (solar oil),
,,	35	,,	asphaltic acid resins,
,,	40	,,	vulcan oil,
,,	5	,,	loss.

Or when distilled for pale lubricating oil—

about	6	per cent. of	petroleum,
,,	14	,,	gas oil (solar oil),
,,	10	,,	asphaltic acid resins,
,,	40	,,	spindle oil,
,,	25	,,	tar,
,,	5	,,	loss.

Galician petroleum furnishes on the average—

about	5	per cent. of	benzine,
,,	50	,,	petroleum,
,,	5	,,	paraffin,
,,	10	,,	lubricating oil,
,,	10	,,	gas oil,
,,	3	,,	coke.

Galician Oils.

Specific Gravity.	Petroleum Spirit and Benzine.	Lamp Oils.	Vaseline Oil.	Paraffin.	Residue and Loss.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
0·856	8·50	56·45	12·62	2	22·43
0·800	15·10	50·00	8·00	8	24·00
0·782	20·00	...	...	8	22·00
0·859	10·00	50·00	10·00	...	31·00

Italian Oils (according to E. Graefe).

Veleja crude, specific gravity 0·787, at 17·5° C., flashing-point below $-2°$ C.

Paraffin Content.—Owing to the low percentage of paraffin present, the determinations made with the oil itself gave no result. For this reason the oil was evaporated down to one-twentieth its volume, and the residue tested by the Zaloziecki method, giving 0·12 per cent. of paraffin melting at 45·4° C. in the capillary tube.

Montechino crude oil, specific gravity 0·789 at 17·5° C., flashing-point below − 2° C.

The paraffin content of this oil, determined by the same method, was 0·027 per cent.

Baku Petroleum.

Specific Gravity of Crude Oil.	Lamp Oil.	Lubricating Oil.	Residue and Loss.
	Per cent.	Per cent.	Per cent.
0·783	67·80	29·30	2·90
0·775	89·10	9·00	1·90
0·770	92·90	...	7·10

(The last of these oils is white naphtha from Durachany, and contains no product of the lubricating oil class)—

Per cent.	Per cent.	Per cent.	Per cent.
0·905	32·95	41·70	25·35
0·910	32·13	43·50	24·15
0·886	48·00	32·00	20·00
0·896	40·60	39·70	9·70
0·900	29·40	60·10	10·50

Caucasian crude petroleum from Tscheleken Island. According to Charitschkoff, the specific gravity is 0·868 at 15° C., and the flashing-point 51·5° C., Abel-Pensky. Fractional distillation yielded the following distillates :—

	Per cent. of distillate.	Specific gravity at 15° C.
At 200° C.	1·8	0·7712
„ 250° „	11·9	0·7950
„ 270° „	7·6	0·8095
„ 310° „	6·6	0·8218

At ordinary temperatures the residuum had the consistency of ointment; specific gravity 0·900 at 22·5° C.; paraffin content 5·5 per cent., from which a yield of 6–7 per cent. would seem to be recoverable on a manufacturing scale.

Argentina Petroleum (Baker).

6·00 per cent. of naphtha, specific gravity 0·700,
29·00 ,, lamp oil, ,, 0·814,
53·00 ,, heavy oil, lubricating oil, specific gravity 0·900,
10·00 ,, coke,
2·00 ,, gas.

New York Petroleum.

17·00 per cent. of naphtha, specific gravity 0·700,
9·00 ,, benzine ,, 0·700,
64·00 ,, lamp oil ,, 0·783,
10·00 ,, residuum and loss. The residuum is said to contain 2 per cent. of paraffin. This oil is typical of the class of petroleums which furnish only benzine and lamp oil, and no lubricating oils at all.

California Petroleum.

	Specific Gravity 0·818.	Specific Gravity 0·826.
	Per cent.	Per cent.
Petroleum spirit (0·715 and 0·745) . .	16·20	22·80
Light oil (0·760)	6·80	...
Lamp oil (0·810)	56·30	...
Burning oil (0·820)	...	50·10
Lubricating oil	11·00	18·30
Paraffin	0·50	0·65
Coke	4·80	2·60
Loss (gas)	4·40	5·75

Canadian Petroleum.

	Specific Gravity 0·835.	Specific Gravity 0·843.
	Per cent.	Per cent.
Oil of specific gravity 0·720–0·820	48·30	50·45
Lubricating oil	43·70	39·40
Paraffin	3·00	4·34
Coke and loss	5·00	5·81

Pennsylvania Petroleum.

	Specific Gravity 0·813.	Specific Gravity 0·802.	Specific Gravity 0·800.	Specific Gravity 0·795.
	Per cent.	Per cent.	Per cent.	Per cent.
Lamp oil	71·00	58·70	41·00	50·65
Lubricating oil	17·50	39·40	39·00	36·20
Paraffin	2·00	2·00	2·00	2·85
Coke and loss	10·00	2·90	18·00	10·00

Chandler gives the following general classification of the distillation products of American petroleum:—

Cymogene, boiling at 0° C., liquid only under heavy pressure.

Rhigolene, boiling at 18° C., condensable by means of refrigerating mixtures. Used as an anæsthetic.

Gasoline, 1·5 per cent., condensable by cold water.

Naphtha, 10 ,,

Benzine, 4 ,,

Kerosene or refined petroleum, 55 per cent.

Vaseline oil, 19·5 per cent.

Coke and loss, 10 ,,

Turkestan crude petroleum from Zakany yields on distillation—

1. Various grades of benzine, such as petroleum spirit, ligroin, benzoline, motor spirit, extractive benzine, etc.

2. Ordinary petroleum (kerosene), the yield of which can be raised from 30 per cent. to 46 per cent. by means of the cracking process.

3. Liquid fuel, which has a great future before it in Turkestan.

4. Vaseline and paraffin. The amount of the latter is 8 per cent. of the residuum, so that with an annual output of 8 million pood of crude oil, the paraffin therein contained amounts to 5000 tons.

Berikel Crude Petroleum (Daghestan).

This oil, of specific gravity 0·8741 and flashing at 35·5° C., gave on distillation—

1·5	per cent. of	benzine and ligroin,
34·5	,,	lamp oil, specific gravity 0·4957, flashing-point 28·5° C.
60·7	,,	residuum, specific gravity 0·921, flashing-point 127° C.

Only about 0·3 per cent. of paraffin was detected.

Mineral Oil from New South Wales (Torbanehill).

The crude mineral oil obtained by the distillation of Torbanehill consists mainly of paraffins and olefines, the latter forming 70 per cent. of the light distillates, whilst the rest of the oil, distilling between 280 and 400° C., is almost exclusively paraffin. The naphtha, forming 9 per cent. of the crude oil, consists of nearly equal parts of paraffins and olefines, with the specific gravity 0·660–0·800 and boiling at 30–200° C. The paraffins, pentane to decane, amount to 30–50 per cent. The solar oil, boiling at 200–270° C. and of specific gravity 0·800 to 0·870, contains 50 per cent. of paraffin in the lower fractions, increasing, with the boiling-point and specific gravity, to 80 at 270° C. (dodecane to pentadecane). The product commences to boil at about 270° C., the distillate depositing solid paraffin on cooling. The specific gravity ranges from 0·870 upward.

Burgmann gives the following particulars on the percentage of various distillates in different mineral oils :—

New York Crude Oil.

Naphtha (specific gravity 0·700)	12–15 per cent.
Benzine (,, 0·730)	9–12 ,,
Lamp oil (,, 0·795)	60 ,,
Paraffin	2–2½ ,,

Galician Crude Oils.

	Bobrka.	Boryslaw.
	Per cent.	Per cent.
Lamp oil (best quality)	50	...
,, (second ,,)	...	50
Light oils	10	20
Heavy oils (suitable for use)	10	8
Paraffin	...	14
Tar for wagon grease	...	...
Residuum	...	...
Loss	20	...

Russian Oils (Caucasus).

	Per cent. of Kerosene (lamp oil).
Black naphtha from Baku yields	33
White ,, ,,	90
Naphtha from Tiflis yields	22
Naphtha from the Caucasus yields	40–46

One hundred parts by weight of peat furnish the following average results on distillation :—

Gas	5–15 per cent.
Peat water	25–40 ,,
Tar	2– 6 ,,
Residue	30–40 ,,

According to Vohl, 100 parts by weight of peat tar yield—

Number.	Light Oil. Specific Gravity 0·820.	Lubricating and Gas Oil. Specific Gravity 0·870.	Paraffin.	Asphaltum.
	Per cent.	Per cent.	Per cent.	Per cent.
1	8·36	8·51	0·142	7·00
2	15·22	18·25	3·10	20·20
3	18·00	19·45	3·64	24·30
4	19·40	19·50	4·00	26·80
5	21·54	26·50	4·68	26·00
6	27·70	27·50	5·32	42·22

Creosote, carbolic acid, and loss: 10 per cent. (1), 38·52 (2), 36·50 (3), 39·80 (4), 40·00 (5), 40·50 (6).

Minimum yield of—

Light oil, specific gravity 0·820	10·62 per cent.
Heavy oil, ,, 0·860	19·37 ,,
Paraffin	1·30 ,,

Maximum yield of—

Light oil (0·820)	33·41 ,,
Heavy oil (0·860)	40·06 ,,
Paraffin	6·70 ,,

According to Schädler, the average percentage content of peat-tar is—

Peat oil (benzine)	4–8 per cent.
Photogene	11–30 ,,
Solar oil	15–30 ,,

Lubricating oil	15–45 per cent.
Paraffin	½–10 ,,
Asphaltum	15–25 ,,
Gas	3–10 ,,
Water	5–20 ,,

With regard to the products of the distillation of peat, Professor Zaloziecki says that if the mean figures be taken and calculated to percentages of the oil distillate (5 per cent.), the following results are obtained :—

1·00 per cent. of	benzine at 150° C.
3·10 ,,	petroleum at 150–300° C.
0·90 ,,	heavy oils above 300° C.

The heavy oils can be worked up in the usual manner to creosote, paraffin, etc. The quantities of phenols in the oil distillates are small, but usually increase with the still temperature, the average yield being about 0·05 per cent. of the total weight of raw material. The percentage and yield of paraffin in the oils are about the same, when crude oils, low in paraffin, are used, which is preferably the case, it being inadvisable to employ residues high in paraffin for this purpose, because the high boiling-point of the paraffin causes it to remain for the most part in the peat in the still. The best material to use for the process is heavy oil (gas oil) that has been previously refined, because this class of oil is always a saleable article, which is not always the case with residuum (masut); and besides, the working conditions are more favourable. The peat water from the still, when titrated to ascertain the acetic acid content, shows that the higher the still temperature, the larger the yield of acetic acid. In one case under consideration, the actual proportion of acetic acid in the peat water amounted to 1·11 per cent., or 0·16 per cent. of the total yield. Ziegler gives the yield of sulphate of ammonia as

0·47 per cent., and of methyl alcohol 0·27 per cent., so that a total charge of 10 tons of material, *i.e.* 7½ tons of air-dry peat and 2½ tons of petroleum residuum (masut), would yield—

14,740	lb. of	peat charcoal,
495	,,	naphtha coke,
220	,,	benzine distillate,
682	,,	petroleum distillate,
11	,,	crude phenols (creosote),
11	,,	paraffin,
176	,,	lubricating oils,
244	,,	crude acetic acid,
88	,,	sulphate of ammonia,
44	,,	methyl alcohol.

MANUFACTURE OF PARAFFIN FROM PYROPISSIT.

Pyropissit is a special variety of lignite, which, in the state in which it comes from the mine, is a light yellow, almost greasy mass, containing up to 70 per cent. of water, and drying in the air to form a whitish mass of earthy appearance. On being heated it fuses to a hard mass, which intumesces strongly as the temperature is raised, and burns with a very bright, smoky flame, leaving behind a fair amount of ash. Pyropissit suitable for the recovery of paraffin should not reveal any vegetable remains in its structure, but should be yellow, almost greasy, and assume an earthy character on drying. When properly distilled, this substance will yield up to 16 and even 20 per cent. of tar, from which gas oil, photogene, solar oil, and vaseline oil can be recovered. The density of the tar fluctuates between 0·840 and 0·910, the lighter kinds being well adapted for the recovery of paraffin. The method of distillation has a considerable influence on the quantity of tar formed, and still more so on the percentage constitution of the tar itself. Overheating must be avoided in any event, since contact with the hot walls of the still will greatly reduce the amount of valuable products contained in the vapours. Formerly the operation was conducted in retorts similar to those used in gas-works; but latterly this system has been found defective, and abandoned in favour of distillation by steam. By this means, not only is a maximum yield of tar obtained, but at the same time the decomposition of the valuable tar constituents is prevented. In fact, the introduction of steam distilling may be said to have raised the tar distilling industry to a stage of development which has rendered it independent and fit to assume an important place among the chemical industries.

The apparatus used in distilling consists of vertical, horizontal, and rotary retorts, such as those of Larsen, Bakewell, Lahore, Gingembre, and others, though this latter class has not proved successful in practice.

The old lignite distilling plant was very similar to that used in gas-works, except that far larger retorts could be used, the temperature required to expel the liquid products from pyropissit being considerably lower than for coal. A comparison of the yield of tar obtainable in the laboratory and on a manufacturing scale respectively showed, however, considerable differences—in some cases as much as 50 per cent.—which were traceable to the decomposition of a large proportion of the distillation products by contact with the red-hot walls of the still. Owing to this circumstance and to the inconvenience of the intermittent working necessitated by the use of the ordinary form of retort, many attempts were made to modify the apparatus in such a manner as to prevent overheating and render the process continuous. One of the most extensively used types of still at that time was the upright cylindrical, or shaft form, into which the material to be distilled is fed at the top and sinks down by its own weight, thus gradually coming into hotter regions of the shaft, which is heated from the outside. By means of pipes arranged at intervals from that portion of the shaft where distillation commences, down to the point at which it may be regarded as finished and nothing remains but coke, the vapours given off in the process are conducted to condensers, and thus removed from the influence of the heat at the moment of their formation. The finished coke sinks down still lower, and is removed through lateral doors; but before this is done, the space below the column of distilling material must be shut off by means of a sliding plate, to prevent the ignition of the material which would ensue if air were admitted. Notwithstanding this device, it is impossible to shut out the air entirely; and this is shown by the presence in the tar of products that can only be formed when free oxygen gains access to the charge in the still, and which are not found in the tar from tightly closed retorts. The best results, both in the quantity and uniformity of the tar, are obtained by distilling with superheated steam, the apparatus for which process is of extremely simple construction. The stills consist of upright cylinders, closed at the bottom by a sliding plate for the removal of the coke, the coal or pyropissit to be distilled being fed in through the top. Each still is provided at the upper end with a pipe for carrying off the vapours, which pipes debouch into a main trunk where the vapours condense, the tar running down into a tank. Each pipe is also fitted with a valve, so that any of the stills can be shut off when desired. The steam for the distillation is generated in a boiler plant of suitable dimensions and is passed through a superheater.

In commencing operations, the steam must be admitted in large quantity and at a high temperature, so as to warm the whole of the charge right through; but as soon as the temperature is high enough for distillation to proceed vigorously, the admission of steam is checked, and is regulated thereafter in accordance with the progress of distillation.

The distillates condensing (with the water) in the warm, are led into a receiver, where they are left for a while, to allow the water to separate, and are then distilled. The recovered tar may be distilled in the same kind of apparatus that is used for fractionating crude petroleum; but in most cases a simpler form of still is used, the distillation being an easier process than is the case with petroleum, and the number of fractions smaller.

The stills for lignite tar are generally horizontal cylinders, of small diameter, this form being preferred because it gives the greatest uniformity of heating. In some cases trunk stills are used, with rather flat tops in order to retard the cooling of the upper portions that are out of contact with the fire. The condensing plant, which is a very important feature, is of the ordinary worm type, and must be fitted with steam pipes, in order to warm the condensing water at the commencement of distillation, so as to prevent the products, which are solid at the ordinary temperature, from choking up the apparatus and forcing the distillates back into the still.

It is beyond the bounds of possibility to free the tar from water completely, and consequently the first portions of the distillate are rendered turbid by the presence of small drops of water in suspension, so that they have to be left to rest for a time to allow the water to separate out. To enable the water to separate from the tarry distillates in the receiver, the liquid dropping from the mouth of the condenser is caught in an apparatus of the same type as that used for collecting the distillates from ethereal oil stills. This consists of a Florentine flask, fitted with a pipe that rises from the bottom to nearly the height of the neck and is bent twice at right angles, after which it dips downward for a short distance. When two liquids of different density—*e.g.* benzine and water—collect in this flask, the heavier (water) sinks to the bottom, and as the quantity of the lighter liquid increases, the hydrostatic pressure forces the water into the upcast pipe, whence it overflows, whilst the flask itself fills up with the benzine. This receiver is usually connected direct with the mouth of the condenser, a lateral pipe, issuing from the junction, leading the uncondensed vapours under the fireplace, where they are consumed and utilised for heating. When it is desired to recover these products, the pipe in question is connected with a condenser immersed in a mixture of ice and salt; but up to the present this process has not been applied in tar distilleries, the uncondensed products being consumed as fuel.

Since the tar recovered from pyropissit and other lignites yielding tar always contains a certain amount of sulphur, it is treated with $\frac{1}{4}$–$\frac{1}{2}$ per cent. of perfectly dry quicklime, and the still is charged with the crude tar until the latter reaches to within 20 inches of the manhole cover when the still is cold.

At the commencement of distillation, the gaseous constituents of the tar form the bulk of the first distillate, including sulphuretted hydrogen, ammonium sulphide, and ammonia. When the tar is rich in sulphur, these gases should be led directly into the chimney stack and not burned under the fire, or the sulphur dioxide formed during combustion will corrode the metal of the still. At the start the temperature is raised to 100° C. as quickly as possible, because up to that point nothing but gaseous hydrocarbons, water, ammonium sulphide, and ammonia is driven off. When the temperature exceeds 100° C. the light hydrocarbons begin to pass over, and care must then be taken to set the condenser in operation at once. Whilst the temperature of the charge is rising from 100 to 250° C., the light oils are distilled over, their amount being usually very small. When the temperature reaches the last-named limit, and the volume of liquid coming over recedes to a minimum, the receiver is changed, and the temperature is carefully raised. This done, an increased quantity of distillate soon begins to come over, and the operation is allowed to proceed until the vapours begin to be accompanied by paraffin. The men in charge of the still usually have a very simple test for finding out when this is the case—namely, by dropping a small quantity of the distillate on a cold plate of bright metal. If the drop remains perfectly liquid, the distillate still consists of liquid hydrocarbons; but if it thickens like ointment, this is a sign that solid hydrocarbons are coming over, whereupon the receiver is again changed, steam being also admitted to the condenser tank for a short time and the supply of cold water shut off. When the bulk of the paraffin-bearing oils has distilled over, it is followed by a distillate that, in addition to containing a large proportion of paraffin, is also charged with other solid bodies such as chrysene, pyrene, and anthracene. This brownish or greenish distillate is known as paraffin grease, and is collected separately. Of late, the anthracene content of this product has rendered it more valuable, and it is treated for the recovery of that substance. In many cases, however, the percentage is very small, though in others the paraffin grease is fairly rich in anthracene. At the end of the distillation a coky mass, utilised as fuel, is left behind in the still.

The yield of the various products recovered from tar by fractional distillation fluctuates between rather wide limits, according to the character of the pyropissit used and the manner in which distillation is conducted. As a rule, the following quantities are obtained:—

	Per cent.
Gas	1·5– 2·5
Liquid products, and oil containing paraffin	90 –86·5
Paraffin grease	7 – 8
Coke	2 – 3

To refine the oils containing paraffin, the first thing to be done is to free the mass from water as completely as possible, the simplest way being to melt the paraffin and keep it in that condition for several hours, during which period the small amount of water present separates out completely. The molten mass is next transferred to a lead-lined tank, where it is treated with 3–5 per cent. of commercial sulphuric acid, poured in, as a fine spray, through a leaden sieve, air being blown simultaneously through the mass, in order to secure intimate admixture of the acid and paraffin. After this treatment has been continued briskly for 15–20 minutes, the contents of the tank are kept liquid for several hours (4–8), to allow the impurities time to subside. The sediment is next removed, and the mass is washed repeatedly with warm water, and finally with a solution of sodium carbonate to neutralise the residual sulphuric acid completely. In many cases, especially when the material under treatment is strongly contaminated with oxidation products, it is redistilled at this stage; but usually it is now ready for crystallising.

The large, sheet-iron crystallising tanks are arranged in special rooms that can be cooled to a low temperature, the degree of cooling depending on the nature of the tar.

After being suitably cooled for a sufficient period, the paraffin forms a pappy mass, which is treated in filter presses to expel the greater part of the products that are still liquid at the temperature employed. The scaly crystals of paraffin remaining in the press cloths after this gentle preliminary pressing are refined further by means of hydraulic presses in which the mass is treated, cold at first and afterwards in the warm, until finally the residual mass has a melting-point not lower than 50° C., though generally the treatment is continued until a product melting at 55° C. is obtained.

The resulting paraffin scale is next refined with benzine, as mentioned elsewhere. With regard to the benzine used, it should be remarked that the kind employed for the first treatment of the paraffin should have the density 0·74–0·75, but that for the refining of the scale from the first treatment must not be heavier than 0·70–0·71. The reason for this is because the final traces of benzine adhere very tenaciously to the paraffin, and can only be expelled by blowing steam through the molten mass, so that if heavier benzine, which has a higher boiling-point, be used, high-pressure steam will be necessary, and this is attended with numerous inconveniences. Hence, the proper kind of benzine is desirable, though it must not be supposed that it is better in other respects; for, as a matter of fact, this light benzine is so volatile that considerable losses by evaporation during the process are unavoidable.

The paraffin, after this refining, is pure white in colour, but does not exhibit the similarity to alabaster that is characteristic of the best grades of paraffin. Consequently, it has to be filtered by means of bone-black, the residue from cyanide manufacture, or aluminium-magnesium hydrosilicate, as mentioned elsewhere.

The final distillate (paraffin grease), coming over after the true paraffin-bearing oils, is treated in the same manner as these latter, the fatty mass being well pressed in filter presses, treated with benzine, etc. A smaller amount of benzine is, however, sufficient, and in many cases the treatment is not carried so far as to produce perfectly white paraffin, the value of the finished product being too low to render the full treatment profitable, the paraffin being of low melting-point and unsuitable for candle-making.

SCHULKE'S METHOD OF RECOVERING PARAFFIN FROM LIGNITE AND OTHER TARS.

That tar is distilled under ordinary or reduced pressure, and with or without the aid of ordinary or superheated steam, is a matter of common knowledge, as is also the pressing of the resulting paraffin in filter presses, centrifugalising machines, etc. In addition, numerous attempts have been made to recover paraffin from tar without distillation, by treating the crude tar with alcohol. Among these is the patented method of A. Schultze (German Patent 162, 341) in which the tar is merely freed from the most volatile constituents, such as photogene, the lighter paraffin-bearing oils and similar products, the residual granular-crystalline mass, consisting of the heavier oils, being separated into its components—solid paraffin and heavy oils—by filter pressing, centrifugalising, etc., as usual. The volatile fractions are driven off by distillation in any usual manner. The paraffin present, as such or in the form of bitumen, in the tar oils, can be recovered by fractional distillation in the usual way. The paraffin taken out of the press, etc., is ready for use immediately after being refined—with bleaching earths, bone-black, and volatile mineral oils—in the ordinary manner.

It is claimed that this method effects in a simple manner what has hitherto been attempted in vain—namely, the recovery of all the paraffin formed in the distillation of the raw material, without the necessity for distilling the tar. The chief advantages hereby secured are stated to be that the crystallised product is rendered independent of atmospheric conditions; that a larger yield of hard paraffin is obtained; that a commercial article is obtained from the pressings by a simple refining, thus reducing the accumulation of soft scale to a minimum; and that, finally, the method can be applied in existing works without difficulty.

The following is a typical example of the use of the method in the case of lignite tar :—

Two and a half tons of lignite tar are heated to 130° C. in a still, and steam is blown in until about 6 cwt. of distillates have passed over. The still residue is then left for about 36 hours to crystallise, and the crystalline product separated by the aid of a centrifugal machine, etc., the heavy oils being expelled and leaving the solid paraffin in the form of a cake.

This mass is afterwards pressed, if necessary, to remove any small amount of adherent non-crystalline matter, and then refined.

RECOVERING PARAFFIN FROM WOOD-TAR.

At present the recovery of paraffin from wood-tar is no longer of any practical importance, since the product can be obtained far more cheaply from petroleum and lignite tar; nevertheless, the process should not be omitted from a work like the present. The tar is first carefully distilled, the condensed water and light distillates being collected separately. The heavier oils containing the paraffin are put through a process of rectification, the distillates then formed being placed in wooden tanks in a cellar to crystallise. With a medium temperature, crystallisation will be complete in a fortnight to three weeks. The crystallising tanks may be of wood or sheet iron, and should be large enough to hold one day's output from the stills. At various heights they are fitted with draw-off taps to enable the liquid oil to be drawn off as crystallisation progresses. They are set up in a cool cellar, and mounted about 30 inches above the floor, so that receptacles can be placed underneath to catch the oil drawn off from the tanks. The scaly paraffin crystals are taken out of the tanks and separated from the still adhering tar oil in a centrifugaliser, the residual mass being wrapped in linen or woollen cloths and exposed to a pressure of 200,000–300,000 lb. in a hydraulic press. Twelve cloths form the usual charge for a press, but more can be taken if thinner cakes are desired; and in this case the pressing is more effectual, but the output of the press is reduced. The pressed paraffin is left standing in the press for $\frac{1}{2}$–$\frac{3}{4}$ of an hour, pressure being again applied in the meantime. The cakes are then taken out to make room for a fresh charge, which has been got ready in the interim. The cakes from the press are yellow, brown, or even black in colour, and are next transferred to a vessel where they are melted by steam, the liquid mass being left for the impurities to settle down, which done, the clarified paraffin is strained through a cloth, treated with 10 per cent. of rectified oil of turpentine or benzine, and pressed into cakes about 1½ inches thick. When cold, these cakes are wrapped in cloths again and subjected to a hydraulic pressure of up to 500,000 lb., which forces out the soft adherent hydrocarbons. The pressed cakes, which are nearly white, are melted by steam, which is allowed to blow through the mass for some time, so as to remove any still adhering oil. The liquid mass is transferred to a lead-lined tank, where it is treated with 8–10 per cent. of sulphuric acid, well stirred, left to settle, the clarified paraffin being drawn off into a clean tank. Here the adhering acid is washed out with warm water and neutralised with a little caustic soda lye. To complete the clarification, the paraffin is remelted with 2 per cent. of stearine, saponified with a small quantity of caustic soda lye, left to settle, and separated from the solution of soap by skimming or decantation. If it be desired to omit the soda treatment, the acid-refined paraffin is remelted with benzine, pressed in a hydraulic press when cold, and treated in a settling tank with a powerful current of superheated steam, which completely removes all volatile and odorous oils, the liquid mass being finally filtered through purified bone-black. This latter method furnishes a very fine quality of paraffin, but is attended with a considerable loss of material, especially in the second pressing.

To obtain paraffin from the heavy wood-tar oils on a small scale, the oily distillate, which contains a quantity of crystals, is treated gradually with ordinary rectified spirit, until it becomes very cloudy and separation of paraffin occurs. The paraffin is filtered off, washed with cold spirit, then dissolved in hot spirit and recrystallised. A further quantity of paraffin is recovered by evaporating the mother liquor, and is purified by recrystallisation.

To complete the purification, Reichenbach advises suffusing the paraffin with twice its volume of sulphuric acid, warming the mixture to 100° C., stirring well, and digesting for a short time, followed by washing with water, dissolving in alcohol, recrystallising (several times repeated), and finally drying.

Pure wood-tar paraffin has the following properties: when melted and recooled it forms a white, transparent mass, as clear as glass, in thin layers, with a crystalline laminated structure and a faint nacreous lustre resembling spermaceti, which it also feels like, though more tender and slippery; it is also soft and fragile, but tasteless and inodorous, and does not make a greasy mark on paper. The specific gravity is 0·870, and at 43·75° C. it melts to a colourless oil, which volatilises without change when heated more strongly in closed vessels. It will not ignite in the mass, but burns with a fine bright flame in a wick. Heated to evaporation point in presence of air, it can be easily ignited, and burns with a bright clear flame, without leaving any residue. Paraffin is insoluble in water, somewhat soluble in alcohol, and more so in ether and ethereal oils; and it can be melted in association with stearine and fatty oils. It is characterised by great indifference toward other substances, since at the ordinary temperature it is neither acted upon nor decomposed by chlorine, nitric acid, hydrochloric acid, or sulphuric acid.

SINGER AND MUNKELT'S METHOD OF RECOVERING PARAFFIN FROM MINERAL OILS AND SIMILAR HYDROCARBONS.

The English Patent No. 3702 of 1881 describes a method of separating paraffin from mineral oils by dissolving the oils in a mixture of petroleum spirit (benzine, gasoline), naphtha, and alcohol (methyl, ethyl, or amyl alcohol), and then depositing the paraffin by cooling. On the other hand, the German Patent No. 123,101 protects a method of separating paraffin from lignite tar by dissolving the tar in hot alcohol (up to

90 per cent. strength) in a closed vessel, and causing paraffin of various melting-points to separate out by cooling. This method is also applicable, under certain conditions, to the recovery of paraffin from mineral oils, thus avoiding the drawbacks accompanying the use of the English method—namely, that the presence of the solvents neutralises the precipitating action of the alcohol, soft paraffins in particular being thus prevented from depositing; and even the separation of the paraffins of medium melting-point is impracticable except by the aid of costly refrigerating plant.

On the other hand, alcohol of 90–95 per cent. strength is not a solvent of mineral oils, so that even the German process mentioned above is not directly applicable to the recovery of paraffin from mineral oils. Hence, this operation is still carried on in the old complicated, troublesome, and imperfect manner.

It has now transpired that, under certain conditions, all the paraffin-bearing oils and mineral oils, especially distilled oils, with the exception of small quantities of contaminated oxidisable hydrocarbons (resins), can be dissolved with ease, and a very clean separation effected between the oils or liquid hydrocarbons and the paraffin and resin, by means of the method specified in German Patent No. 140,546—namely, using alcohol of at least 95 per cent. strength, and preferably 99 per cent. The mixture of oil and the necessary quantity of ethyl alcohol to effect solution is suitably heated. So long as the solution is maintained at 50–80° C. the paraffin is kept in solution, whilst the insoluble hydrocarbons (resins), which occur more particularly in water-free oils that have been stored for some time after distillation and are strongly acid in reaction, separate in the form of small drops, which fall to the bottom and can be easily removed. The temperature at which the paraffin is deposited depends on circumstances, and especially whether the whole of the paraffin in solution is to be thrown down, as well as on the desired melting-point and yield of paraffin, and on the cold test of the residual oils to be worked up into lubricating oils; but in no case is it necessary to lower the temperature to the extent required in existing methods. The chief advantages of the method rest on the employment of one and the same liquid as the dissolving and precipitating agent, and also on the fact that the solvent can be recovered, by distillation, for use over again, at little expense and with comparatively little loss. Solution and precipitation can be carried on in relatively smaller vessels than those used in existing methods to produce the same effect. The solution of oil can be readily and completely separated from the paraffin mass by pressing or other suitable means, so that the resulting paraffin scale is perfectly free from oil. Moreover, the elimination of the colouring matters—which mostly remain dissolved in the alcohol—and of the oxidisable hydrocarbons —which are removed mechanically—greatly facilitates and cheapens the subsequent treatment of the scale. The yield of both soft and hard paraffin is much higher than in the ordinary methods, in which considerable losses are sustained, both in the separation of the soft paraffin and oils from the hard paraffin and in the refining and bleaching with bone-black.

Further investigations have shown that the foregoing method can be modified by substituting a more dilute and cheaper spirit (93 per cent. strength) provided the solution process be carried on under a pressure of 2–4 atmospheres, thus reducing the cost of the treatment.

GARVEY AND STRANSKY'S METHOD OF RECOVERING WAX-LIKE OIL-FREE PARAFFIN FROM MINERAL OILS.

The paraffin-bearing crude oils contain, in addition to crystalline paraffin, wax-like modifications of paraffin, which cannot be recovered by existing methods. The importance of being able to isolate these waxy paraffins may be gathered from the circumstance that the non-crystalline ceresine of high setting-point—which is produced only in the vicinity of Boryslaw, and that too in comparatively small amount—is worth four or five times as much as ordinary commercial paraffin.

Garvey and Stransky claim that the following method enables a product equal to ceresine in wax-like appearance and high setting-point to be isolated from mineral oils and from the crystalline paraffins dissolved in the latter.

The method consists in first removing the paraffin that crystallises out from the oil at ordinary temperature by aspiration, filtration, or hydraulic pressure. This paraffin is chiefly of the waxy modification. By the ordinary method of cooling the oil down first, a mixture of crystalline and waxy paraffin is obtained, in which the former predominates, and the two cannot be separated by any known means, whereas the present method furnishes an almost pure non-crystalline paraffin, which can be purified by pressing in hydraulic presses heated by steam, or preferably hot water, and controlling the temperature. Provided the temperature of the press plates be gradually raised to about 65° C., while pressure is being applied, the adherent particles of crystalline paraffins, with a setting-point of 69–74° C., are melted out and the non-crystalline paraffin is left.

It should be noted that the object of warm pressing in the ordinary paraffin process is to eliminate the adherent oil, which is more easily effected at high temperatures. On the other hand, the purpose of warm pressing in the present method is to get rid of the crystalline paraffin retained in the waxy paraffins which have been separated from the oil before cooling.

RAGOSIN'S METHOD OF RECOVERING PARAFFIN FROM LUBRICATING OIL RESIDUES, ETC.

The presence of paraffin in Russian petroleum was unknown previous to 1881, and nearly all petroleum technologists were opposed to the idea of its existence therein. In fact, the absence of paraffin was made the criterion for differentiating Russian oil from American. The first detection of paraffin in Russian oils was made accidentally at the Ragosin works, owing to a stoker having allowed the temperature of one of the stills charged with lubricating-oil "goudron" to fall below the proper temperature of 350° C., and having then cut off the steam supply until the correct temperature was regained. This, of course, resulted in dry distillation, and decomposition products were expelled from the still with violence, on to the floor, and yielded crystals of paraffin. Ragosin then carried out the process to a finish, without altering the conditions, and obtained a solution of paraffin in solar oil, the still residue consisting of coke. In continuing his research work on petroleum, Ragosin found that the crude oil contained no paraffin, but ozokerite, and that this latter distilled over as paraffin at a higher temperature, accompanied by light distillates. The same result was obtained on repeating the experiment with more highly superheated steam, and Ragosin afterwards replaced the latter by benzine vapours, with which a new force is brought into action—namely, the almost infinite mutual solubility of benzine and petroleum.

A certain minimum quantity of benzine is, however, required, so that the mixture of benzine and petroleum vapours distilling over may have sufficient pressure to issue from the apparatus. The approximate proportion of benzine is about 60 per cent. by weight of the petroleum distillate vaporised. In distilling residues with superheated steam, the proportion of steam to oil vapours at the commencement of distilling solar oil is 1 : 1, but only 6 : 1, or even 20 : 1, at the end of the process. The benzine method, however, has its disadvantages. On the one hand, the condensing vapours, especially the lighter fractions, retain part of the benzine vapour, and the latter in turn carries away some of the vapours of the light fractions. At a mean temperature of 300° C. the amount of heat required per 100 parts by weight of the raw material, is equal to that sufficient to vaporise 47 parts of water; and as 300° C. is obtained by the combustion of 1½ lb. of fuel oil, the consumption of fuel amounts to 5 per cent. of the oil to be treated. In the present distillation process, in which a yield of 70 per cent. is obtained from the residue, the calculated amount of fuel is 30 per cent. Ragosin's figures, even if considerably exceeded, will always keep below 30 per cent., and still more because about 2½ times as much steam (by weight) is required as in the case of benzine, and the heat consumed in vaporising the former is three times as great as with the latter. In addition, the distillation with benzine is carried on at a lower temperature throughout.

In addition to paraffin, Ragosin found Russian petroleum to contain ozokerite, the existence of which depends on the extent to which this substance has been converted into paraffin in the depths of the earth. He also points out that Galician ozokerites can be transformed into paraffins. From Galician crude oils he obtained up to 50 per cent. of burning oil, then useless lubricating oils, paraffins (decomposed ozokerite), true ozokerite, and petroleum gases. In these petroleums the decomposition process was terminated at an earlier stage than in the case of American oil. From his researches Ragosin forms the following conclusions: Every freshly tapped petroleum and every heavy residue from a light petroleum contains in a recoverable form—

1. Hard petroleum pitch, forming a valuable material.
2. Ozokerite, unless this has already been converted into paraffin in the earth.
3. Paraffin.
4. Lubricating oils, provided the crude oil is of the primary type.
5. The remainder, except the usual products already distilled off, can be transformed into petroleum or residuum free from pitch.

According to Wischin, the benzine method enables the distillation to be carried on at a lower temperature, because the benzine vapour dissolves successively higher-boiling fractions out of the oil, thus lowering the boiling-point of the mixture and eliminating the evil effects produced on the progress of distillation without decomposition by excessively high temperature. Another point in which benzine vapour is superior to steam is that the solvent action ensures better separation of the fractions and increases the yield of light hydrocarbons. This is because it dissolves the light hydrocarbons out of residues in which they are present in association with asphaltic bodies, without carrying off the latter, which, according to Holde and others, are only sparingly soluble in benzine. The solid hydrocarbons occurring as protoparaffins in the crude oil and converted into the crystalline modification by decomposition in distillation, are recovered as protoparaffins during the Ragosin process, whereas when steam is used they are usually found in the residue. To produce decomposition with the Ragosin apparatus, the vapours are passed through a heated cylinder on their way to the condenser. Owing to the more uniform constitution of the vapours, by reason of the causes already mentioned, the decomposition proceeds in a more uniform manner, without loss from the formation of gas and coke.

The fundamental principles of the Ragosin method may be briefly enunciated by the statement that the fractionation of the petroleum or residuum is effected in a manner different from ordinary processes of distillation (*i.e.* by the progressive heating of the hydrocarbon mixture, and thus gradually vaporising fractions of higher boiling-point)—namely, by passing a current of light hydrocarbon vapour at a lower temperature than that required for the decomposition of the mixture, which vapour produces a species of fractional solution advantageous to the course of the operation, whether subsequent decomposition be effected or not. The relatively low temperature on the one hand, and the lower consumption of fuel needed for vaporising the benzine on the other (in comparison with water), leads to a considerable saving of expense.

The Ragosin still consists of two cylinders of elliptical section, mounted at a converging angle, the lower one being the still and the other the cracking cylinder. At the end where the cylinders approach each other, a vaporising pipe, *p*, is provided, extending into the upper part of the cracking cylinder. When the cracking stage is to be omitted, the upper cylinder is disconnected by closing a valve (not shown in the drawing) in the pipe *p*, and opening a second

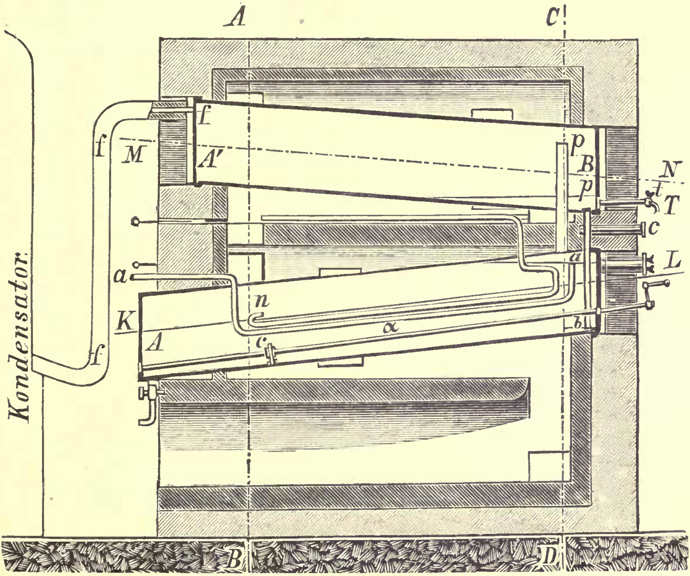


FIG. 4.—Ragosin Still (longitudinal section).
(Kondensator = Condenser.)

valve, through which the vapours pass direct to the condenser without traversing the upper cylinder. The fireplace is situated under the still proper, though the hot furnace gases lave the walls of the cracking cylinder, except when the latter is disconnected, in which case they are diverted into the smoke stack by closing a valve.

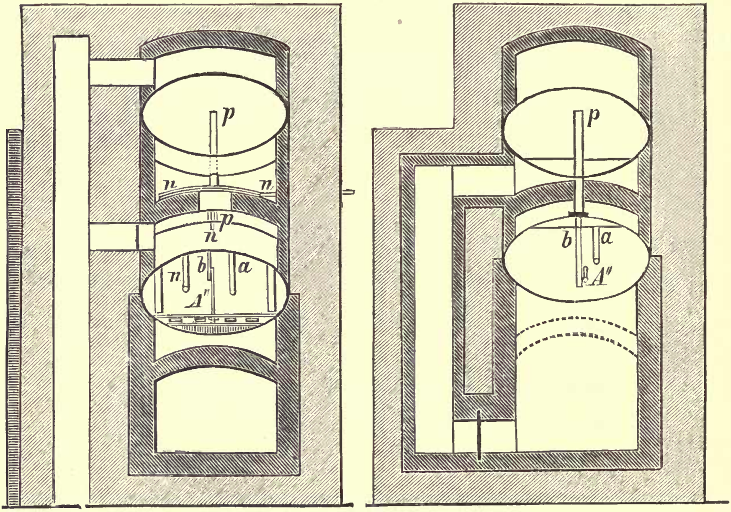


FIG. 5. FIG. 6.

Ragosin Still.

Cross section along A—B of Fig. 4. Cross section along C—D of Fig. 4.

During the distillation, fresh supplies of the raw material are run continuously into the still from a high-level storage tank through the pipe *n*, whilst benzine vapour is blown into the liquid through a second pipe (not shown). This pipe may be situated at any convenient part of the still, taking care, however, to secure the most uniform distribution possible of the vapour in the bottom of the still by means of fine perforations in the feed-pipe. Though it is not difficult to regulate the inflow of oil by setting the feed-taps so as to maintain a constant level in the still, this level is also controlled automatically by a safety pipe, *a*, forming a water seal. Any surplus of oil overflowing is carefully collected apart. An outlet for the residue is provided at the deepest point of the

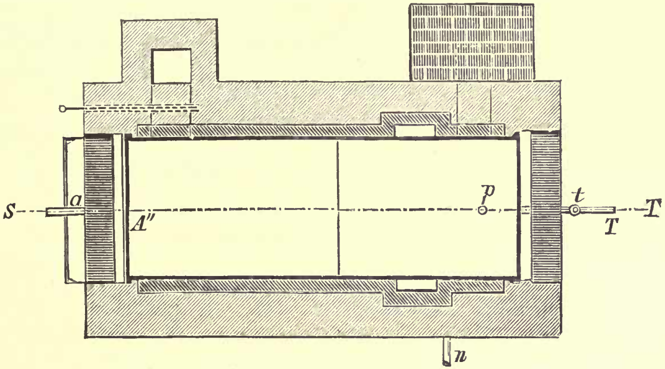


FIG. 7.—Ragosin Still.
Section through the cracking cylinder along M—N of Fig. 4.

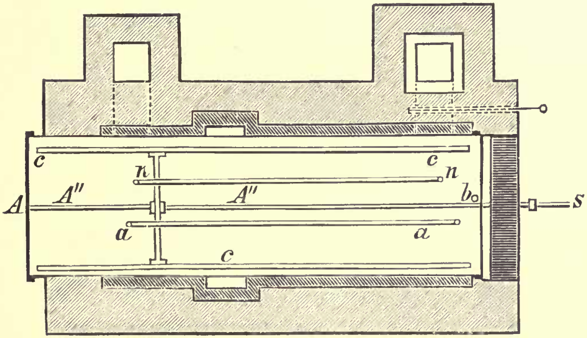


FIG. 8.—Section through the Still along K—L of Fig. 4.

cylinder, the residue being swept out by means of a steel wire brush, *c*.

The benzine vapours are generated in a separate apparatus. The most suitable benzine is that boiling at 80–160° C.; and the pressure in the generator is allowed to rise to about $\frac{3}{4}$ of an atmosphere. The stage at which the benzine vapour is admitted to the still depends on the character of the material under treatment—*e.g.* with Russian lubricating-oil residue of specific gravity 0·950, the vapour is admitted when the still temperature reaches about 280° C. The relatively low temperature of the operation when benzine is used may be gathered from the fact that in the case under consideration the distillation is already over at 360° C. The volume of the condensed benzine should be about 40 per cent. of the distillate.

When the cracking cylinder is used, the oil vapours carried off in solution by the benzine vapour are decomposed in the upper cylinder, which is suitably heated. Light hydrocarbons are formed, and these pass on to the condenser, whilst an oil rich in paraffin accumulates in the cracking cylinder and is drawn off through the tap *t*. Any form of fractionating condenser may be used, but Ragosin has constructed a special condensing tower in which the vapours pass upward through a succession of chambers, so that they undergo compression on issuing from each chamber and then re-expand in the one next above. Each chamber has its own draw-off valve for the condensed products, the heaviest fraction being of course collected in the first chamber and the lightest in the last of the series. A number of these condensing towers can be connected to form a system. As a rule, the last chamber of all merely condenses benzine vapour, and any portion of the latter that escapes condensation here is recovered in a water condenser for use over again. The light petroleum fractions present in the benzine condensed in the final chamber are separated by blowing superheated steam through the liquid in a separate vessel, this treatment expelling all the benzine and leaving the petroleum behind, the latter running away continuously through a pipe fitted with a water seal.

Trial distillations of Russian lubricating-oil residues (specific gravity 0·95) gave the following results:—

Without Cracking.

	Per cent.
Distillate free from paraffin	40–42
,, containing 50 per cent. of proto-paraffin (m. p. 40° C.) . .	42–40
Residue (m. p. 60–70° C.)	16–18

With Cracking.

Cracking petroleum (obtained by condensing the vapour from the cracking cylinder and recracking the expressed paraffin-bearing oil)	62
Paraffin (recovered from the oil drawn off at *t*)	20
Residue	13

METHOD OF SEPARATING LIQUID AND FUSIBLE SOLIDS, SUCH AS OIL, STEARINE, PARAFFIN, AND THE LIKE, IN FILTER PRESSES.

In existing methods of separating mixtures of liquid and fusible solid substances by filtration, the mixtures are placed in chamber or plate filter presses and filtered under suitable fluid pressure, the solid residue being afterwards placed between plates in a hydraulic press and subjected to higher pressure than is attainable in filter presses. This system possesses a series of drawbacks, chief among them being the necessity for using two kinds of apparatus, and the trouble involved in transferring the residues from one press to the other by hand, an operation particularly injurious to the health of the workmen in the case

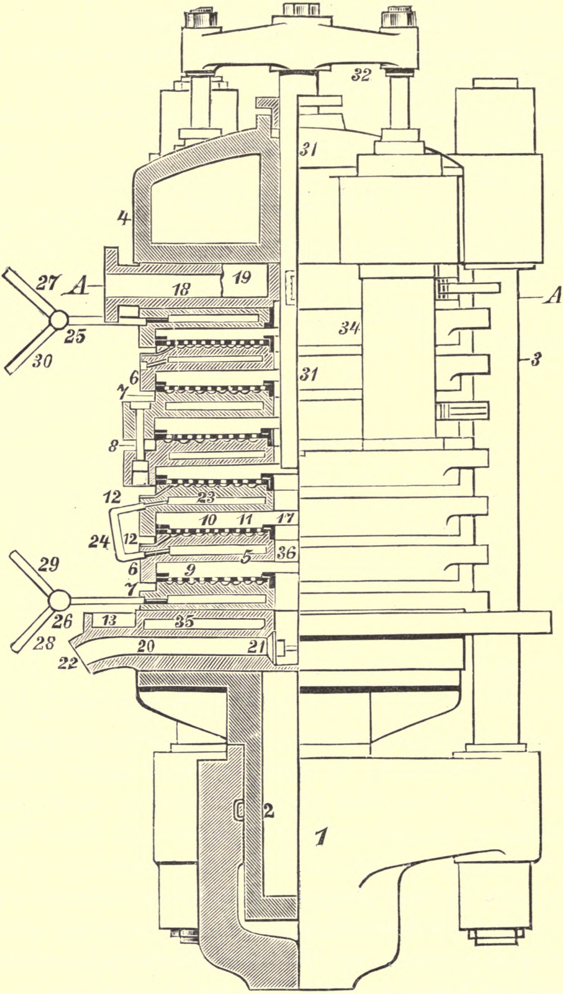


FIG. 9.—Filter Press for separating liquid and fusible solid bodies.

of the separation of paraffin from petroleum. The method described in German Patent 173,129 is claimed to remove these defects by effecting the filtration of the liquid and the transfer of the solid residue without hand labour and without dismounting the press, the melted residue being run out of the telescopic filter plates through a special device. The filter press is cooled, to separate the fusible solids (*e.g.* stearine or paraffin) from the liquids, and afterwards warmed to melt the residue, which is thereupon run out of the press unfiltered. The drawings show a press for carrying out this method, Fig. 9 being an elevation in partial section, and Fig. 10 a cross section along A—A, whilst Fig. 11 shows a detail, on a larger scale, of the arrangement of the passages in the plates. The press consists of the cylinder (1), plunger, standards, and press head. The filter plates are arranged between the plunger and the head, so that their edges (6) form guides for the next lower plate in each case, the escape of the material under treatment through the sides of the press being prevented by layers of packing material (7). The maximum distance between each pair of plates is determined by screws (8). The upper sides of the plates are also provided with a number of communicating grooves (9), covered by a sheet of metal or the like (10), and this in turn by the filter cloth (11). The expressed liquid runs away from each plate through passages or channels (12), and finally collects in an annular channel (13), mounted on a plate (20) on the press plunger (2), and separated from the plates by a space (35). Each plate is in two parts (Fig. 10), connected together by screws (16) and by a suitable central screw collar, which also holds the filter cloths firmly, and leaves a free space (36) in the centre for the passage of a plunger, as also for the introduction of the charge to be pressed. This latter operation is effected through a feed-valve (10) in the upper plate (18), whilst the melted residue is run off through an outlet valve (21) and pipe (22) in the plate (20).

The filter plates are hollow, for the admission of a cooling or heating liquid, and each of the hollow spaces is connected with its neighbour by flexible pipes (24) to enable the cooling or heating liquid to circulate through the whole press. The liquids are admitted through the tap (25) and flow out through the tap (26), steam being preferably introduced through the pipe (27) and drawn off through (28), whilst pipes (29) and (30) form the inlet and outlet respectively for the cooling liquid. The central orifices (36) are closed by means of a plunger (30)

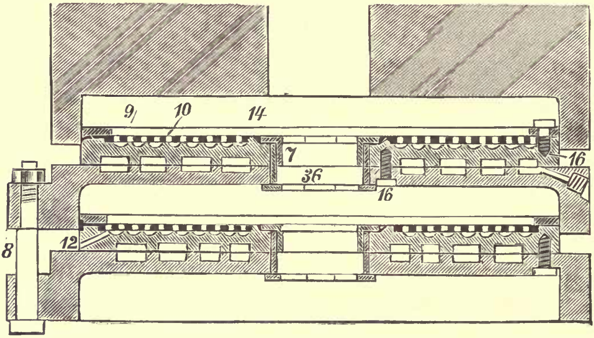


Fig. 10.—Filter Press for separating liquid and fusible solid bodies.

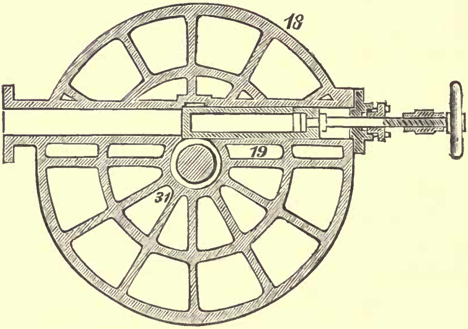


Fig. 11.—Filter Press for separating liquid and fusible solid bodies.

entering through a bore in the press head (4) and head plate (18), and being operated by water or steam, for which purpose it is connected by a cross head (32) with the plungers (33) of two cylinders (34), mounted by the side of the press standards, the said plungers being preferably operated by hand. The filter plates having been cooled by means of liquid admitted through the pipe (29), the paraffin-bearing oil is pumped into the press through the feed-valve (10) or run in from a high-level tank. The low temperature in the press causes the whole of the paraffin to deposit in the solid form, the pressure of the liquid forcing the oil at once through the filtering orifices into the space (9) and through the channels (12) into the collector (13). When the accumulated paraffin presents a greater resistance than the liquid pressure is able to overcome (*i.e.* when the hollows in the press are nearly filled with paraffin), the pressing begins, the central plunger having first been lowered into position in the press in order to prevent obstruction of the orifices (36) and between the plates, and in order to fill the interior of the press with residue under convenient pressure that is independent of the feed-pump. In its upstroke the plunger (31) sucks in material, and in its descent forces the latter into the cavities of the press, the operation being repeated until these are fully charged with residue under a certain pressure. This done, the plunger (2) is operated, the filter plates being heated if necessary, and forces the press plates together, thus squeezing the residue. In this operation each press plate forms the plunger acting on the next and transmitting the pressure from the plunger (2) throughout the mass in all the cavities of the press. When the pressing is finished and the outer surfaces of the plates and the collector (13) have been freed from oil, the residue is melted by steam, admitted through the pipe (27). The melted product runs away quickly through the opened valve (21) and pipe (22) without opening the press.

RECOVERING PARAFFIN BY THE AID OF ALCOHOL.

The method, which enables the whole of the paraffin in a tar to be recovered within ten hours, is based on the power of alcohol to dissolve all the resins, creosotes, and oils present in tar, without application of heat, thus obviating the separation of the resins by means of acid and alkali, hitherto indispensable in the production of commercial grades of paraffin. One hundred parts by weight of the tar are treated with 1000 parts of 90 per cent. alcohol in an autoclave at 80° C., the solution being run into iron cells and there cooled down to a given temperature. When the paraffin has crystallised out, the mass is centrifugalised, the whole of the alcohol, with the resins, creosote, and oils, being expelled. The residue (weighing 10–20 parts, according to the character of the tar) is washed with 100 parts of alcohol and freed from the final traces of the alcohol by heating it to 80° C. with a steam coil, the mass being finally decolorised with bone-black in the usual way. The resulting paraffin is superior to the ordinary products on account of its snow-white colour.

RECOVERING PARAFFIN FROM OZOKERITE.

Burgmann described a method for this purpose; and though at the present time, when refined ozokerite (ceresine) is so valuable that no one would think of treating ozokerite for the production of paraffin, the method is nevertheless worthy of mention.

The crude ozokerite is melted, and the light fractions are expelled by heating with steam, after which the temperature is raised, use being made of the Ramdohr method of superheating the steam by means of the liquid to be distilled. The apparatus consists chiefly of a still containing a coiled pipe communicating with a boiler. When steam is passed through the coil, the heat transmitted to the pipe effects the distillation of the volatile products. In proportion as the boiling-point of the contents of the still rises, so also does the temperature of the steam coil increase, the steam being thereby superheated up to a point corresponding with the distillation temperature of certain products. When the distillation temperature of paraffin is reached, any increase in the supply of steam merely accelerates distillation, without further raising the temperature in the still. It is evident that the apparatus used for the distillation of crude petroleum can also be modified without difficulty for the purpose now in view, this being the more easily effected since the number of products to be considered in the case of ozokerite is smaller than with petroleum. In working on a very large scale it is even advisable to distil off all the lighter fractions and leave only the paraffin behind, the distillates being fractionated in a suitable apparatus into products of various boiling-points. As soon as the still temperature has risen so as to cause the paraffin to begin distilling over, the taps through which cooling water is admitted to the condenser must be turned off, or the condenser will become choked with deposited paraffin and a serious explosion will result. Usually it will be sufficient to stop the flow of water shortly before the liquid fractions have ceased to run, since these will then warm the water in the condenser to a suitable extent. For the sake of safety, however, it is preferable to provide the condenser with a steam pipe, so that the water therein can be warmed rapidly in case of need. Small crystallising pans being unsuitable for working on the large scale, the paraffin-bearing oils are run into large tanks, from which the soft scaly masses of paraffin are afterwards removed. The preliminary refining of the mass is preferably effected in the filter presses already described, by which the liquid ingredients of the mass are run off without difficulty, the semi-solid to greasy crude paraffin with which the press was charged being changed into a fairly compact mass of crystals.

To reduce the number of pressings and keep the consumption of chemicals within reasonable limits, it is advisable to conduct the distillation in such a manner that two qualities of crude product are obtained, the light-coloured distillate coming over first being collected separately from the later dark portions. This second-grade mass may either be worked up separately or else used in the preparation of lubricants without any special refining.

The chief advantage in separating the distillates in this way is that the true paraffin mass is obtained in greater purity and can be refined with greater ease and at less cost than when the whole of the distillate is worked up together. In most factories benzine is used in the refining process, the crude paraffin being melted and treated with about 4–10 per cent. of benzine according to the extent of its contamination by impurities. As soon as the paraffin has dissolved, the solution is run into large sheet-iron or cement tanks and allowed to solidify under a layer of water. The lumps obtained by cutting up the solidified mass are then pressed in a hydraulic press: at first in the cold, so long as any liquid drains off; afterwards with gradual application of heat, the pressure being kept at the maximum. The melting-point of the paraffin should be carefully ascertained before pressing, the pressure being then regulated so as to keep it a few degrees below that point whilst in the press. A single pressing is rarely enough to purify the paraffin, two and even three pressings being generally needed. The benzine treatment should be repeated after each pressing, but with progressively smaller quantities of benzine, because of the reduced quantity of impurities present. Since the mass always retains a certain amount of benzine, which cannot be separated by pressing, it must finally be melted in a vessel by means of direct steam, which is blown through the mass and carries off the last traces of benzine. The steam and vapour are recovered in a condenser. The benzine process is followed by treatment with about 4-8 per cent. of commercial sulphuric acid and careful washing, finally with water containing a little stearine ($\frac{1}{8}$–$\frac{1}{4}$ per cent.) and 1–1$\frac{1}{2}$ per cent. of caustic soda lye (23–24° B.), to eliminate the acid and any impurities that can be removed by the alkali. Steam heat is applied until the stearine is completely saponified and the colouring matters of the paraffin are nearly all dissolved in the soap. The presence of nitric acid in the sulphuric acid is objectionable as it stains the paraffin yellow, which colour can only be removed by the aid of alcohol or sulphur dioxide. Finally the paraffin is filtered through bone-black, in iron boxes measuring about 40 inches in depth, 16 inches across the top, and 12 inches across the bottom. The granulated bone-black is laid in the bottom between two strainers, and a number of these boxes are placed in a tank or vat heated by direct steam sufficiently to keep the paraffin in a liquid condition. To accelerate the filtration, it is advisable to combine three to five of these filters to a battery, and to arrange them in tiers one above another, so that the liquid is forced through them, either upwards or downwards, by hydrostatic pressure. In the former case the vessel containing the melted paraffin to be filtered is mounted at a sufficiently high level to cause the filtered paraffin to flow spontaneously out of the topmost filter. The final traces of paraffin retained by the spent bone-black are recovered by passing direct steam inside each filter through a tap provided for that purpose, the admission being continued until steam begins to issue from the pipe through which the recovered melted paraffin drains off. When powdered bone-black is used, the recovered paraffin, though colourless itself, has a greyish appearance, due to the presence of fine particles of bone-black, which, however, can be removed by keeping the mass liquid for several hours, thus allowing the bone-black to subside and yielding a perfectly white supernatant paraffin. In practice this slow method of clarification takes up too much time, and the same effect is obtained by filtering the grey mass through felt, closely woven fabric, or filter paper, the pores of which must be too small to allow the finest particles of bone-black to pass through. The paraffin is ladled into moulds and is allowed to solidify, with all the precaution rendered necessary by its high contraction, the solidification being carried on very slowly.

RECOVERING WAX-LIKE PRODUCTS FROM PEAT, ETC.

According to E. Bauchard-Praceig, peat, lignite, or the like is extracted with suitable solvents, such as alcohol or ethylic ether, and the solution evaporated. The residual mass, which greatly resembles beeswax, is hard, of high melting-point, and has a specific gravity higher than that of water.

REFINING PARAFFIN.

As already remarked, the oil fractions obtained from the various tars, mineral oils, etc., are subjected to a cooling process, which results in the crystallisation of the paraffin, the latter being then freed from oily impurities by pressing, etc. This treatment is practically the same in all cases, the object being to purify the paraffin as much as possible. The products are soft-scale paraffins, and are cooled afresh in large tanks by the aid of ice or cooled brine, so that the temperature is very gradually reduced from 50–80° C. at the outset, to enable crystallisation to proceed to the best advantage. The larger the crystals, the easier the process, which usually takes from four to six weeks, according to the quantity and initial temperature of the paraffin. The still liquid portions are drawn off through tap holes near the bottom of the tank, and the residual paraffin is refined without any previous separation of the adherent oils.

More than half a century ago, Kernot reported, with regard to the refining of paraffin, that the best treatment was to suspend the crude paraffin in a vat provided with a perforated false bottom covered with a filter cloth, and to raise the temperature to the point at which the paraffin, but not the usual impurity, tar, would melt. The melting-point of paraffin being between 110 and 112° F., whilst that of the tar is 180° C., it is preferable to warm the mass to about 130° F., so that the paraffin will drain readily through the filter cloth. The heat is applied by passing steam through a coiled pipe in the vat. The tar and other impurities remain on the filter cloth, the paraffin being obtained as a pure filtrate. If necessary, the filtration is repeated. The liquid oil obtained as a by-product from the cooled and pressed paraffin may be completely decolorised by treating it at 110–200° F. with chlorochromic acid or the gaseous form of same, the paraffin being finally washed with warm water.

According to Mitchell—at about the same period—paraffin is refined by melting and digesting it for several hours with about 10 per cent. by weight of bone-black, the two being afterwards separated by filtration.

Subsequently, other methods of refining were introduced—namely, with sulphuric acid alone; with albumin and zinc chloride; by melting with stearine, followed by saponification; with bleaching powder, zinc chloride, etc.; but the treatment with sulphuric acid and caustic soda has proved the best for yielding pure grades of paraffin.

The quantities of these reagents employed vary according to the nature of the raw material, *i.e.* the tar, and the method of working. Graefe gives the following recipes:—

Reagents for Purifying Soft-Scale Paraffin.

Mix with $\frac{1}{2}$ per cent. of boric acid (50° B.) for a quarter of an hour, leave to settle for an hour, and draw off the product of the reaction.

Mix with 5 per cent. of commercial sulphuric acid (66° B.)

for half an hour, leave to settle for three hours, wash with 1½ per cent. of water of condensation, and leave to settle for an hour.

Mix with 2½ per cent. of soda lye (38° B.) for half an hour, leave to settle for two hours, and wash twice with 1½ per cent. of condensed water.

Reagents for Purifying Hard-Scale Paraffin.

Mix with ½ per cent. of boric acid (50° B.) for a quarter of an hour, leave to settle for an hour, and draw off the product of the reaction.

Mix with 6 per cent. of commercial sulphuric acid (66° B.) for half an hour, leave to settle for three hours, wash with 1½ per cent. of condensed water, and leave to settle for an hour.

Mix with 2 per cent. of soda lye (38° B.) for half an hour, leave to settle for two hours, and wash with 1½ per cent. of condensed water.

BURGMANN ON REFINING PARAFFIN.

The crude paraffin obtained by cooling and pressing mineral oil distillates still contains variable quantities of impurities, which must be separated or destroyed before the paraffin can be obtained as a colourless crystalline mass. The destruction of these impurities is effected by means of concentrated sulphuric acid, which leaves the paraffin unaltered. The cold-pressed cakes are pressed again at a temperature considerably below the melting-point of the paraffin, and usually at about 30° C., the residual mass being melted by steam heat in lead-lined vats and treated with 3–5 per cent. of concentrated sulphuric acid, the whole being well mixed and stirred. The acid chars or destroys the impurities and turns the mixture a dark colour, which done, the acid is eliminated by washing with hot water. The paraffin is next melted along with up to 6 per cent. of colourless photogene (specific gravity 0·790–0·810) and subjected to heavy pressure in a hydraulic press. Decoloration is effected by means of bone-black, which is placed in a steam-jacketed cylinder, the melted paraffin being filtered through it. Bone-black possesses the faculty of exerting a powerful attraction on colouring and odoriferous substances, and is therefore largely used in the chemical industry as a decolorising and deodorising agent.

The number of filters required for decolorising crude paraffin depends on the character of the crude product; the greater the contamination, the more frequently must the filtering process be repeated. Although the colouring matters can be removed so completely, by the aid of bone-black, that the paraffin appears perfectly white, even when moulded into thick blocks, this treatment will not entirely deodorise the mass; and as the absence of smell is regarded as a criterion of purity, the paraffin has to be put through a special treatment to secure this result. The substances causing the smell being far more volatile than the paraffin itself, though not so much so that they can be expelled by simply heating the mass, use is made of the known property of a current of steam for carrying off substances that are otherwise difficult to vaporise. An example of this is afforded by the case of boric acid, which is not volatile at the highest temperatures in ordinary circumstances, but can be rendered so to a considerable extent by the use of a current of steam. To apply this property in the refining of paraffin, the cakes of that substance are placed in a vat provided, at the bottom, with a finely perforated steam coil communicating with a steam pipe. By turning on the steam, the paraffin is rapidly melted, and the admission of steam is continued until all the odoriferous substances have been expelled, the colourless and deodorised paraffin being then poured into moulds, where it solidifies into blocks. In order to obtain a fine and highly transparent product, the crystallising process should go on very slowly, the formation of very large crystals being essential to produce the suitable degree of transparency and resemblance to alabaster. With this object the crystallising vessels are set in a place, usually a crystallising room, where they can be kept at a temperature of about 30–40° C., the room being closed after the vessels have been filled. The heat radiating from the hot paraffin keeps the room warm for a considerable time, thus retarding the cooling of the mass and favouring the production of large crystals.

This is, broadly speaking, the method generally adopted for refining paraffin, and is suitable for any kind of paraffin, whether derived from petroleum, ozokerite, or lignite tar. Various modifications of detail are, however, rendered necessary by the nature of the impurities accompanying the paraffin.

One method frequently employed is based on saponification, or rather emulsification, the paraffin being melted along with any inodorous fatty oil such as cotton-seed oil, olive oil, etc. This mixture is subjected to heavy pressure in hydraulic presses, to force out the greater part of the oil, which at the same time removes a large portion of the impurities. The operation is repeated several times, finally in a press the plates of which are moderately heated by steam, and furnishes a fairly pure paraffin, which, however, still retains a certain amount of fat that cannot be separated by mechanical means. To eliminate these final traces and the rest of the impurities, a saponification process is employed. The paraffin is melted by steam in a vessel fitted with stirrers, and a small quantity of weak caustic soda lye is added, the whole being kept warm and stirred until the oil has been saponified. The resulting soap is separated by washing with water, the paraffin being afterwards treated for a short time with a little sulphuric acid, and then transferred to a decolorising apparatus charged with bone-black. Although new methods are constantly being introduced, they all (says Burgmann) agree in this respect, that the paraffin-bearing distillates are strongly cooled, the crystalline mass being then subjected to heavy pressure and the residual paraffin refined with sulphuric acid and caustic soda, followed by filtration through bone-black, etc.

Another method for purifying paraffin (described in the *Oel-und Fett Zeitung*) consists in dissolving the solid hydrocarbons in benzine, so as to obtain a solution containing about 20–25 per cent. of solids, which solution is then intimately mixed with a decolorising agent, the relative proportions depending on the percentage of impurities present and on the reagent used, but generally amounting to 2–3 parts of reagent to one of paraffin, ozokerite, etc. The decolorising agent recommended is meerschaum dust, or, since this is expensive and rarely available in sufficient quantity, an artificial substitute prepared from water-glass (alkali silicate) and sulphate of magnesia, or else from magnesium silicate, anhydrous silica, and aluminium hydrate. When the decolorising agent is spent, it is washed with benzine, to recover all the adhering solid hydrocarbons, these being then freed from benzine by treatment in a current of steam. The regeneration is completed by drying (in the air or by artificial means) and by calcination in covered crucibles to destroy the colouring matters remaining from the solid hydrocarbons. This regenerated material can be used over again, preferably by mixing it with a certain quantity of fresh material. It is very important that all these decolorising agents should be used in a perfectly dry state; and as they are highly hygroscopic, they should be calcined before use. To obtain good results with a fresh charge of decolorising agent, the latter should first be saturated with a small quantity (about $\frac{1}{8}$ per cent. of the total) of the substance to be treated, this being dissolved in benzine. The solvent is expelled by heat and recovered in a condenser, and the saturated decolorising agent is calcined in crucibles or pots, which treatment increases its efficacy.

The necessary intimate admixture of decolorising agent and benzine solution is effected in a closed pan fitted with stirrers, the benzine being then recovered by the aid of a reflux condenser. When the mixing is complete, the mass is maintained at a temperature approaching boiling-point for ½–1 hour by means of a steam coil in the pan, the stirrers being kept in work the whole time. The entire mass is then either extracted by means of a large volume of benzine, or else is left to settle for 11–12 hours, so that the decolorising agent subsides, whereupon the clear, colourless solution of paraffin, or the like, is drawn off into a still where the benzine is expelled, the liquefied hydrocarbon residue being run into a collecting vessel outside. The spent decolorising agent is extracted with benzine (which is afterwards driven off by steam) and dried.

ADULTERATION OF PARAFFIN. TESTING.

Paraffin is rarely adulterated, but when this is the case, resin is the chief substance employed. In view of the considerable difference between the various grades of paraffin in respect of hardness, apart from the presence of added materials, the melting-point forms the chief criterion of value; and the higher this melting-point, the more valuable the sample under examination.

Burgmann gives the following particulars on the melting-point and specific gravity of various paraffins:—

		Melting-point.
Paraffin from	Boghead coal . .	45·5–52° C.
,,	peat	46·7° ,,
,,	Rangoon tar . .	61·0° ,,
		Specific gravity.
,,	Saxon lignite . .	0·871–0·875
,,	Boghead coal . .	0·873

	Specific gravity.
Paraffin from petroleum	0·878
,, Rangoon tar . . .	0·869
,, beechwood tar . . .	0·877
,, Rhenish shale . . .	0·877

MELTING-POINT AND REFRACTIVE INDEX OF VARIOUS PARAFFINS.

Soft Paraffin.

Melting-point . .	36–38° C.	40–42° C.	43–44° C.
	45–46° ,,	47–49° ,,	50–52° ,,
Refractive index .	23·6–23·8	23·7–24·2	24·6–25·3
	25·1–26·9	27·3–28·1	28·6–29·0

Paraffin Scale.

Melting-point	44–48° C.
Refractive index . . .	26·2–27·0

Hard Paraffin.

		German.	
Melting-point . .	50–51° C.	52–53° C.	59–60° C.
Refractive index .	27·9–28·4	28·9–30·5	30·0–30·8

	Javanese.	American.
Melting-point . . .	50–51° C.	52–53° C.
Refractive index . .	28·0–28·5	28·2–30·4

(Berg.)

R. Kissling gives the following instructions for the determination of the solidification-point of crude and commercial paraffin :—

A strong glass beaker (the thickness of the glass is comparatively immaterial) is charged with about 10–30 grms. of the melted, and perfectly clear, paraffin, enough being taken to ensure that the bulb of the thermometer used for stirring the mass will be constantly submerged to a depth of several millimetres.

In the case of ordinary commercial paraffin, the melting-point of which is generally between 50 and 60° C., Kissling uses the water-bath thermometer supplied with the official petroleum tester, but for crude paraffin he uses the oil thermometer of the same instrument. In any case the thermometer need only be graduated between 25 and 75° C. (a special form is only needed in exceptional cases) and should not be more than about 8 inches in length. The substance in the beaker is warmed or cooled to about 10° C. above its solidification-point, and the beaker is placed in a slightly wider glass charged with water at the same temperature. Though the relative size of the beakers does not greatly influence the results, it is preferable to choose them so that when the outer vessel contains 100 c.c. of water and the inner one 20 c.c. of substance, the level of the water is about 20 mm. higher than that of the paraffin, etc.

The substance is stirred with the thermometer until a flocculent deposit commences to form. The thermometer reading at this moment gives the solidification temperature of the substance.

J. Klimont mentions the following methods for detecting the presence of colophony in paraffin:—

1. The mixture is shaken up repeatedly in the cold with a small quantity of absolute alcohol, the filtered alcoholic solution is concentrated, and the residue is treated in the same manner until no more paraffin is deposited. This method is troublesome and cannot be recommended.

2. About 10 grms. of the mixture are heated to boiling in the water-bath with about 50 c.c. of 10 per cent. alcoholic caustic potash, then treated with an excess of water, and shaken up with ether in a separating funnel. The paraffin can be recovered by distilling off the ether; and the resin is determined quantitatively by decomposing the aqueous

solution with sulphuric acid and filtering. For obvious reasons this method is less suitable than the following:—

3. The same procedure is adopted, but the alcohol is evaporated to dryness after the caustic potash treatment, and an aliquot part of the residue is extracted with ether in a Soxhlet apparatus, the paraffin being weighed after the expulsion of the solvent.

4. For rapid, approximate determinations it is sufficient to ascertain the acid value of the substance under examination. Schmidt and Erban give the acid value of Austrian colophony as 146·5 (172·2 according to Klimont), and Williams found the corresponding values of refined and ordinary colophony to be 179·2–177·8 and 166·6 respectively. Taking the mean acid value as 170, whilst that of pure paraffin is nil, the calculation may be performed as follows: Assuming m to represent the volume of substance used for titration, s the acid value of the mixture, and x the percentage of resin present,

$$x = \frac{100\ s}{170\ m}$$

By means of this method, 13·72 per cent. of resin was found in a paraffin mixture containing actually 14·46 per cent. of resin.

The German association for the testing of technical materials has set up the following standards relating to paraffin in its regulations for the testing of petroleum products:—

The term paraffin shall be held to apply to solid hydrocarbons formed in the distillation of bituminous raw materials. The testing regulations relate only to crude paraffins, paraffin scale, and technically pure paraffin, "paraffinum liquidum" being left out of consideration.

1. The colour is the only external characteristic to be considered.

2. There seems no reason for prescribing any test relating to the fastness to light.

3. The specific gravity test for paraffin is chiefly an identification test, and may be performed by the specific gravity instrument at room temperature, taking care that the paraffin is free from air, or with the Mohr instrument at 100° C.

4. The determination of the solidification-point by the Shukoff method shall form the criterion of value for commercial paraffin, and also in the case of mixtures of paraffin with other substances. The Hall method is also permissible, as well as the melting-point test in a capillary tube, the commencing and final temperatures of liquefaction being determined.

The addition of other substances to paraffin is not admissible in general; in special cases the usual chemical tests should be applied.

The paraffin content must be determined by the Holde method after the removal of mechanical impurities and water. The percentage of impurities shall be determined by melting the paraffin and washing it with a solvent on a tared filter; the water content by distillation and weighing the condensed water collected, or else by heating a weighed quantity of the paraffin, the loss sustained by the evaporation of paraffin during the process being controlled by a check experiment with a weighed quantity of perfectly dry paraffin.

VERTICAL RETORTS FOR DISTILLING LIGNITE.

The vertical retorts preferably employed for the dry distillation of lignite are cast-iron cylinders (see A A Fig. 12), which were first introduced by Rolle. The dimensions are: height 13–17 feet, diameter $3\frac{1}{4}$–$6\frac{1}{2}$ feet. Internally they are fitted with a series of super-imposed bell-shaped rings, *a a a*, the sides of which slope outward, these rings measuring 6–12 inches in height, according to the size of the cylinder, and being attached, on the one hand, to a central arm by stays, and on the other, held apart by internal lugs. The bells thus

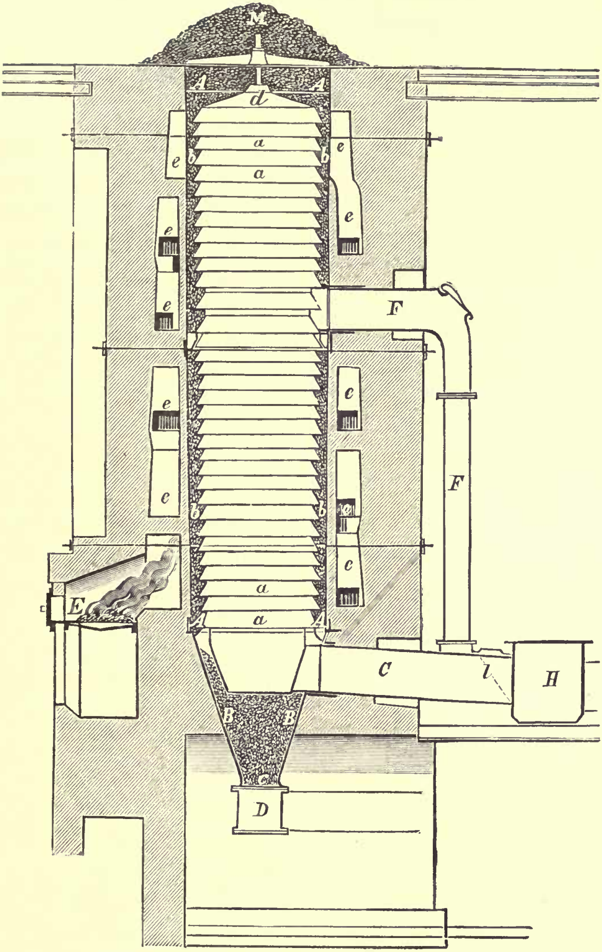


FIG. 12.—Vertical Retort for distilling lignite.

form a cylindrical chamber, communicating with the stratum of air *bbb*, between the bells and the cylinder, by means of the gaps left between them. The diameter of the bells is such that the air space *bb* has a width of 2½–4 inches. At the bottom the cylinder terminates in a cone BB, the usual height of which is rather more than one-fourth that of the whole cylinder. By means of an aperture *c* at the bottom, the cone communicates with a cylindrical chest D, with a capacity of 1–2 cubic feet, which can be shut off from the cone by a sliding door and emptied below by another door. Rolle also obtained good results with stills in which the iron walls were replaced by firebrick. Vertical cylinders are superior to the horizontal type, in the first place because they allow of automatically regulated continuous distillation, and also keep out the external air except the small quantity entering with the charge, thus preventing the oxidising and cooling effect to which the charge is exposed in horizontal retorts. Consequently the distilled tar is less charged with oxidation products than retort tar, and is therefore specifically lighter and is produced with a smaller consumption of fuel than the latter. As a rule ¾–1 ton of fuel will be needed for distilling a ton of lignite in retorts, as compared with only ½–¾ of a ton in the cylinders. On the other hand, however, a higher yield of tar, which though of greater density is also richer in paraffin, is usually obtained from retort tar, the larger amount of paraffin being probably derived from the empyreumatic resins which undergo conversion into paraffin during the subsequent distillation. The higher density of the retort tar is certainly due to the heavy resins present, since the distillates from these tars after the removal of the creosote are always specifically lighter than those from cylinder tar of the same density. The central cylindrical space of the cylindrical still is closed at the top by a bell cap, on to which the charge of lignite is thrown and from which it descends between the wall of the still and the column of rings. The still walls, which are about 2 inches thick and are covered externally with a strengthening surface of firebrick, are heated to incandescence by hot gases circulating round them through flues *eee* from the furnace E, and cause the distillation of the descending lignite, the products from which pass into the spaces between the rings and are led away, through two suitably arranged flues F and G, into the trunk H leading to the condenser, the flow being accelerated by an exhaust fan. The coke collects in the lower cone BB, and is let down at intervals of $\frac{3}{4}$–$1\frac{1}{2}$ hours, through the slide *c*, into the box D for removal. At the same time, fresh portions of the charge of lignite slip down from the pile of material heaped above the top of the cylinder. Whilst the coke is being discharged, the outflow pipe at the top of the cylinder is closed by a valve, to prevent air being drawn in from the coke box. The distillation process in this apparatus is therefore continuous, and the cylinders only need cleaning out at intervals of 6 weeks to 3 months, after which the work can be resumed. A cylinder about $16\frac{1}{2}$ feet high and $6\frac{1}{2}$ feet wide will treat about 20 tons of fresh lignite in 24 hours. The weight of the coal decreases later, on account of the removal of resin and creosote originating almost entirely in the contained heavy oils. This latter circumstance seems to favour the idea that the gases undergo considerable molecular rearrangement in the cylinder, and that paraffins are converted into oils, which must naturally be heavier than those distilling over without decomposition. This short description will suffice to show that the vertical retorts are not always superior to the horizontal type, although the latter may be the more difficult to operate. The choice of the best system for any particular case must be based on local conditions, and especially on the character of the raw material, fuel, and the labour market. As a rule, the cylinder is the best for lean materials, poor in tar and difficult to work, the more bituminous kinds, rich in tar and paraffin, being preferably treated in retorts, provided good fuel and a sufficiency of labour be available.

Another system, differing from both, is that of Ramdohr of Aschersleben, who distils the lignite in 6–7 ton retorts with superheated steam, condensing the resulting gases with cold water, and thus recovers about 65 lb. of tar per ton, a yield far exceeding that from any other process.

HORIZONTAL RETORTS FOR DISTILLING LIGNITE AND OTHER BITUMINOUS MATERIALS.

These retorts are of cast iron, of oval section (see Figs. 13 and 14) and measuring about 10 feet long, nearly as wide, and 18 inches high. The front end is closed by a manhole cover *b*, the rear end being fitted with a pipe *c*, which tapers to a diameter of about an inch and is fitted with a valve connecting it with a collecting main *d*, 3–4 inches in diameter and serving a whole battery of retorts 12–20 in number. The battery is heated from a common furnace *e*, the furnace gases being led transversely under the retorts (mounted on checker-work arches *ggg*) and then back over the top (which is covered with 3–4 layers of brickwork) through the flue *hhh*. The gases driven

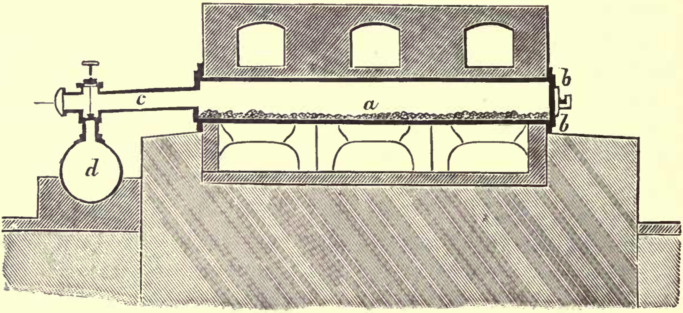


FIG. 13.—Horizontal Retort for distilling lignite.

off from the charge escape into the collecting main (which is sloped downward) and thence through a collecting chamber into the condenser, which is formed of a number of upright or horizontal sheet iron pipes, like those used in gas-works, the tar running back into the collecting chamber. The permanent gases, which are about equal in quantity to the tar produced, are discharged. Unsuccessful attempts have been made to collect these gases and utilise them for lighting purposes; but on the one hand the rate of flow may become so high as to lead to imperfect condensation, whilst on the other it may be retarded so as to set up back pressure in the retorts and delay the distilling process, besides leading to decomposition of the gases by prolonged contact with the hot retorts. The tar is run out of the collector and sent to the refinery. The retorts are charged through the door at the front, the charge being spread to a uniform depth of about $2\frac{1}{2}$–3 inches over the bed of the retort; and while this is being done, the draw-off valve at the front rear end must be closed, to keep the atmospheric air from being drawn into the collecting main along with the gases from the other retorts which are still in operation, since this would lead to gas explosions. Before recharging, the coke from the old charge is raked out

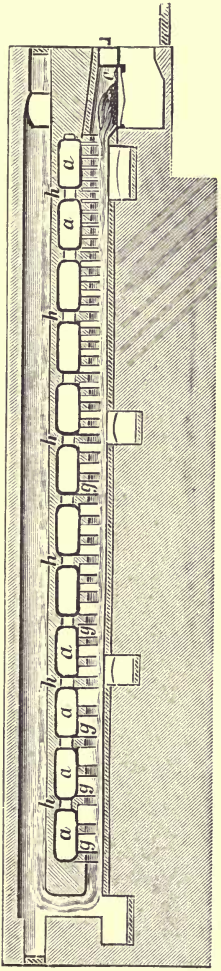


FIG. 14.—Horizontal Retort for distilling lignite.

and removed. About $2\frac{1}{4}$ tons of lignite can be treated per 24 hours in each retort, the time occupied for each charge being 6–8 hours. No exact figures, however, can be given, so much depending on the class of lignite and the strength of the firing. The lignite is preferably distilled in the state in which it comes from the pits—that is to say, so damp that it balls in the hand. In some works the distillation is assisted by steam, which is introduced through an $\frac{1}{8}$-inch pipe at the front end of the retort, when all the hygroscopic water has been expelled—that is to say, about half-way through the shift—the steam being turned off again before the retorts are emptied. The purpose of this steam is to protect the gases from being overheated, which it does in part by enveloping them and partly by accelerating their exit from the retorts.

COLOURING CERESINE AND PARAFFIN.

The object of colouring ceresine, and less frequently paraffin, is to increase the resemblance to beeswax. The colouring matters used are of vegetable origin, though latterly coal-tar dyes are also employed, especially the so-called Soudan dyes, a small quantity of which suffices to produce the requisite shade in a rapid and simple manner. Other colouring matters also used are annatto, gamboge, and turmeric.

Annatto is used in the form of the commercial paste, 1–2 per cent. of which is boiled with the ceresine over water until a white froth shows that the colouring matter has been taken up by the ceresine.

Gamboge is used for producing a pale lemon shade, but is generally too expensive. The ceresine is melted over water in a jacketed pan and boiled for a short time, the gamboge being added in the form of an emulsion with water. Boiling is continued until the colouring matter has been taken up, which stage is indicated by a greyish white scum, whereupon the whole is left to settle and the ceresine is drawn off from the sediment.

In the case of turmeric powder, the ceresine is heated to 100° C. in a large vessel, and 1–3 per cent. of the colouring matter is stirred in by degrees. Heating is continued for a short time, to extract the colouring matter, and the fine yellow-coloured liquid ceresine is drawn off from the sediment after a short rest.

The Soudan dyes are simply dissolved in the melted ceresine, but, as in the case of all the other coal-tar dyes used for this purpose, the temperature must be kept as low as possible. Dyes that are soluble in spirit may also be used in the state of a concentrated alcoholic solution, which is mixed with an alcoholic solution of stearine and left to set hard. A quantity of this preparation sufficient to impart the desired shade is then added to the melted ceresine.

Care should be taken that the dye-stuffs used do not impair the illuminating power of the candles any more than can be avoided, though this power is always affected, despite the very small quantity of dye used, black being the worst offender in this respect on account of the larger amount necessary to impart the colour.

Another point to be borne in mind in packing candles of different colours together is that some colouring matters—especially the Soudan dyes—are very liable to volatilise out of the material.

COLOURING CERESINE AND PARAFFIN CANDLES.

Candles and other articles made of ceresine and paraffin may either be coloured in bulk before being made up, by incorporating the dye-stuffs in the melted material, so that the whole is coloured right through; or else they may be made up in the uncoloured state and coated with a thin coloured film afterwards, by dipping the candles, etc., in melted coloured paraffin for a moment and taking them out of the bath again, thus leaving the central portion white. With regard to the choice of colours for this purpose, all those that are injurious to health must be banned, since these will decompose on contact with the flame, and are also injurious when used on other articles, such as toys. Vermilion, for example, if used for colouring candles, is decomposed by the heat of the flame and liberates mercury vapour; white lead will give off lead fumes; and arsenic compounds, which are occasionally used for colouring candles, will give off arsenical vapours. Apart from the coal-tar dyes already mentioned, it is difficult to find any safe colours for this purpose, since all the most poisonous pigments give the brightest and handsomest colours, but must be avoided for hygienic reasons. The harmless colouring matters include: turmeric, saffron, fustic and quercitron for yellow; alkanna root, madder, dragon's blood for red; indigocarmine for blue; indigocarmine and turmeric for green; alkanna root and indigocarmine for violet, etc. The following are decidedly injurious: vermilion, red lead, red chrome, chrome yellow, zinc yellow, orpiment, Schweinfurt green, verdigris, and in fact all mercury, copper, lead, and arsenic colours.

In the majority of cases the harmless colours named above will suffice, if suitably mixed, to give any desired shade. Thus, all shades of yellow up to deep orange can be produced by mixing alkanna root with turmeric. An addition of indigocarmine to turmeric produces a bright green, the tone being more yellowish or bluish in proportion as one or other ingredient preponderates.

Yellow.—Two parts by weight of ceresine or paraffin are boiled on the water bath with 0·150 part of turmeric powder for half an hour, and then strained through linen. Another recipe consists of 2 parts of ceresine or paraffin and 0·01 part of saffron, or 0·1 of quercitron.

Red—

2 parts of ceresine or paraffin,
0·2 ,, alkanna root or
0·1 ,, safflower, or
0.13 ,, ground dragon's blood.
Mixed as above.

Blue—

2	parts	by weight of	ceresine or paraffin and
0·1	,,	,,	indigocarmine.

Green—

3	,,	,,	ceresine or paraffin,
0·2	,,	,,	alkanna root,
0·05	,,	,,	indigocarmine.

Violet—

2	,,	,,	ceresine or paraffin,
0·1	,,	,,	alkanna root,
0·05	,,	,,	indigocarmine.

Brown.—Two parts by weight of ceresine or paraffin are melted and mixed with a solution of—

0·50	part of	Cassel brown and
0·20	,,	potash, in
1·80	,,	boiling water,

the whole being boiled until all the water has been expelled.

Black.—Three parts by weight of ceresine or paraffin are melted and incorporated with 0·05 part of finest lampblack by continued stirring.

For very cheap articles, use may be made of earth colours, such as ochres, umber, colcothar, terra di Siena, green earths, etc., and Frankfurt black, the finely powdered colours being added to the melted ceresine or paraffin and the whole stirred till the mass begins to set.

Graefe recommends the following dye-stuffs for colouring ceresine and paraffin candles:—

Black: nigrosine and typophor black. Blue: Victoria blue, typophor blue. Green: brilliant green, malachite green. Yellow: quinoline yellow, picric acid. Orange: diamond orange. Red and pink: erythrosine, rhodamine 6 G and rhodamine B. Violet: methyl violet, crystal violet.

CHAPTER III.

MINERAL (MONTAN) WAX.

THIS product, which has been brought on the market within the last few years, is obtained from Thuringian lignite by a process described elsewhere, and forms, in a refined state, a white mass of high melting-point, resembling wax and far superior to stearine or paraffin. Not much is known of the article, which, however, has been reported on by E. von Boyen, this authority stating that in his earlier researches into the products from lignite he paid special attention to the bitumen, which seemed from the investigations of Brückner and Wackenroder to consist of a whole series of different substances. Pyropissit being available in nearly all the lignite works in Thuringia, that substance was extracted with various solvents, and furnished a product which is now manufactured, at the Bölpke works in the province of Saxony, as the material from which mineral wax is produced at that establishment. This bitumen is an inodorous mass, as hard as ozokerite, and melting between 80 and 90·6° C. This variation in melting-point, which was frequently observed in the earlier experiments, is not, as then seemed probable, due to differences in the lignite from the several mines. Photogene or lignite benzine, as well as coal-tar oils, will extract a darker-coloured bitumen of higher melting-point and in larger quantity than benzine, ether, or amyl alcohol; but, on the other hand, this increase is counterbalanced by the diminished yield of mineral wax. Numerous attempts to refine the bitumen with various chemical reagents proved unsuccessful, and even the method recommended by Brückner failed to yield a pure substance. Repeated precipitation from solution and treatment with bone-black and other decolorising agents invariably furnished non-crystalline yellow or yellow-brown masses, devoid of all technical or even scientific value. Paler and yellow products were obtained with nitric acid and chlorine, but were contaminated with chlorine and nitro products, and proved unsuitable for candle-making owing to their smell and defective capillarity. Acid and alkaline solutions of potassium permanganate and potassium dichromate caused decomposition, without purifying the bitumen; and concentrated or fuming sulphuric acid charred the material, even when paraffin was added as a protection. An almost white mineral wax was obtained by treatment with more than 100 per cent. of fuming acid and decolorising powder and by extracting the black asphaltic reaction product with benzine; but the yield is too small and the cost of the treatment too heavy to make the operation profitable. The distillation of bitumen in a glass retort furnished von Boyen with a tallowy non-crystalline mass, melting at 55–60° C.; and repeated distillation yielded progressively more fusible products, until finally oil and a little paraffin were obtained. The matter reached a novel and more satisfactory stage when he was able to prepare larger quantities of bitumen and distil them with steam. It was then found that the bitumen could be transformed into a pale yellow, fairly dry, crystalline mass, with a melting-point of over 70° C., which mass was not decomposed into paraffins and oils by redistillation, but was rendered more crystalline. The bitumen distillate when treated with benzine, followed by repeated pressing and treatment with decolorising agents, yielded a white mass of crystalline character and melting above 70° C. This surprising result was utilised as the basis of a new manufacturing process, the hard white product not having been previously obtained by any of the previous methods of treating lignite bitumen. To maintain the validity of his patent, von Boyen had to combat numerous oppositions and demonstrate that his product was not paraffin, but consisted of a high-molecular acid and a compound readily decomposable by sulphuric acid, these two substances being easily separable and distinguishable.

Mineral wax contains these two bodies in nearly equal proportions. The one body ("montanic acid") has been examined by Prof. Karl Hell, who ascribes to it the formula $C_{29}H_{50}O_2$, and the melting-point 83–84° C. Its potassium and sodium salts are readily soluble in hot water, from which they are thrown down, by a large excess of water, in the gelatinous form. These salts are less readily soluble in alcohol, and crystallise out in needles. The acid itself is sparingly soluble in alcohol, and crystallises out well, though it dissolves more readily in hot alcohol, from which it recrystallises in the form of needles. It is charred by concentrated sulphuric acid. When cooled after melting, it exhibits a lustrous, radial texture, similar to that of Chinese wax.

The second constituent of mineral wax, which is probably an alcohol, was also investigated by Hell. It is separated from the less fusible acid by hot pressing with benzine or other analogous solvent, and is recovered by fractional crystallisation, the fraction crystallising at 61–65° C. being collected and treated with ordinary alcohol and caustic soda to bring the final traces of acid into solution. On the addition of water, the hydrocarbon separates out satisfactorily, solidifying to a transparent cake, which can then be refined in the same way and recrystallised several times from benzine. In presence of solvents it behaves similarly to montanic acid, and is soluble in benzine but less readily in alcohol. Strong sulphuric acid at a high temperature decomposes it more readily than montanic acid; and nitric acid, after prolonged boiling on the water bath, oxidises it into fusible fatty acids which have not yet been more closely examined. The formula, which has not been determined, should help to explain the composition of lignite bitumen. It may, however, now be assumed that this bitumen is the non-crystalline ester of montanic acid, and therefore a wax, which is decomposed by distillation with steam, the bitumen itself being non-crystalline, whereas its components are crystalline. Of course, the circumstance that montanic acid is obtainable direct by treatment with strong sulphuric acid is no proof that the acid exists in the free state in bitumen, since the reagent may, as in the case of most high-molecular fatty acid esters, effect a similar dissociation to that produced by superheated steam.

An important fact for the scientific investigator is that only the two components in question are obtained from the bitumen by distillation with steam. That mineral oils are also formed is immaterial, since these result from a secondary decomposition alone, and their formation can be reduced to a minimum by careful working. Moreover, this fact is unaffected by the appearance of fusible fatty acids in working on a practical scale, because von Boyen has shown that a fusible fatty acid is formed by the oxidation of mineral wax by nitric acid, which oxidation may also be produced in practice by the action of atmospheric oxygen.

The discovery of mineral wax has led to the establishment of a new branch of industry, apparently destined to become important, the origin of which is largely due to the foresight and activity of Ernst Schliemann.

VON BOYEN'S METHOD OF PRODUCING MINERAL WAX.

The usual method of distilling the highly bituminous lignites furnishes lignite tar, which serves for the production of paraffin and mineral oils. The tar is obtained by distilling the lignite in suitable furnaces, the tar being condensed from the distillate. This tar consists chiefly of liquid and solid hydrocarbons—namely, oils and paraffin—and is yellow-brown or black in colour, melting at about 33° C. and giving off a characteristic smell due to the presence of aromatic acids and basic substances. The tar is repeatedly distilled and is thereby decomposed into several mineral oil fractions, which are refined with concentrated sulphuric acid and caustic soda. Paraffin crystallises out from the highest fractions and is purified and decolorised by repeated pressings with oils of the benzine class and treatment with decolorising powders, the total yield obtained being about 13 per cent. of the tar. The mineral oil fractions and the oils from the paraffin press are separated according to their specific gravity and colour, the lightest being used as benzine, the middle fractions as gas oils, and the heaviest as lubricants. The total mineral oil is about 70 per cent. of the tar, whilst about 8 per cent. is made up of phenols and pyridin bodies, separated by the acid and alkali treatment and recovered by distillation. The rest consists of gases and retort coke. In this method of treating lignite tar the paraffin forms the most valuable product although the smallest in quantity, and apart from the fact that paraffin is now offered cheaply and in large amount. The bulk of the tar consists of distillates containing barely ½ per cent. of paraffin, and hence the usual method of distilling has become unprofitable. The method now to be described enables a large quantity of a new product, mineral wax, to be recovered from lignite, 7–10 per cent. of bitumen being obtained by extraction or steam distillation. This bitumen in turn furnishes about 30 per cent. of mineral wax, which should sell at a higher price than paraffin on account of its hardness and high melting-point. Mineral oil is obtained merely as a by-product, to the extent of about 10 per cent. of the bitumen, the residual 40 per cent. being made up of waste gases and retort coke.

The bitumen for the production of mineral wax may be obtained either by steam distillation or extraction. In the distillation method, lignite fresh from the pit is distilled in cylindrical retorts until charred, by the aid of steam superheated to 250° C. and by a slow fire. The distillate, which differs from the ordinary lignite distillate by its higher melting-point, viz. above 70° C., forms the raw material for mineral wax. In the extraction method dried lignite is treated with benzine, solar oil, or analogous solvents, and furnishes a brown to black extract, which is lustrous, hard and brittle, inodorous, and melts at 85° C.

The bitumen obtained by either of the foregoing methods is melted in a retort and heated to above 100° C. By repeated distillation with superheated steam at 230° C., the bitumen is converted into a pale yellow crystalline substance with a high melting-point—namely, mineral wax—which can be refined by dissolving it in solvents and afterwards treating it with decolorising agents. Refined mineral wax is a hard white substance, melting at 70° C. or higher, and differing from paraffin in that it can be readily saponified with alkalis and completely charred by concentrated sulphuric acid. It consists of two well-defined substances, an acid and an unsaturated hydrocarbon, which can be separated by saponification with alkali in alcoholic solution, the acid forming a potash salt that is soluble in water and crystallises from alcohol, whilst the hydrocarbon, which is insoluble in water and alcohol, is deposited. The montanic acid, obtained by precipitation with sulphuric or other mineral acid, melts at 80° C. after repeated recrystallisation from organic solvents, is soluble in benzine, benzol, glacial acetic acid, ether, and alcohol. When melted and recooled, it solidifies in radial crystals, and has the specific gravity 1·013. The potassium and sodium salts are readily soluble in water, from which they separate out again, on dilution, as a gelatinous mass. They are sparingly soluble in alcohol, and recrystallise therefrom in the form of matted needles. The other component of mineral wax is an unsaturated hydrocarbon, readily soluble in benzine and benzol, but only sparingly so in glacial acetic acid, alcohol, and ether. It crystallises from benzine as lustrous white scales, and melts at 60° C. It differs from paraffin in being completely charred by concentrated sulphuric acid. Both components form useful candle-making material.

PROCESS OF THE MONTANWACHSFABRIK FOR THE PRODUCTION OF MINERAL WAX.

The process described in German Patent 101,373 for recovering mineral wax (a substance composed of an acid and an unsaturated hydrocarbon) from lignite bitumen, consists in treating the bitumen with highly superheated steam, followed by repeated distillation with steam. The chief value of the resulting product resides in its content of montanic acid of high melting-point. However, the process as set forth in the above patent does not recover the maximum quantity of this valuable constituent from the raw material, the action of the superheated steam at atmospheric pressure resulting in partial decomposition of the montanic acid into mineral oils and a substance similar to paraffin, thus reducing the yield of the final product to a corresponding extent. At the same time, the melting-point of the mineral wax is considerably lower than would be the case if it contained a larger proportion of montanic acid. To obviate this defect, use is made of steam distillation under reduced pressure—a method largely adopted in the chemical industry for the recovery and refining of readily decomposable substances—experiments in this direction having proved satisfactory. Experience has shown that mineral wax can be distilled over without decomposition under a pressure of 10 mm. mercury gauge; and accordingly, the bitumen obtained from lignite in the manner already described is distilled with superheated steam in a vacuum plant. The product differs from that obtained by the original process in containing a larger proportion of montanic acid and in being much paler in colour, so that its commercial value is higher. The superheated steam may be replaced by inert gases, such as hydrogen, carbon dioxide, etc., and in this way the method may be extended to cases in which one or other of these gases is obtained under favourable conditions—for example, as a by-product.

MEYER'S PROCESS FOR RECOVERING WAX-LIKE PRODUCTS FROM LIGNITE.

This method relates to the manufacture of bitumen and briquettes from Thuringian lignites containing waxy products of high melting-point and greater value than the paraffins and oils usually recovered from lignite tar. This bitumen is soluble in benzine as soon as the water in the lignite has been expelled (German Patent 99,566). To save the expense of drying the lignite (which may contain up to 50 per cent. of water) by artificial means, use is made of a mixture of alcohol and benzine, the alcohol absorbing the water, whilst the benzine dissolves the bitumen, etc., so that each acts as an auxiliary of the other. The advantage of this treatment is that the alcohol and benzine can be recovered from the extracted product at less expense than would be involved in drying the lignite, etc. Still greater economy can be effected by first treating the lignite with alcohol alone, the alcohol-benzine mixture being added only when the density of the first alcohol has fallen to about 69° Tralles; or the alcohol treatment may be continued and the benzine added afterward, so that the mixture is formed within the mass of lignite itself and extracts the bitumen therefrom. The first runnings of extract contain only a small quantity of bitumen, in the form of flakes, owing to the predominance at this stage of dilute alcohol, which precipitates the bitumen; but the subsequent portions of the extract are richer in bitumen. It is advisable to keep the two extracts separate, since the richer portion is free from water and gives a higher yield of tar in the still, whereas the first runnings deposit the contained bitumen in the form of a flocculent scum, very soon after the expulsion of the benzine or alcohol, which flakes can be easily recovered by mechanical means when the vapours of the solvents have been driven off by distillation. The still retains all the water originally present in the lignite, this water having been condensed in the columns and returned to the still, leaving the alcohol and benzine in a more concentrated form for recovery separately. The bitumen is converted into refined products by the methods used in the preparation of substances allied to paraffin and ozokerite. In choosing the form of extractor to be used, preference should be given to one in which the spent lignite can be heated, to distil off the adhering traces of solvent, and from which the warm lignite can be cleaned out by mechanical means for the purpose of converting this residual matter into briquettes, which can be produced therefrom cheaply and of good, firm quality, free from water, by the application of only a moderate amount of heat. The fuel thus obtained is of greater value than the retort coke, the calorific power and binding quality being still further increased by the addition of one of the viscous waste products of the tar or petroleum industry, either in a heated condition or dissolved in benzine. If added to the lignite, these pitch-like adjuncts are distributed throughout the mass during the expulsion of the benzine-alcohol mixture, and improve the value of the briquettes. The small quantities of benzine and alcohol lost in the recovery of these solvents is insignificant in comparison with the higher value of the bitumen and fuel residue obtained by the alcohol-benzine extraction process.

INFUSORIAL WAX (DIATOM WAX).

This name is applied to a wax-like product obtained by extracting diatoms with toluol and driving off the sulphur contained in the mass. These diatoms, which are the siliceous envelopes of microscopic animals, are sometimes found in large masses, and have latterly been regarded (as a result of the Engler-Höfer theory) as having played some part in the formation of certain deposits of petroleum. A discussion of this theory of the formation of petroleum is beyond the scope of the present work, but mention must be made of the diatom wax described by Krämer and Spilker, because this product may become of considerable importance if a successful method of extracting it should be discovered.

Krämer and Spilker obtained about an ounce of diatom wax from 1½ lb. of dry peat from the Franzensbad peat moss, and it is calculated that something like 100,000 tons of this wax are contained in the diatomaceous deposit in the drained lake at Ludwigshof in the Uckermark district. This deposit is covered by about a foot of peat and consists of a stratum of lake mud averaging 23 feet in thickness. On account of its richness in nitrogen (3 per cent.) this mud is largely used as a fertiliser, and for the same reason might be utilised for the recovery of ammonia on a large scale.

According to Krämer, the mass, which covers an area of over 2000 acres, consists of 80–90 per cent. of water and ammonia, with a number of various bacillariaceæ, desmitiaceæ, and fragments of higher plants. When dried on clay slabs, the mass yields 50 per cent. of ash, 70 per cent. of which is composed of silica, together with alumina, ferric oxide, and 7·6 per cent. of calcium. The dried mass of bacillariaceæ yields, on extraction with benzol, a yellow to dark brown wax, which can be separated into a yellowish white wax, melting at 70° C., and a greasy paraffin-like mass, by recrystallisation from alcohol. Treated with hydrochloric acid, the dried lake mud yields 50 per cent. of soluble matter (including a large proportion of reduced carbohydrates and organic acids). The insoluble portion when extracted with toluol yields 3·6 per cent. of wax containing about 1 per cent. of sulphur, 2·42 per cent. of ash, and considerable quantities of oxygen (and therefore probably contains esters). The carbon content is 73·5 per cent. and that of hydrogen 11·2 per cent. When heated to 250° C., 20 grms. of wax from the Franzensbad moss (which, in contrast to Ludwigshof mud, contains large quantities of free hydrogen) furnished 1 litre of gas, containing about 10 per cent. of sulphuretted hydrogen, in addition to carbon dioxide and carbon monoxide. On continuing distillation to 300° C., 1·6 grm. of water and 11 grms. of oil were obtained, from which latter a crystalline substance ($C = 84$, $H = 14{\cdot}3$ per cent.), melting between 51 and 60° C., was obtained by recrystallisation from alcohol.

Natural Galician ozokerites, examined for comparison, all exhibited the same chemical behaviour, the whole of them—whether of high or low melting-point—furnishing up to 80 per cent. of Beilstein's lekene (m. p. 79° C.). The so-called marble wax alone furnishes a substance (m. p. 98° C.) containing fluctuating proportions of oxygen compounds of a resinous character, which are saponifiable and are decomposed by concentrated sulphuric acid. The sulphur content of the melted ozokerite amounted to 0·15 per cent. No water was driven over by distillation; and the gas formed was almost entirely free from carbon monoxide and carbon dioxide, but contained small amounts of sulphuretted hydrogen. Hence, in contrast to diatom wax, ozokerite does not contain any appreciable quantity of oxygen; and, conformably with this discovery, only small quantities of substances saponifiable by alcoholic caustic potash are furnished by ozokerite, whereas diatom wax yields up to 90 per cent.

When diatom wax is distilled under pressure in fused glass tubes, bent at an obtuse angle, the products, in addition to gases (carbon monoxide, carbon dioxide, and sulphuretted hydrogen), contain liquid hydrocarbons almost entirely devoid of solid paraffin, which hydrocarbons, apart from their content of unsaturated compounds, exhibit the characteristics of petroleum. The distillates always contain small drops of water, in contrast to the distillates obtained from ozokerite under pressure. The latter also failed to yield the same gases, except for a small quantity of sulphuretted hydrogen. The fractions of the first light pressure-distillates from ozokerite had the specific gravity 0·69 (b. p. 70° C.) to 0·72 (b. p. 290° C.). Like the corresponding fraction from diatom wax, the third fraction, boiling up to 290° C., decolorised only 3·6 per cent. of bromine, and when treated with concentrated fuming sulphuric acid, gave a product of the same composition as petroleum from the Tegernsee. Various ozokerites, on the other hand, gave light distillates containing 50–60 per cent. of petroleum fractions, 13–17 per cent. of unsaturated hydrocarbons being eliminated by treatment with fuming concentrated sulphuric acid. The refined product contained 84·4 per cent. of carbon and 15·4 per cent. of hydrogen, or practically the same as Tegernsee petroleum.

Carnauba wax and Japan wax behaved in an analogous manner to diatom wax, the resulting oils having the same composition as that just given. Krämer and Spilker believe that diatom wax has been formed from ozokerite (to which it is allied by its content of saponifiable matter and sulphur), probably by the action of ammonium carbonate; but they did not succeed in adducing any experimental proof in support of this view, so they rightly classify diatom wax with animal and vegetable waxes for the time being.

In their opinion, petroleum was formed from ozokerite or diatom wax. The cover rock of the Galician ozokerite deposits has many resemblances in point of composition with the siliceous scales of diatom mud, containing as it does 49–62 per cent. of silica, 20 per cent. of alumina, 8 per cent. of lime, and 6 per cent. of carbon dioxide. The boring detritus in the Tegernsee wells contains 15–33 per cent. of silica and 6–17 per cent. of alumina, according to the depth. The silica diminishes as the depth increases, but the inorganic constituents of the lake mud are still found in the lowest strata of the cover rock, though entire diatom shells are not found there, having probably been destroyed by the prolonged heavy pressure. The action of water and the ammonium carbonate originating in the vegetable material may also have had a destructive influence during the immense geological periods in question. The authors consider that most of the widespread petroleums rich in paraffin (Pennsylvanian, Galician, etc.) were formed at low temperatures and under little or no pressure. Heavier pressure, perhaps accompanied by overheating, caused the formation of the oils low in paraffin (Baku, Ohio), the sulphur invariably present forming volatile sulphur compounds. The third class of petroleums (Roumania, Elsass), which is characterised by a high asphaltum content, would be formed in consequence of the intensified action of the sulphur in conjunction with that of atmospheric oxygen. Owing to the fact that distillates formed under pressure always contain large quantities of substances capable of decolorising bromine, which bodies are of insignificant occurrence in natural petroleums, the authors, sharing Engler's views (supported by experimental data) on the autopolymerisation of petroleum, hold that the petroleums formed under pressure gradually became polymerised with formation of viscous constituents of high boiling-point. They also succeeded in producing a pure hydrocarbon oil (89 per cent. carbon and 10·9 per cent. hydrogen) by distilling colophony under pressure, which highly viscous product represents (theoretically) 1 molecule of abietic acid deprived of 1 molecule of carbon dioxide and 1 molecule of water. The principal geological factor on which the authors found their theory of the origin of petroleum is the extensive deposits of diatoms and allied organisms found in so many parts of the world. Even if we subscribe to the Engler-Höfer theory of the collaboration of the corpses of marine animals (fishes, mollusca, etc.) in the formation of certain deposits of petroleum, it is difficult to imagine the accumulation of such gigantic masses of animal fat as would be required to form the petroleum found in Galicia, Caucasia, and the Alleghany Mountains. A point favouring the accumulation of diatom remains is the extremely rapid reproduction exhibited by these organisms. The Ludwigshof Lake alone contains 63 million tons of lake mud, with 6·3 million tons of dry matter, containing 3·6 per cent. of wax. The authors also refer to Stahl's doubts on the applicability of the Engler-Höfer theory to the petroleum deposits in the Kalmuck and Kirghis steppes, his idea being that the oil there was formed, not from animal fatty remains, but from the decomposition products of other forms of life (diatoms, etc.), though at the time he apparently did not know of the existence of diatom wax.

MOFETTI WAX.

This name was given by Töllner of Bremen to a cheap substitute for beeswax, ceresine, and similar products. Though no particulars as to its origin have been given, it is probably an artificial product closely allied to paraffin or mineral wax. According to Töllner, it is perfectly suitable for most of the purposes for which waxes are used, but was shut out from extensive employment owing to the smallness of the output, which, however, is to be increased.

Mofetti wax is a white or yellowish wax-like substance, which has a greasy feel, though it does not leave a fatty mark. It is insoluble in water and alcohol, but soluble in ether, amyalcohol, benzol, petroleum spirit, chloroform, carbon disulphide, and fatty and volatile oils. It can be melted to a homogeneous mass with wax, tallow, resin, etc. When melted and mixed with oil of turpentine (or turpentine substitutes) and hydrocarbons, it makes a cheap and good polish for flooring, etc., that is easy to work and gives a very high and durable gloss; and when incorporated with black coal-tar dyes (soluble in fat) and lampblack, it furnishes a good polish for preserving leather. A good, cheap vaseline, perfectly homogeneous and resembling American vaseline in appearance, is obtained by mixing the melted wax with liquid paraffin. Mofetti wax is rather hard for some purposes, *e.g.* waxing twine, but it can be softened and made more adhesive by the addition of 10 per cent. of wool fat. As a candle material it burns with a bright, quiet flame. No generally applicable instructions can be given for the application of mofetti wax, but it is so simple to manipulate that there should be no difficulty in drawing them up.

CHAPTER IV.

VARIOUS APPLIANCES FOR EXTRACTING, DISTILLING, AND REFINING OZOKERITE, ETC.

EXTRACTION APPARATUS.

IT would extend the scope of the present work too far to describe all the known forms of extraction apparatus, and this is the less necessary because they are all based on the one principle—namely, the use of a volatile solvent to obtain the extracted matter in the purest possible condition, and then expel the solvent and condense it for use over again with a minimum amount of loss. In large works a number of these apparatus are united to form a battery working continuously.

The Lindner and Merz Universal Extractor (Fig. 15).—The vessel M, fitted at the bottom with a steam coil *f*, surrounds a receptacle L, which is charged with material through the manhole *d*. The solvent is run in from the combined storage tank V and condensing apparatus K, the extract solution being drawn off through the syphon *ggg* to M as soon as it reaches the level of *g*. The solution is evaporated in M, the vapours ascending and heating the walls of the receptacle L, and reaching the reflux condenser N, where they are condensed, the still warm liquid falling into L and again overflowing into M when the level of *g* is attained. This automatic circulation is not interrupted until a sample, drawn off through *h*, indicates that the extraction is complete. The supply of cooling water to N is then cut off, and the vapours ascending from L pass through M to the condenser K, whence they are returned into the tank V, the final portions of the solvent being expelled from the mass and the residue by a current of steam. The extract is drawn off through *n*, and the extractor is emptied at *l*. The apparatus can also be arranged to work continuously, by adjusting the flow of the solution so that the liquid is maintained at a constant level in *h*. The advantages of this apparatus include: (1) Rapid and complete evacuation of the charge; the constant renewal of the solvent and the fact that it acts at boiling temperature ensure complete extraction, whilst the continuous circulation and simultaneous supply of heat accelerate the operation so that it is finished in 3–4 hours. (2) Perfect security from fire and explosion. The special

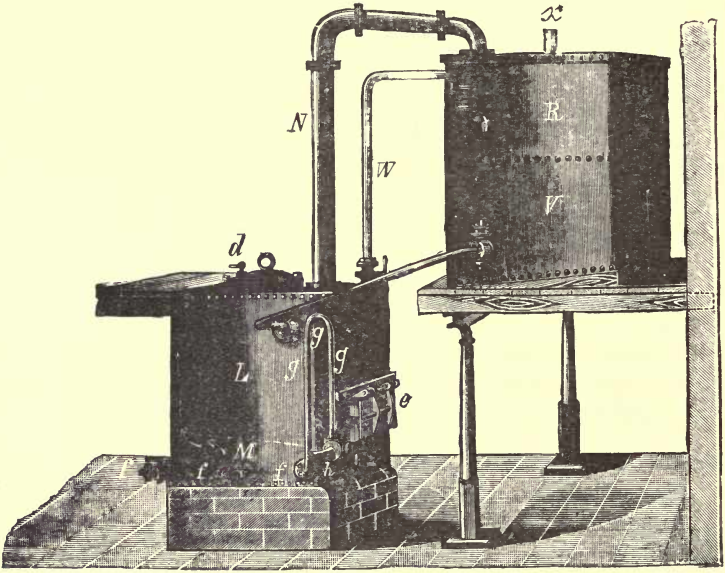


FIG. 15.—Lindner and Merz Universal Extractor.

disposition of the apparatus prevents expansion, though the solvent is at boiling temperature, the danger arising from such readily inflammable vapours under pressure being entirely avoided. In view of this safety, the apparatus is sanctioned by the authorities in Germany for use without the precautions usually insisted upon. (3) Minimum consumption of solvent. The absence of expansion, owing to the effectual condensation, ensures almost entire freedom from loss, practically the whole of the solvent being recovered at the close of the extraction. Neither the extracted material nor the residue has any smell of the solvent. (4) Simplicity and economy of working. The apparatus works automatically, and when it is charged and the steam and water taps are turned on, requires no further attention. The utilisation of the heat from the vapours of the solvent ensures economy, and the work of charging and emptying the apparatus is easy. (5) Dryness. The residue is quite dry when it leaves the extractor, the method adopted precluding any wetting of the charge, with the attendant losses of constituents soluble in or alterable by water. (6) No inconvenience caused to the surrounding neighbourhood. The apparatus being completely enclosed, no disagreeable smells are given off, and the effluent condensing water is perfectly clean. (7) No auxiliary appliances, such as air-pumps, vacuum-superheaters, etc., are required, the apparatus being complete in itself and ready for use as soon as it has been connected with the steam and water supply. It is therefore very compact and takes up little room.

Seifert's Extraction Battery.—This apparatus consists of three, six, or eight cylinders, C^1, C^2, C^3, etc., which are steam-jacketed. Each cylinder also contains a wire-gauze cage for holding the material to be extracted. As soon as the apparatus is charged, the extraction is commenced by allowing the solvent—benzine, benzol, carbon tetrachloride, etc.—to run in from a high-level tank through S and a to C^2. When this vessel is full, the liquid flows into C^3, through a^2, b^2, c^2, and thereafter through a^3, b^3, c^3, into C^n. The benzine or other solvent, saturated with extracted material, is led through d^n and p into a storage vessel, the flow being accelerated by the action of a suction pump. When the storage vessel is charged with a quantity of liquid equal to that in C^n, the completion of the extraction in any of the vessels composing the battery is indicated by the colourless appearance of the solvent passing through a gauge glass provided in the draw-off pipe. The vessel in question is then disconnected, and the solvent allowed to flow through the others by means of a two-way tap. To recover the solvent with which the residue is saturated, the liquid is expelled by compressed air, followed by admitting steam through the pipe D into the jacket space. The benzine vapour thus generated is passed away to a condenser coil. Finally, the cage containing the spent charge is taken out of the vessel and replaced by a fresh one. By this time the contents of the second vessel will be completely extracted, so that this is ready for treatment in the way thus described, so that the process goes on continuously. When the work is performed on a large scale, a still will be required, of sufficient size

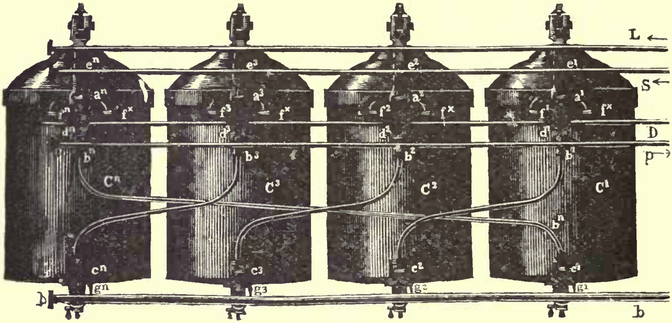


FIG. 16.—Seifert's Extraction Battery.

to take all the extract furnished by the extractors in a given time, to enable the solvent to be recovered and used over again as quickly as possible. This combination of extractors and still is important, as enabling the quantity of solvent required to be reduced to a minimum; and only the slight loss occasioned by leaky connections will have to be made good. The solvent is recovered by distillation in a steam still A (Fig. 17), jacketed in the lower part for the admission of high-pressure steam through the pipe D into the space B and through the coil S.

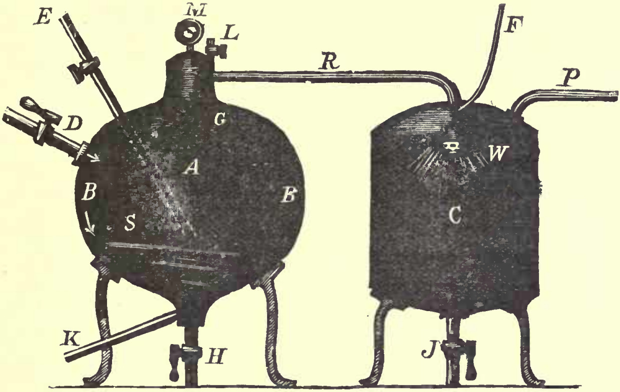


FIG. 17.—Benzine Still.

The water of condensation runs away through the pipe R. The dissolved extract is run from a high-level tank through the pipe E into the still, which is fitted with stirrers to facilitate the uniform ebullition of the liquid. To prevent a greater liberation of vapour from the boiling liquid than can be properly treated in the condenser, the flow of steam is regulated so that no smell of the solvent is apparent in the still-room, since otherwise there may be a considerable waste of solvent, as well as risk of explosion. When distillation is ended, the tap of a finely perforated pipe in A is opened, and a powerful current of air is forced through by a pump, to expel the final traces of solvent which adhere tenaciously to the product and are difficult to eliminate even by prolonged heating, though they are driven off by air in a few minutes. It is advisable to place the mouth of the pipe R in a cylinder C, connected with the condenser worm by a pipe P, for the purpose of introducing a very fine spray of water through the pipe F and sprinkler W into C. By this means the bulk of the benzine vapours are condensed in C, the mixture of benzine and water being run off through J into a separator. This device greatly assists in minimising the loss of solvent.

FILTER PRESSES.

The filter presses largely used in many branches of industry serve to free liquids or liquefied solids from finely divided solid matter in suspension, and enable the former to be recovered in a perfectly clear and pure state. In this case they act as true filters, and may be used for even the thickest and most impure liquids, the object being the recovery of a clear liquid. Filter presses are also largely used for getting rid of the liquid matter in a pappy mixture and recovering the solids therefrom—*e.g.* paraffin from paraffin-bearing oils. In certain cases filter presses may also be employed as washers.

Filter presses are of many forms, and consist of a variable number of conveniently shaped filter chambers with solid partition walls, the whole being mounted between a fixed and a movable press head. Filter cloths, which must be as large as the plates, since they have to tighten the outer edges as well, are suspended between the chambers. The entire system is united to form a solid whole by means of levers, screws, or hydraulic pressure, thus constituting a series of adjacent hollow spaces, with communicating feed-openings, and into which the liquid to be filtered or pressed is forced by a pump or by its own weight. By this means the liquid portion is separated from the solids by traversing the filter cloths and running away clear, through suitable passages, into a collector for removal, whilst the solids remain behind in the press.

Filter presses are classed into chamber presses and frame presses, according as the feed-passages are arranged in the middle or at the side, and as the space for the pressed cakes is formed by the projecting edges of each adjoining pair of plates (the cakes falling out when the press is opened) or by the frames interposed between each pair of plates (so that the cakes are taken out with the frames).

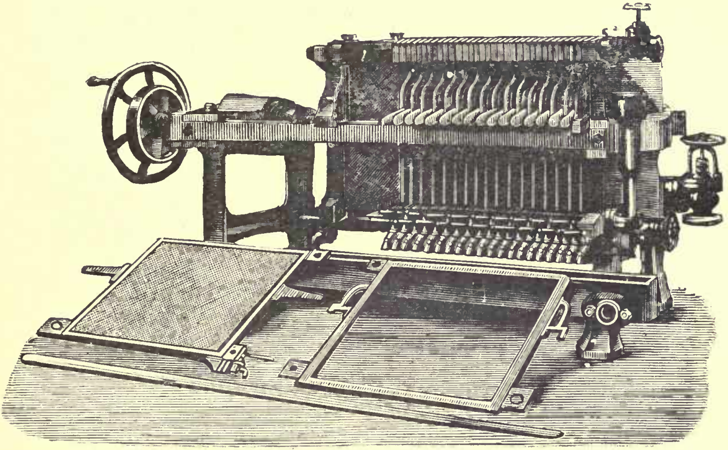


FIG. 18.—Filter Press.

Chamber presses offer the advantage of greater strength, and consequently increased capacity. In these presses there are in every case two cloths in contact, so that a tight joint is made; and the large central feed-openings prevent all risk of obstruction. The drawback consists in the loss of time involved in fitting and fixing the filter cloths in position, even when the cloths have been cut out to correspond with the openings. The cakes made in these presses have a central opening and are not solid right across. Frame presses have the advantage of requiring a smaller quantity of filter cloths, only one cloth being needed for each joint; besides which, the cloths do not require stretching, and solid cakes are obtained. On the other hand, they are more likely to choke up, owing to the narrowness of the passages, so that some of the chambers may remain empty, and the frames have to be carefully scraped clean each time the press is used, to make sure of getting a tight joint.

Another type is the three-chamber press, which differs from the foregoing in having no cloths at all, sand, carbon, etc., forming the filtering medium. These presses furnish a particularly limpid and pure filtrate. A plate is mounted between each pair of frames, and plates are also inserted on the other sides of the frames, for leading away the liquid set free in making the filter ready. The filtering medium (sand, carbon, etc.) is introduced in the form of pulp and forms a solid mass, whilst the liquid drains away through the channels in the two adjoining plates. When the filter has been got ready in this way, some of the plates are taken out and replaced by frames, the rest of the plates and frames being left in position. Where the apparatus is intended for pressing rather than as a filter, the presses are fitted with lixiviating devices suitable to the class of material under treatment.

BONE-BLACK FILTERS AND THE LIKE.

In this form of filtering apparatus, the product to be refined is run through bone-black or other decolorising agent, which bleaches it at the same time. The filter consists of a certain number of upright cylinders each of which is fitted with one manhole at the top and another at one side. A sheet of iron is placed inside between the two manholes of each cylinder, thus forming a space for the filtering medium (bone-black, residue from the ferrocyanide process, aluminium-magnesium hydrosilicate, etc.). Pipe connections are provided on the two manholes and fitted with vertical communication pipes. These latter pipes are connected by valves with four horizontal pipes near the top of the apparatus, so that each horizontal pipe can be connected with any of the vertical pipes. Below the intersection of these pipes is, in each case, a valve and a pipe connection attached to each overflow pipe and forming the commencement of the effluent pipe. From each manhole rises an air pipe, and on each overflow pipe is a draw-off tap (not shown in the drawing). The liquid to be filtered is introduced through the pipe *s* (after the valve *o* has been closed), and through the upper part of the pipe *u*, into the top manhole, running down thence through the bone-black in the cylinder and rising in the upcast pipe *n*. When the valve *o* is open, the liquid enters the manhole in the second cylinder, traverses the filtering medium therein and passes afterwards through the next cylinder in the same way, to escape finally through the effluent pipe. The filtering medium is introduced and discharged through the manholes. When thick heavy oils or solid substances like paraffin are to be

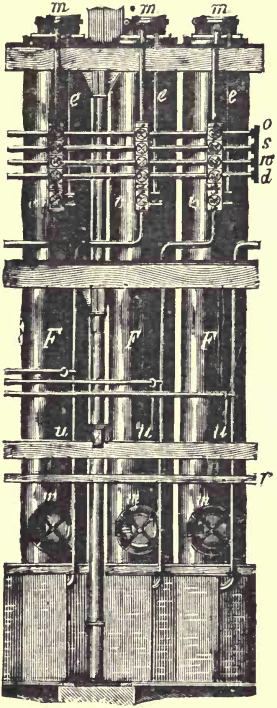


Fig. 19.—Bone-black Filter.

filtered, the apparatus must be steam-jacketed in order to keep the material as fluid as possible during filtration.

CENTRIFUGAL SEPARATORS.

The soft crude paraffin, more or less charged with crystalline sediment, may be treated in a centrifugal separator, in order to separate the solid paraffin from the liquid oil. Centrifugal separators have become an indispensable device in all chemico-technical industries, being able to separate liquids of different density as well as solids from liquids, and also to mix liquids together, besides serving a variety of other purposes. They are driven by steam or electricity, through intermediate gear or by special motors built for this purpose, and having to be run at very high speeds must be very strongly constructed. These reasons are sufficient to justify the requirement laid down by the German Industrial Assurance Association for the Chemical Industry, to the effect that all centrifugal separators must carry a tablet clearly inscribed with the maximum permissible load and speed, and those driven by a special motor must be fitted with a tacheometer marked with the maximum permitted speed.

The separator may be driven from overhead through a second motion mounted on the standards of the machine, or from below by some method of transmitting motion to the vertical shaft, the latter system being now almost universally adopted, top driving entailing even greater care in supervision and strength of construction than bottom driving. In addition, a stronger foundation is necessary, and there is greater risk of the contents of the separator being contaminated with lubricating oil, whilst access to the drum is hindered by the projecting driving shaft. On the other hand, bottom-driven machines take up more floor space. C. H. Haubold, junr., who has had a long experience in the construction of bottom-driven separators, puts forward the following claims for the superiority of this type: (1) Perfectly quiet and noiseless running, with low cost of upkeep; (2) non-necessity for a brick-work foundation, this being replaced by a strong wooden frame enabling the machine to be set up anywhere, even in top floors; (3) reduced consumption of motive power, and pan always accessible; (4) driving gear underneath the pan, thus preventing contamination of the contents by lubricating oil; (5) careful, strong construction and good finish of the various parts, with a minimum of wear and tear; (6) high speed with perfect safety. The pans are constructed and arranged in such a manner that uniform separation is effected. They are mostly made of sheet-copper, though for special purposes steel, wrought iron, cast iron, porcelain, aluminium, brass, bronze, etc., may be used, and the pans may be lined with vulcanite, enamel, etc., or else tinned, lead-lined, silver-

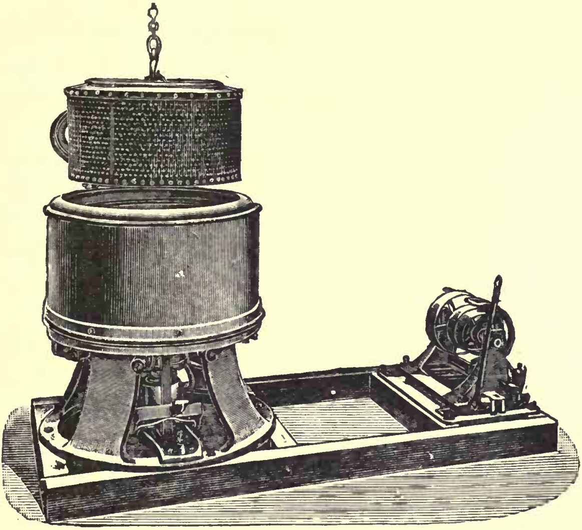


FIG. 20.—Centrifugal Separator with Removable Pan.

plated, etc. Vulcanite linings are recommended when the materials under treatment must not come in contact with metal or would corrode the latter; and this form of lining is superior to porcelain or earthenware on account of its relative lightness, so that the pan can be run at the highest permissible speed. It is also superior to lead lining, since the latter must be at least $\frac{1}{8}$th inch thick, which makes the pan too heavy to run at anything beyond a moderate speed.

All bottom-driven centrifugal separators can be fitted with removable pans, which can be lifted out by overhead tackle, and tilted, for emptying, in any convenient spot. The advantage of this form over the older type of pan taken out sideways consists in the greater ease and quickness of the operation without complicated mechanism and the necessity for taking the outer casing apart. The work is also accelerated by employing a duplicate pan, which can be kept ready charged and put into the machine whilst the first pan is being emptied and refilled. The cover is firmly attached to the pan, to prevent waste of the contents during the process. The firm already mentioned makes a special form of centrifugal separator in which the contents of the pan are discharged through four outlets and a shute attached to the casing of the machine.

CHAPTER V.

USES OF CERESINE, PARAFFIN, AND MINERAL WAX.

As stated in the Introduction, ceresine, paraffin, and mineral wax are chiefly used in making articles which would otherwise require to be made of animal or vegetable waxes. All three materials are soluble in the same solvents as wax, but are not saponifiable (if we except mineral wax), and in this respect are inferior to true waxes. It is, however, possible to render ceresine and paraffin emulsifiable in alkaline solutions—a point of special importance. The industrial application of ceresine, paraffin, and mineral wax is making continued progress, and new products are constantly being introduced and making their way into favour. The ease of manipulation, the possibility of incorporation with resins, fats, and oils by simple melting, and of staining to any desired colour, together with the power of resisting acids and alkalis, all contribute to the extended application of these substances to industrial and technical purposes.

PREPARATION OF ARTIFICIAL WAX FROM CERESINE, PARAFFIN, ETC.

The term artificial wax is applied to mixtures of paraffin, ceresine, and more or less hard, natural vegetable or animal waxes, cheapened by additions of tallow, colophony, and other resins. It is evident that the proportions of the mixture may be varied considerably according as a harder or softer, cheaper or dearer product is desired; and the mixing may be varied to suit the purpose in view. Other varieties of artificial wax are prepared from the same ingredients with the addition of ketones.

LEWY'S ARTIFICIAL WAX.

The preparation consists of a ketone of the acetone series mixed with an excess of paraffin, ceresine, and stearine.

DELAHAYE'S ARTIFICIAL WAX.

A substitute for beeswax for various purposes, especially encaustic painting, is prepared by melting paraffin and ordinary resin together slowly and adding water-white petroleum together with a suitable colouring matter dissolved or suspended in alcohol, oil, oil of turpentine, or ketone. The above materials may be replaced by stearine and pitches or resins generally, as well as by benzol, oil of turpentine, etc.

Artificial Wax (French Patent 347,493).

1.	Paraffin	35 parts by weight.
	Japan wax	25 ,,
	Carnauba wax	15 ,,
	Colophony	10 ,,
	White pitch (resin)	10 ,,
	Tallow	5 ,,
	Yellow dye (soluble in oil)	0·03 ,,
	Wax perfume	0·01 ,,

The proportions may be varied according to the desired appearance, purpose, etc., some being omitted or replaced by an equal quantity of others.

The following recipes are recommended for special purposes:—

2.	Paraffin	35	parts by weight.
	Japan wax	25	„
	Carnauba wax	15	„
	Colophony	20	„
	Tallow	5	„
3.	Paraffin	35	„
	Japan wax	25	„
	Carnauba wax	15	„
	White pitch	20	„
	Tallow	5	„
4.	Paraffin	55	„
	Japan wax	25	„
	Carnauba wax	25	„
	Tallow	5	„

Colouring and perfume as under 1. The waxes, colophony, pitch, tallow, etc., are melted together, coloured, taken off the fire, left to stand till nearly set, then adding the paraffin and pouring into moulds.

Carnauba Wax Substitute.

	Unsaponifiable.	Saponifiable.
Paraffin	25 parts by weight.	nil.
Ceresine	4 „	nil.
Colophony	6 „	3·5 parts by weight.
Carnauba wax	8 „	4·5 „
Mineral wax	8 „	15 „
Yellow beeswax	... „	7·5 „

Cyprien, Sabaton and Moreau's Artificial Wax.

Solid dry paraffin	100	parts by weight.
Colophony	50	„
Coloured tallow	5	„

These ingredients are melted together, clear pale colophony being necessary to give a limpid yellow product. One part of carnauba wax will make the mass firmer, and a pale yellow colour is obtained by adding gamboge. This artificial wax is suitable for technical purposes, and also makes a good floor polish.

Many artificial waxes consist merely of ceresine adulterated with colophony ; *e.g.*—

	(1)	(2)
Ceresine . .	75 parts by weight.	50 parts by weight.
Colophony . .	25 ,,	50 ,,

FLOOR-POLISH WAX PASTES.

Ceresine and paraffin, with or without additions of other waxes, have been found specially adapted for the preparation of floor polishes which shine well when simply rubbed with a woollen rag, preserve the wood, and will also stand washing. The method of preparation is very simple, the ingredients being melted together, and the solvent—oil of turpentine, turpentine substitute, benzine, etc.—stirred in, the whole being then left to set hard.

	1.	2.	3.	4.
Paraffin	15	...	10	10
Ceresine . . .	10	20	10	15
Oil of turpentine . .	45	35	45	45
Spermaceti . . .	...	...	5	...
Colophony . . .	...	...	...	5

In all cases, half the oil of turpentine may be replaced by heavy benzine.

5.		
5. Vaseline oil	8	parts by weight.
Colophony	10	,,
Ceresine	20	,,
Oil of turpentine . . .	40	,,

6. Ceresine	4	parts by weight.
Petroleum	1	,,
Oil of turpentine	3	,,
Benzine	4	,,
7. American colophony	10	,,
Hard resin	0·5	,,
Wood oil	0·3	,,
Ceresine	5	,,
Cocoanut oil (diluted with oil of turpentine)	0·4	,,

CERESINE AND PARAFFIN FLOOR POLISH.

For large dancing halls, and especially those in country places frequented by the middle and lower classes, the fine wax pastes, which require considerable trouble in polishing, are replaced by preparations which give the necessary smoothness when applied in a finely divided condition. These masses may also be melted by heat and sprinkled over the floor with a brush, the further distribution being effected by the feet of the dancers themselves.

1. Ceresine (yellow)	7·5	parts by weight.
Paraffin (soft)	18	,,
Asbestos	0·5	,,
2. Paraffin (soft)	10	,,
White ceresine	2	,,
Yellow palm-oil	2	,,
Asbestos	2	,,

The asbestos is added to the melted mass, the whole stirred until nearly set, and then poured into moulds.

SHOE AND LEATHER POLISHES.

1.	Carnauba wax	5	parts by weight.
	Paraffin	5	,,
	Oil of turpentine	35	,,
	Nigrosine (sol. in oil)	3	per cent.
2.	Ceresine	10	parts by weight.
	Carnauba wax	35	,,
	Japan wax	7	,,
	Colophony	10	,,
	Oil of turpentine	120	,,
	Black, yellow, or brown coal-tar dye (sol. in oil)	3	per cent.
3.	Ceresine	3·6	parts by weight.
	Japan wax	1	,,
	Carnauba wax	0·4	,,
	Wool fat	0·5	,,
	Nigrosine (sol. in oil)	0·2	,,
	Lampblack	0·3	,,
	Oil of turpentine	15	,,
4.	Ceresine	4	,,
	Paraffin	0·5	,,
	Yellow beeswax	0·5	,,
	Colophony	0·5	,,
	Nigrosine (sol. in oil)	0·2	,,
	Lampblack	0·3	,,
	Oil of turpentine	15	,,
5.	Carnauba wax	9·39	,,
	Paraffin	9·39	,,
	Oil of turpentine	37·56	,,
	Benzine	37·56	,,
	Black coal-tar dye (sol. in oil)	6·10	,,

6.	Carnauba wax	8·584	parts by weight.
	Paraffin	8·584	,,
	Japan wax	8·584	,,
	Oil of turpentine	68·669	,,
	Black coal-tar dye (sol. in oil)	5·579	,,
7.	Carnauba wax	7·320	,,
	Paraffin	10·019	,,
	Japan wax	7·326	,,
	Colophony	3·663	,,
	Oil of turpentine	65·934	,,
	Black coal-tar dye (sol. in oil)	4·762	,,
8.	Carnauba wax	5·814	,,
	Paraffin	5·814	,,
	Spermaceti	11·628	,,
	Oil of turpentine	69·766	,,
	Black coal-tar dye (sol. in oil)	6·978	,,

PARAFFIN AND RESIN ARTIFICIAL WAXES.

The usual mixtures of paraffin and resins become greasy and sticky when kneaded by the hand, and turn brittle when kept. To obviate these defects, the mixture of paraffin and resin, heated to about 90° C., is treated with a current of air blown in through a perforated pipe at the bottom of the vessel. The resin undergoes a peculiar form of oxidation, with acrolein, acetic acid, carbon dioxide, and water as oxidation products. The mass may afterwards be boiled by steam and then coloured. This treatment converts the hard paraffin into an unalterable plastic mass, of wax-like appearance and free from stickiness, suitable to replace wax or ceresine for technical purposes. The method forms the subject of German Patent No. 101,222.

The process is accelerated by substituting oxygen or ozone for air, the same product being obtained in either case.

Further investigation has shown that liquid or solid oxidising agents may also be used with the same result: such as nitric acid, potassium chlorate, chromic acid, permanganate, and other technical reagents. For instance, if the mixed resin and paraffin be treated at about 90° C. with 8–10 per cent. of concentrated nitric acid, a portion of the resin is rendered insoluble in the paraffin (as in the air treatment) and separates out. The product is equal in quality to that from the air oxidation process, and the same effect is obtained with suitable proportions of the other oxidising agents specified. The use of liquid or solid reagents is calculated to cheapen the production of artificial waxes.

CERESINE MOULDING MASS FOR COPPER AND SILVER PLATING.

Seven parts of yellow wax are melted and mixed with an equal weight of ceresine, followed by 1–2 parts of graphite, the whole being well stirred and formed into cakes. This mass quickly sets hard and will take a clear impression when stamped, the models being easily detachable. The moulds will take a graphite coating well and are not corroded by the plating bath (potassium cyanide and silver). This mass has been known to keep in an unaltered condition, suitable for use, for a whole year.

PARAFFIN FOR BLEACHING CARNAUBA WAX.

Although the pale yellow, greenish, or grey colour of carnauba wax is no drawback to its use for many purposes, a bleached grade is demanded in commerce; but for a long time no means could be found of removing the colour, though this is merely slight, until the idea arose of employing paraffin. This gives a pure white, though of course less completely opaque, product, melting at 73–74° C. and therefore corresponding to a mixture of 30 per cent. of the vegetable wax with 70 per cent. of paraffin. There are two methods of bleaching. In the one a wooden vat is charged with enough water to cover the steam coil arranged in the bottom, and 300 parts of paraffin and 100 parts of the wax are then melted in this vessel by turning on the steam. Twenty parts of caustic soda lye (40° B.) are then added, and the whole is boiled until the impurities in the wax are precipitated. These are allowed to subside and the liquid mass is drawn off and thoroughly washed with water, from which it is separated when cold. In the second method the carnauba wax and paraffin are melted together in a large iron pan over a gas fire, an effective decolorising powder being added. The liquid mass is run through a filter press, collected in moulds, and when cold is preferably exposed to the air for a time, to bleach the outside of the cakes still further. The (unsaponifiable) waste wax from the bleaching process is used for making shoe polish, greasing leather, etc.

WAX FOR INSULATING ELECTRIC CABLES.

A wax for insulating electric cables laid underground is prepared from—

Paraffin (m.p. 40–45° C.) .	50	parts by weight.
Colophony	25	,,
Best tallow	8	,,
Carnauba wax . . .	1	,,

coloured with a coal-tar dye, soluble in oil, or with gamboge or turmeric. The paraffin and tallow are melted together, the other ingredients being incorporated by thorough stirring.

MINERAL ARTIFICIAL WAX.

Solid dry paraffin . . .	25	parts by weight.
Colophony	14	,,
Coloured tallow . . .	6	,,

are melted together. To obtain the requisite pure colour and handsome appearance in the mass, it is necessary to use very pale colophony; and 3 parts of carnauba wax may be added to increase the hardness. Gamboge gives a fine yellow colour. The product is highly suitable for industrial purposes, and also makes a very good floor polish.

CERESINE HONEYCOMB.

Bee-keepers, who attach more importance to the yield of honey than to that of wax, have welcomed a method which enables them to supply the hives with comb in a half-finished condition, so that the bees can complete it and—having less wax to produce—can turn out much larger quantities of honey. This artificial honeycomb is made of ceresine, either alone or mixed with a little beeswax or carnauba wax, and has for some years been a very profitable article of manufacture on account of the ever-increasing demand.

There are four different methods of making artificial honeycomb—

1. By means of engraved metal plates, between which the inserted thin warm sheets of ceresine are impressed under heavy pressure in a screw or hydraulic press.

2. By passing thin sheets of ceresine between engraved rollers. This method originated in America.

3. Moderately hot melted ceresine is poured into wetted plaster or similar moulds, the whole being then dipped in lukewarm water to facilitate detaching the comb.

4. The wax is cast in electrotype or type-metal moulds made by Kämpf of Mittelhufen, near Königsberg (cement), Müssigbreit of Hunichen, Ober-Lausitz (plaster), Rietsche of Bieberach, Baden (electrotype), and Ehring & Fahrenholz of Berlin (type-metal).

The casting method deserves special attention, being simple and yielding a product that is practically free from expansion in use. The chief difficulty in making the comb is to get the cells true to shape, the actual height of the walls being less material; in fact, bees will build up comb from foundation that exhibits scarcely any appreciable depth of cell. All that is needed apart from the moulds is a couple of tubs, one large and one small, and a scoop for pouring the ceresine into the moulds. The small tub containing ceresine is placed in the large tub containing boiling water, and the melted wax is ladled out into the moulds as quickly as possible, the latter being then covered up and left to cool.

ARTIFICIAL WAX MOULDING COMPOSITION.

The production of wax figures and models of various articles, such as fruit, fancy goods, etc., is still practised, though not to such a large extent as formerly. Pure beeswax being too dear for most of these articles, a suitable composition can be made from—

White wax	5	parts by weight.
White ceresine	2	,,
Hard paraffin	1	,,

together with ½–1 part of colourless resin, suitably coloured with mineral pigments or the specifically lighter colour lakes. The moulds are made of plaster or tin, divided into two or more parts, according to the extent of undercutting in the pattern, the parts being held together by any convenient means during the casting process. The inner surface of the mould being coated, thinly but uniformly, with a brush dipped in olive oil, the liquid wax mass (which should not be hotter than 180–185° F.) is poured in, the amount used depending on whether the article is to be hollow or solid. In the latter event, the mould is filled up and set aside to cool; but for hollow wares, only enough wax to fill the mould about one-quarter full is used at first. This is distributed by turning the mould round and round and pouring the surplus out again, the operation being repeated until the desired thickness is obtained. Hollow articles may be strengthened by filling up the central cavity with papier-mâché or plaster of Paris after the wax has set. In the case of heads, glass eyes are placed in the mould before the wax is poured in, and fixed in position with a composition made of 3 parts wax and 1 part Venice turpentine. When the wax has set in the moulds, the latter are taken to pieces and the contents placed in cold water to facilitate hardening, care being exercised to keep the water out of the internal cavity. This may, however, be closed with a wax cover beforehand. The finished article is trimmed up with a sharp knife to remove any casting ridges, and afterwards painted, gilded, silvered, etc.

SCHLIEMANN'S ARTIFICIAL WAX FROM PARAFFIN AND RESINS.

Ordinary mixtures of paraffin and resins have an indefinite, disagreeable colour, become sticky and greasy when kneaded in the warm hand, and turn brittle on keeping. To remove these defects, Schliemann heats mixtures of paraffin or similar hydrocarbons and resin, in any proportion, to 90° C., and blows a current of air through the mass until no further precipitation of insoluble resin occurs and the mass has acquired a peculiar sweetish smell. The object of this treatment is to oxidise the dissolved resin, and thereby modify its properties in a manner impracticable by the same treatment at a previous stage. That portion of the resin left in solution in the paraffin is deprived of its adhesive properties, whilst the other portion, of higher melting-point, is thrown down as insoluble. The products of the reaction include acrolein, acetic acid, carbon dioxide, and water, which are liberated in the gaseous state.

Any kind of paraffin is suitable, but the resin should be American or French colophony, or at any rate derived from coniferous trees and chiefly composed of abietic acid and silvic acid. The mass may be afterwards washed with water, and mixed with small quantities of solid hydrocarbons, colouring matters being added if desired. The product resembles wax in appearance, is unalterable, plastic, and adhesive, keeps well and is suitable for replacing wax or ceresine for many purposes.

SUBSTITUTE FOR CARNAUBA WAX.

The high price of carnauba wax has led to the introduction of cheaper substitutes for the manufacture of turpentine polishes, saponifiable shoe polishes, and other purposes for which the brown colour of mineral wax is not objectionable. These substitutes are generally made of mineral wax mixed with resin, large proportions of ceresine, paraffin, etc., being also added in cases where a saponifiable product is not in question.

Carnauba Wax Substitute for Use with Oil of Turpentine.

Paraffin	18	parts by weight.
Ceresine	3	,,
Colophony	4·5	
Carnauba wax . . .	6	,,
Mineral wax	6	,,

Saponifiable Substitute for Carnauba Wax.

Mineral wax	18	parts by weight.
Yellow beeswax . . .	7·5	,,
Carnauba wax . . .	4·5	,,
Colophony	3	,,

The paraffin or ceresine is melted and the other ingredients are added. When melted, the whole is well stirred, and the finished mass is poured out to cool.

PARAFFIN WRAPPING-PAPER.

Paraffin paper is largely used for wrapping products that contain a certain amount of moisture and are to be protected from drying, such as certain food-stuffs or the like; also for excluding air from preserve glasses, and for wrapping round fatty substances that penetrate ordinary paper.

Strong unsized or only lightly sized paper is laid in piled sheets on a large flat table, and a small quantity of shredded paraffin is laid on the top of the pile. The application of a hot iron melts the paraffin and causes it to penetrate through a number of the underlying sheets in succession. When the iron grows too cold, another one is taken. Instead of shredding the paraffin, a cake or stick of this material may be held in the left hand and brought in contact with the hot iron in the right hand, thus melting the paraffin and causing it to be absorbed by the pile of paper underneath. By this means a quantity of paper may be impregnated with paraffin without much trouble.

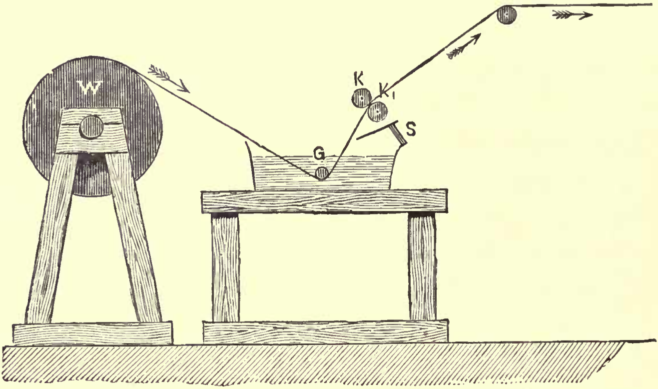


FIG. 21.—Apparatus for making Paraffin Paper.

For working on a large scale, it is preferable to deal with rolls of paper, each roll being mounted on a shaft (Fig. 21) and passed, in unwinding, through an enamelled iron trough containing paraffin which is melted by a gas, oil, or spirit stove.

A glass rod, detachably mounted on a bar, is arranged inside the trough, and a steel scraper with a non-cutting edge is mounted on the trough so as to scrape against the soaked paper as the latter is drawn onward. Any surplus of paraffin is pressed out by passing the paper between a close-fitting pair of porcelain rollers. The impregnated paper is allowed to rest in large loose folds a short distance away, and is afterwards cut into sheets of suitable dimensions or else wound on a drum.

The paraffin used may also be dissolved in benzine, carbon tetrachloride, etc., the paper being then either drawn through the solution or else coated with same by means of a brush, and then hung up to dry by the evaporation of the solvent. Drying is preferably effected in a tightly closed chest, so that the solvent can be recovered.

PARAFFIN (CERESINE) AND NAPHTHALENE MOTH PAPERS.

In these papers for checking the ravages of moths, ceresine and paraffin serve to carry the active ingredients and facilitate their adherence to the paper. When the paper is laid out, the aromatic substances gradually vaporise in a very effective manner, without coming in direct contact with the clothing, furs, etc., to be protected.

1.	Naphthalene	450	parts by weight.
	Eucalyptus oil . . .	20	,,
	Ceresine or paraffin . .	250	,,
	Spirit	110	,,

The wax is melted in an iron pan and then taken from the fire, to be mixed with the spirit containing the eucalyptus oil in solution. Whilst being kept well stirred, the preparation is applied to white blotting-paper with a wide paint-brush, the sheets being hung over threads or cords in a warm place to dry. The naphthalene should be as pure as possible, since the crude article has a very persistent and disagreeable, almost unbearable, smell. If necessary, all the moth papers now described may be scented with 1 part of oil of bergamot to each 10 parts of naphthalene; and it would pay to introduce these scented papers on the market. These papers sell particularly well at the beginning of the warm season, and are much handier to use than moth spirit and moth powder, being simply laid between the garments to be protected. They are packed for sale in packets of ten, wrapt in tinfoil or paraffin paper and well fastened up, or for large consumers they may be packed in tins holding 100. The sheets may be printed with a brand, etc.

2. Carbolic acid	50	parts by weight.
Paraffin or ceresine	50	,,
Naphthalene	100	,,

are melted together and brushed over sheets of unsized paper laid on a sheet of copper or iron.

3. Paraffin	10	parts by weight.
Olive oil	10	,,
Camphor	15	,,
Naphthalene	250	,,

are melted carefully over a coal fire, and the blotting-paper to be steeped is drawn through the mixture. A tight-fitting lid is hung over the pan, for use in the event of the naphthalene taking fire.

USE OF PARAFFIN IN MAKING WASHABLE COLOURED PAPER.

Paraffin is a very useful adjunct in the manufacture of washable coloured papers, except that it is unsuitable for highly glazed papers, though it will acquire a fine gloss under the influence of friction. There are two ways of mixing the paraffin with the colour—namely, by dissolving the paraffin beforehand in a solvent, or else by mixing it direct with the liquid colour by the application of heat. The solution may also be prepared in two ways—

1. The paraffin is melted in a large pan, over a fire or by steam heat, with constant stirring, which is continued after the pan has been removed from the source of heat and until the contents are beginning to set. About six parts of petroleum spirit, carbon disulphide, or other suitable solvent are next added and stirred up until solution is complete. The solution is stored in tightly closed vessels, or used as soon as made. In this method and the following, it is advisable to take due precautions against fire.

2. The paraffin is cut into thin shreds and placed in a hermetically closed vessel, where it is suffused with five parts of carbon disulphide and left for two or three days to dissolve, by which time a thick milky liquor will have been formed.

To prepare the colour for mixing with the paraffin dissolved by either of the above methods, 100 parts of blanc fixe are mixed with the toning colour and well stirred up with 11–15 parts of gelatine previously softened with water. The prepared colour paste is treated with 12–17 parts of the dissolved paraffin and 12 parts of softened wax, such as is usually employed in making this class of paper. If the colour is too thick to be workable, it is thinned down with lukewarm water and passed through a fine hair sieve for use.

The finished colour may be mixed with undissolved paraffin, by shredding the latter into the colour and warming the mixture up to 40° C. with continued stirring, until incorporation is complete. This method, however, is less suitable than the others, as the paraffin is less completely dissolved. The paper made by this process gives a higher lustre when stone-glazed than ordinary glacé papers. The paraffin itself has no action on the colouring matters; but as these may be affected by the carbon disulphide if left to stand before used, owing to the liberation of sulphuretted hydrogen from the size and disulphide, it is advisable to use up the colour as quickly as possible, as no injurious action can go on when the colour is applied to the paper and has become dry.

Other methods of preparing these colour mixtures are as follows :—

One part of paraffin is treated with 5 of carbon disulphide, solution being complete in 2–3 days. This milky solution (16 parts) is mixed with the colouring paste, consisting of 100 parts of blanc fixe, the colouring matter, and 12 parts of size, 11 parts of melted wax being added after a proper mixture has been obtained. Paper coated with this mass will give a high lustre when stone-glazed, and the colours will offer great resistance to the action of water. Since carbon disulphide is highly inflammable, it must be kept out of contact with naked lights or fires.

The most suitable preparation for making satin-glazed papers washable is the following: 550 parts of turpentine are boiled with 600 parts of water in a copper pan and stirred until all lumps have disappeared, whereupon 400–600 parts of an aqueous solution of shellac are stirred in, followed by 1000 parts of blanc fixe. No size or starch is needed. Other recipes for friction-glazed waterproof coloured paper are—

1\.

Colour	50 parts by weight.
Water	10 ,,
Glue	4 parts dissolved in
Water	4 parts by weight.
Paraffin	5 ,,
Wax soap	5 ,,

2\. For waterproof photographic cards—

Colour	20 parts by weight.
Glue	3 parts dissolved in
Water	6 parts by weight.
Paraffin	2 ,,

The colour, which must be properly mixed and sized, is added to the paraffin. The mixture can be applied by hand or machine; is supple, and does not break in the manner characteristic of other coloured papers when an excess of colour has been laid on. A superior gloss to other coloured papers is obtained by wiping with a cloth. After immersion in water, the paraffined coloured paper regains its gloss on drying, a feature that is also of value for photographic cards, since any trace of mounting paste, etc., can be removed by washing, and the original surface will be restored. The paraffin may be added to any colour without risk, and the longer the paper is stored the better it is.

PARAFFIN COMPOSITION FOR WATERPROOFING CARTRIDGES.

One hundred parts of paraffin and 15 of colophony are melted together, and the (cardboard) cartridge cases are steeped in the mixture until no more air bubbles appear (a sign that all the air is expelled from the cases), whereupon they are taken out, drained, and set aside to cool.

WATERPROOFING TEXTILE FABRICS WITH PARAFFIN AND CERESINE.

Paraffin and ceresine are excellently adapted for waterproofing textile fabrics, and also for protecting them from the influence of acids, alkalis, and other corrosive liquids. The method of waterproofing with paraffin and ceresine, together with solutions of fats and tar, was recommended by Heinzerling; and these agents are well suited for the purpose in the case of clothing, umbrellas, etc., which are also required to remain porous to air and not become hard and stiff. The effect obtained is due, on the one hand, to the filling of the pores in the fabric by an extremely thin layer of paraffin, ceresine, or the like on the evaporation of the volatile solvent in which the proofing agent has been dissolved; and on the other, by the fact that the absorbent capacity of the fibres toward water and the mutual adhesion between the water and fabric are entirely eliminated.

According to Heinzerling, the fabric to be waterproofed is dipped for several minutes in a 10–15 per cent. solution of paraffin or ceresine in benzine, and is then wound on a reel to allow the solvent to evaporate. For waterproof coats and ladies' dress materials, a paraffin of high boiling-point and a very pure benzine are required, in order that no bad smell may be left behind on the evaporation of the benzine. In the case of high-class fabrics a very weak solution (6–8 per cent.) is used, so that the softness of the material and the brightness of the colour may not be impaired by an unduly thick deposit of paraffin on the fibre; and care is taken to drive off the benzine afterwards by brushing and gentle pressing. When the treated material is held up to the light, the interstitial spaces between the fibres seem to be quite empty, though for the most part they are closed up by a very thin film of paraffin. Repeated bending and folding of the fabric will gradually reopen these spaces by breaking the paraffin film; but this does not make the material appreciably less waterproof, except in the case of very loosely woven, open fabrics, which of course are seldom used for making waterproof garments. The process is not so well adapted for articles that have to be frequently washed—such as bed linen, for instance—since washing gradually impairs the waterproof character of the material. The process is very cheap, especially when the benzine is recovered by means of suitable appliances, of the type illustrated in Fig. 22. Here *d* is a vessel about 40 inches in length and of sufficient breadth to allow the cloth to pass through its full width. B is a sheet-metal receptacle the bottom, *f*, of which consists of a steam-heated plate, whilst the top is roof-shaped, the ridge being in the form of a trough *k*, from which cold water trickles down both slopes of the roof. The latter is covered with canvas to check the flow of the water and retard evaporation. The water collects in the two gutters *ii*, whilst the benzine condensed on the inside is caught by the gutters *hh*. To work the apparatus, a solution of paraffin is placed in the trough *d*, and the cloth to be impregnated, *b*, is unwound from a reel *a* (fitted with a brake), from which it passes over a roller *c*, then under the detachable roller c_1 in the trough, and thence over the roller c_2, the excess of solution taken up being removed by two scrapers. In passing over the warm plate *f* in B, the solvent is evaporated, the cloth issuing at c_3 and being finally wound on the reel *g*. Both *d* and B must be covered in as well as possible, to check the evaporation of the benzine. Care must also be taken to keep the temperature from rising above 40° C. The cloth must be wound slowly on the second reel, so that it may have sufficient time to become thoroughly steeped with the paraffin solution and to dry properly in B.

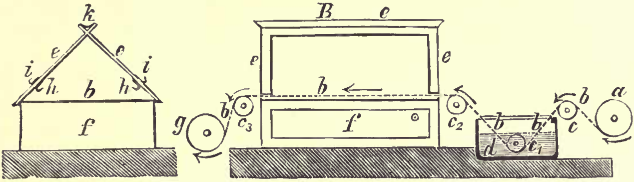


FIG. 22.—Impregnating Apparatus, with Device for recovering Benzine.

Another way of waterproofing fabrics with paraffin is to apply coatings containing a certain amount of that substance, the coatings varying in thickness and sometimes rendering the fabric stiff, brittle, and heavy, whilst in all cases it is impervious to air and perspiration.

According to Gehring, a coating of this kind is prepared from—

Paraffin	60	parts by weight.
Aluminium palmitate . .	20	,,
Yellow wax (dissolved in boiled linseed oil) . .	10–15	,,
Oil of turpentine . .	6–15	,,

The turpentine is added as a diluent, to enable the mass to be applied by the machine.

J. G. Smith prepares a similar composition from paraffin containing 15 per cent. of gutta-percha or caoutchouc dissolved by heating to 100°–110° C., the mass being rolled into contact with the fabric at 70° C. In the process of Thomas Ferneley-Wiley, the waterproofing substances—paraffin, ceresine, ozokerite, or mixtures of these with animal and vegetable waxes—are used in the solid form, made up in the shape of a plate, cylinder, etc., which is brought into contact with the material to be treated, the fabric being heated to a temperature sufficient to liquefy the proofing composition. The melting-point of this composition must not be so high as to injure the fabric. The apparatus is shown in Fig. 23,

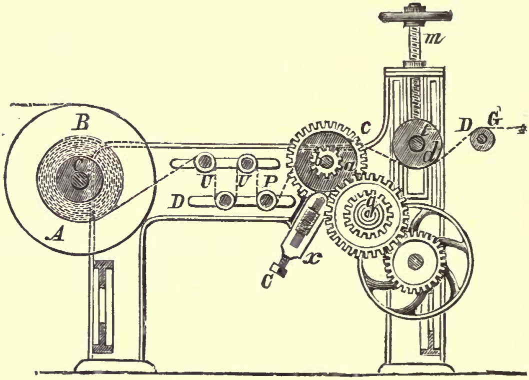


FIG. 23.—Impregnating Machine.

other modifications being illustrated in Figs. 24–26. The fabric is unrolled from a beam A, mounted in bearings on the end frames, and passes over and under a number of tension rollers P, mounted in sliding bearings U, to a hollow rubber cylinder F¹ (filled with water and preferably unprovided with a shaft) which is held between guide-rollers GFH (Fig. 24). The paraffin or ceresine, in the form of plates, is held above the rubber cylinder in a holder J, in such a manner that the lower edge rests upon the cylinder F¹, *i.e.* on the fabric. The holder J is fitted with lateral projections which slide in grooves on the frame U. The weight of the holder presses the paraffin against the fabric; and the pressure can be increased by weights applied to the top of the holder, whilst as the proofing mass is removed the plates in the holder are fed downward by means of a screw T. The plates of paraffin are made rather longer than the width of the fabric in order to allow the latter a little side play; and the ends are

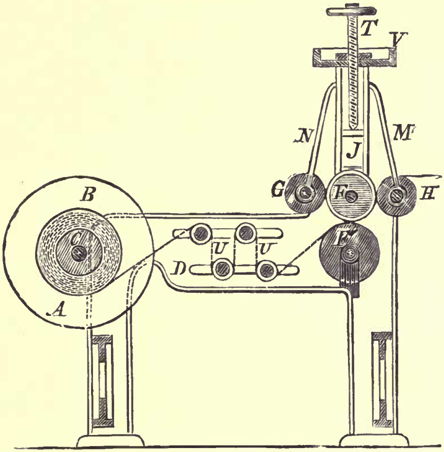


FIG. 24.—Impregnating Machine.

bevelled off, as they are not exposed to the same amount of friction as the parts in contact with the fabric. The cylinder F^1, being hollow and filled with water, is very elastic, and therefore well adapted for pressing the fabric uniformly against the perhaps unevenly worn paraffin plate. After having taken up the proofing material, the fabric, which is attached to an endless band, is drawn into a drying cylinder, where the proofing material is liquefied and caused to penetrate the fabric.

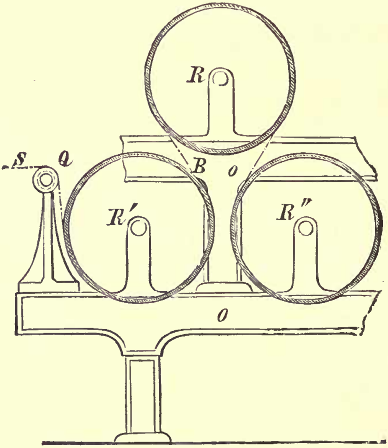


FIG. 25.—Impregnating Machine.

In another form the elastic cylinder is omitted, and the paraffin plate rests on the fabric at a suitable point between the other cylinders. In this case the amount of proofing material taken up is determined by the weight of the plate itself and the tension of the fabric. In one form of machine the waterproofing plate *d* (Fig. 26) is replaced by a cylinder cast on a solid core, with which it revolves, motion being transmitted through pinions from the main shaft. These pinions are interchangeable, in order to alter the peripheral velocity of the proofing cylinder, which is mounted in adjustable bearings by the aid of set screws. A drying cylinder is also employed in this case, to force the proofing material into the fabric, the latter running from the beam A, under and over the rollers U, over the cylinder *a*, under the roller *d*, and over the roller G (Fig. 23) to the drying cylinder. The tension of the fabric and the peripheral velocity of the roller A in relation to the speed of the fabric set up friction between the latter and the proofing material. The cylinder *a* is preferably longer than the width of the fabric; and paring knives are mounted at each end of the cylinder to shave off all the projecting portion of the material, and thus equalise the wear over its full extent. The knives are mounted on rods which are adjustable in slides *x* by the action of set screws *c*. The modification shown in Fig. 25 is similar to the one just described, except that in this case the friction between the lateral rollers is increased by the hollow cylinder filled with water.

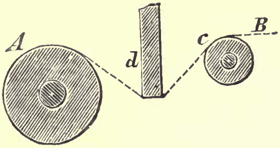


FIG. 26.—Impregnating Machine.

MAKING THIN SHEETS OF PARAFFIN.

To obtain thin sheets of paraffin, such as are used in electrical condensers, melted paraffin is poured over a fluid mass of low melting-point (*e.g.* mercury) and left to solidify before removal.

PARAFFIN STEEPING COMPOSITION FOR MATCHES.

Safety matches have for a long time been steeped with paraffin to facilitate the burning of the wood after the ignition of the match composition. With this object, the match sticks in the carriers are dipped for a certain depth in melted paraffin, and when this latter has solidified on the sticks, they are moved onward to the dipping bath proper.

According to a new process, the matchwood in a suitable form (*e.g.* rope) is boiled with an aqueous solution of paraffin, wax, oil of turpentine, resin, etc., prepared by emulsifying these substances with Turkey-red oil (or other soluble oil or soap) in the warm, and mixing the emulsion with boiling water. The following proportions have been found highly suitable: 100 parts of paraffin, melted in a pan and stirred up with 100 parts of Turkey-red oil, the emulsion being poured into 1000 parts of boiling water and dissolved by stirring. The wood is immersed in this solution, boiled for a short time, and then taken out again and dried.

IMPREGNATING CASKS AND CELLAR UTENSILS WITH PARAFFIN.

The preparation of both wine and beer necessitates scrupulous cleanliness with regard to the appliances and utensils employed, in order to guard against certain maladies, bacteria, and so on, which must be kept out of the vessels and their contents. Thus certain ferments and fission fungi are responsible for such diseases of wine as vinegar flavour, ropiness, souring, bittering, etc., and therefore great care must be bestowed on keeping all the casks and utensils in a clean state.

Germs from diseased wine or beer penetrate into the pores of the vessels and utensils holding the liquid; and these germs are exceedingly difficult to eradicate when they cannot be subjected to the prolonged action of boiling water, so that the disease may be communicated by the vessels to subsequent batches of sound wine, etc. Moreover, wood that is kept in a damp condition is specially liable to infection by mould fungi as well as by fission fungi, which rot the wood. Many attempts have been made to prevent such infection and to keep moisture and other liquids out of the pores of the wood; but the substances used are unsuitable for the purpose now in view, since they must not be brought into contact with wine or beer. For example, wood preservatives composed of mineral salts or strong-smelling tar products cannot be employed, nor such as would react with the wine or other beverage in question. Coatings of oil-paint or boiled oil can only be used for protecting parts of the wooden vessels that do not come in contact with the wine, etc.—namely, on the outside of the casks, vessels, pumps, presses, etc.; whilst the insides must be treated with some material that is incapable of acting upon, dissolving in, or reacting with the contents. Such a material is paraffin, which, when employed to impregnate wood, will entirely prevent the incursion of moisture or fungoid organisms into the pores. Of course, the paraffin used for this purpose must be thoroughly refined, perfectly inodorous, and as transparent as alabaster.

The task of impregnating wood with paraffin is easily performed, provided the wood be thoroughly dry. Otherwise the paraffin cannot penetrate far into the pores, whilst at the same time it will prevent the evaporation of the internal moisture, and thus contribute to the rotting of the inner substance of the wood.

To coat wooden utensils with paraffin, the latter is melted in a suitable iron or copper pan over a gentle fire, and when completely liquefied is worked evenly over the wood with a stiff brush. Since, however, paraffin quickly solidifies, especially when applied to a cold utensil, the coating thus formed does not penetrate deep enough into the wood, but forms merely a superficial layer which is liable to peel off in places. It is therefore necessary to subject this coating to a temperature sufficient to melt it and enable it to flow into the pores. This may be done either by passing a hot iron over the surface or, better still, by means of the flame of a soldering-lamp, the heat of which not only melts the paraffin but also warms the wood to such an extent that the paraffin is absorbed into all the pores and cracks. This treatment may be applied to the insides of casks and to the woodwork of wine-presses, the wooden rollers of grape mills, and all wooden utensils that come in contact with the must or wine (wort and beer in the case of breweries).

Smaller wooden articles, such as taps, spigots, shives, etc., are best treated by drying them thoroughly and then dipping them in the melted paraffin until no more air bubbles are given off, whereupon they are taken out, drained, and finally wiped over. By this treatment the paraffin soaks to a depth of nearly a quarter of an inch into the wood, which is sufficient to keep out any liquid; and the utensils can be cleaned after use by simply swilling them out with cold water. The pores being filled with paraffin, can no longer harbour bacteria or mould spores; neither can the wood swell in the damp or shrink on drying, and therefore vessels impregnated in this way are not liable to become leaky.

Pure paraffin is unacted upon by either weak or strong wine, as well as by water, alcohol, acids, or alkalis, and therefore the impregnated vessels, etc., may be left in contact with the finest wine for any length of time without imparting any strange taste thereto or injuring its character in the least. For this reason it is also highly advantageous to impregnate the insides of casks used for the conveyance of wine, beer, or spirits, since the shrinkage of these liquors is thereby reduced to a minimum, and the empty casks keep sweet and free from mouldiness on the return journey and can be easily cleansed by swilling out with cold water. In comparison with the advantages obtained, the cost of impregnating with paraffin is insignificant; and moreover, it enables expensive and heavy oaken vessels to be replaced by those of cheaper and lighter wood, the treatment making them last quite as long as good oak. In addition, fermenting vessels for red wine are rendered free from infection, and thus many diseases of wine, such as mouldy flavour, oily taste, taste of the wood, and those due to the action of mould fungi and disease germs, are prevented.

It should, however, be remembered that wooden vessels impregnated with paraffin must not be used for hot liquids, on account of the relatively low melting-point of the paraffin.

PARAFFIN AS A WOOD PRESERVATIVE.

Although paraffin is a good preservative for wood by reason of its absolute inalterability under the influence of the various reagents, it is not employed on the large scale on account of the expense and the large quantity of paraffin required in the melted condition. Whether the method recommended by Heinzerling for impregnating wood—namely, by steeping it in a mixture of 1 part of paraffin, 3 parts of colophony, and a suitable quantity of benzol or tar oil—has found any practical application, cannot be ascertained, but at any rate the method and apparatus used may now be described. Heinzerling states that the solution penetrates more easily and deeply into the wood than the liquefied solid paraffin; and this cannot be denied. The solution is preferably forced in under pressure, the pneumatic apparatus being suitable, with slight modifications enabling the solvent to be recovered by distillation. The manner in which this is effected can be gathered from Fig. 27. Here A is the impregnating vessel, BB the steam jacket, and C the condenser. The vessel A is fitted with a pipe *h*, for the admission of steam; a pipe *i*, leading to the air-pump; a draw-off pipe *p*, for the vapours formed during the distillation; and with a draw-off cock *m*. In addition there are a pressure gauge and a removable door *n*. The steam-jacket is provided with admission and exhaust pipes for the steam. The solution is forced under pressure into the previously dried wood, and when the latter is sufficiently impregnated, the pressure is relieved and steam is admitted into the jacket to distil off the solvent, the vessel being connected up with the condensing coil. Distillation is continued as long as any considerable amount of vapour comes over.

The impregnating solution is prepared by melting the paraffin and pouring it into twice its volume of benzine, the crushed colophony being added. The latter dissolves more readily in the fairly concentrated benzine solution than in benzine alone. The operation is best carried on in a vessel fitted with a water-seal cover and arranged for heating by indirect steam. Provided the distillation be continued long enough, and the condenser acts efficiently, the loss of benzine is comparatively slight. It must, however, be remembered that the light hydrocarbon vapours of the solvent are highly inflammable, and consequently the preparation of the solution and the condensing of the recovered vapours must be performed in an unenclosed space at a safe distance from anyfire, in order to prevent the accumulation and ignition of the vapours.

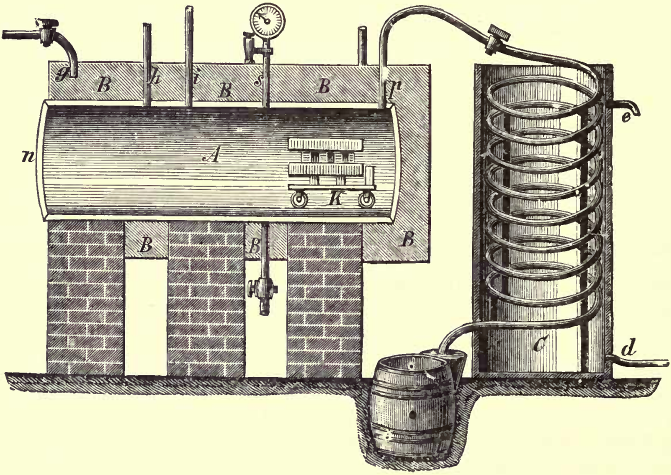


FIG. 27.—Heinzerling's Impregnating Apparatus.

Resin has valuable preservative properties, as is shown in the case of the resinous coniferous woods, but is unsuitable for impregnating timber when simply melted by heat, being too consistent and viscous, though it can be brought into suitable condition for this purpose by the aid of light hydrocarbons and paraffin, as described above.

PARAFFIN AS A BASIS FOR OINTMENTS.

A new basis for ointments has been introduced under the name of "parenol," which, according to A. Kopp, is prepared from solid and liquid paraffin, wool fat, and similar materials, the paraffins being emulsifiable with water when mixed with substances like wool fat, wax, spermaceti, etc., that are rich in higher alcohols. Specimens of these preparations are given below:—

1. With wool fat—
   Paraffin, 65 parts; wool fat, 15 parts; water, 20 parts.
2. With spermaceti—
   Paraffin, 70 parts; spermaceti, 5 parts; water, 25 parts.
3. With wax—
   Paraffin, 70 parts; bleached beeswax, 5 parts; water, 25 parts.
4. Liquid—
   Vaseline oil, 70 parts; white wax, 5 parts; water, 25 parts.

PAINT AND VARNISH REMOVERS CONTAINING PARAFFIN.

German Patent No. 150,881 describes the preparation of a medium for removing old paint and varnish, by dissolving paraffin or other wax-like substance in a liquid hydrocarbon and reprecipitating it, in the gelatinous state, by means of an alcohol that is miscible with the solvent. The best solvents for this purpose are: benzol and its homologues toluol and xylol; also ether, chlorides of carbon (dichloromethylene, chloroform, carbon tetrachloride), light hydrocarbons, etc., that dissolve paraffin and other wax-like substances and mix readily with alcohol. The gelatinising agent—methyl, butyl, ethyl, amyl, allyl, and benzoyl alcohol—must be miscible with the solvent and be practically inert towards the paraffin, etc. Paraffin, ceresine, ozokerite, beeswax, carnauba wax, mineral wax, Japan wax or hard curriers' grease, metallic soaps (aluminium gelatinate or oleate, zinc or magnesium stearate), and barium or manganese resinate, are suitable as the basis. The curriers' grease consists of a mixture of fats of high melting-point, *e.g.* the glycerides of palmitic or stearic acid, solid hydrocarbons of the paraffin type, cholesterin ether, dégras formers, etc. A particularly useful preparation is made from—

Paraffin or hard curriers' grease .	4	parts by weight.
Benzol	8	,,
Methyl alcohol	7	,,

the benzol being added in the warm, and the mass stirred till cold after the addition of the alcohol.

Eberson's (French patent) paint-remover consists of a mixture of 55 parts of alcohol, 20 of benzol, and 25 of carbon disulphide, 0·5 part of paraffin, ceresine, or wax being added to retard the evaporation of the benzol and alcohol. The last named may be replaced by 30 parts of wood spirit (methyl alcohol) and 25 parts of acetone. The paraffin, etc., is dissolved in the mixed benzol and carbon disulphide, and the alcohol is stirred in. The quantity of paraffin, etc., depends on the degree of consistency and miscibility required.

PARAFFIN ANTICORROSIVE FOR STEEL BARS, ETC.

1. One hundred parts of hard paraffin are dissolved in 200 parts of petroleum and mixed with 100 parts of quick-drying copal varnish, then thinned down to workable consistency with petroleum and ground along with two-thirds the volume of micaceous iron ore.

2. One hundred and twenty parts of ozokerite are dissolved in 300 parts of petroleum or turpentine substitute, and mixed with 200 parts of asphaltum varnish.

PARAFFIN PAINT.

A method of making paraffin paint has been patented by F. L. Colignon, the usual drying oils being replaced by a solution of paraffin in light mineral oil. Solutions of this kind are claimed to furnish good results in certain cases, the paraffins being very inert bodies, which do not oxidise in the air and are rarely if ever attacked by strong acids, so that paraffin paints should be very durable. The vegetable drying oils, on the other hand, continue to oxidise after they are dry, and are converted into brittle, resinous substances which gradually peel off, the coating of paint succumbing to the action of the air, sun, and moisture. Paraffin paints are impermeable by water—an important consideration in paints for outdoor use. Wood coated with paraffin paint does not warp, since water cannot penetrate it. The oxidation of drying oils results in the formation of free acids which alter the colour of the paint, whereas paraffin remains neutral and leaves the colour unchanged. These latter paints are also unaffected by dissolved substances like putty, which have a destructive influence on oil paints. Paraffin paint can be applied to surfaces that are already coated with other paints; and they have also a greater covering power than ordinary paints. The work also proceeds more quickly, since it is unnecessary to wait a certain time for them to dry. A by no means unimportant feature of paraffin paints is their relatively low cost. Moreover, they are applied as easily as ordinary washes, and the brush need not be drawn repeatedly over the same place. Should the paint get too thick to use, it may be easily thinned down with a little of the paraffin solution or a light hydrocarbon.

BOROTT'S CERESINE PRESERVATIVE PAINT.

Casks, beams, or timbers embedded in damp earth or water—and therefore also the keels and bottoms of ships—may be protected from rotting, and also from destruction by worms, insects, mussels, and rodents (*e.g.* rats), by giving them two coats of the following composition:—

Ceresine	4	parts by weight.
Beef or mutton tallow	32	,,
Resin (colophony)	6	,,
Powdered glass	4	,,
White arsenic	1	,,
Boiled oil	4	,,

The powdered glass is stirred into the melted tallow, resin, and ceresine, and is followed by the arsenic, which has been previously mixed with part of the boiled oil, the remainder of the latter being ground with a suitable pigment and added last of all. The mass must be melted for use and laid on whilst warm. The cans, etc., in which it is kept must be marked "Poison," and the same applies to the brushes used. The application of this composition to ships' bottoms is claimed to facilitate their passage through the water.

PARAFFIN PAINT FOR WALLS.

A solution of 1 part of paraffin in 2–3 parts of heavy coal-tar oil is prepared by the aid of warmth. It is said to be particularly suitable for walls, especially such as are exposed to the weather and are consequently damp. The solution should be warmed by dipping the can in hot water, and the walls should be heated in order to keep the paint fluid.

PARAFFIN AS AN ANTICORROSIVE.

The prevention of rust by covering iron with paraffin paper was proposed by Louis Parker, an employé of the Pennsylvania

Railway Co. Railway bridges are particularly liable to corrosion by the fumes from the locomotives, the sulphurous gases, saturated with moisture, readily attacking the steel, even when the latter has been coated with ordinary anticorrosive preparations. Coatings of vegetable oils have a very short life, linseed oil suffering considerably under the action of sulphurous gases and steam. Attempts made to use asphaltum proved successful in the experimental stage, but the practical application of this material is almost impossible in many cases, bridges in particular; and solutions of asphaltum in volatile oils, etc., form merely a porous coating, offering but slight resistance to the passage of the corrosive vapours. After having experimented for more than 10 years and with upwards of 50 different substances, Parker believes he has now solved the difficulty. A close examination of the steel bars used in the tests showed that, in many cases, the coating had remained completely intact except for a few small bubble-like projections, under which the metal was found to be spotted with rust, showing that the paint had not been impervious to moisture. On the other hand, steel bars covered with paraffin paper were found to be entirely free from rust after 2½ years in an atmosphere strongly charged with sulphurous gases. The method is very simple: In the case of an existing iron bridge, the rust is scraped off and the surface is thoroughly cleaned with wire brushes. A single coat of ordinary paint is given to the metal, and over this is laid paraffin paper, special care being taken to obtain good joints. The stratum of paper is topped with another coat of paint. The work is quickly performed—an important feature in the case of very high structures or parts difficult of access—and it can all be finished off in one operation, so that only one set of scaffolding has to be erected. In America this method has been tried successfully under the most unfavourable conditions possible—namely, in the case of iron girders which were partially immersed in brackish water, the portions covered with the paraffin paper being found entirely free from rust at the end of more than a year.

PARAFFIN COMPOSITION FOR COATING IRON.

Twenty-six parts of paraffin are dissolved in 80 parts of crude petroleum, and then incorporated with a mixture of 40 parts of linseed oil, 20 parts of flowers of sulphur, and 36 parts of pine resin. The paraffin renders the coating soft and liable to injury, but is advisable for certain purposes.

ROBSON'S COMPOSITION FOR COATING METALS.

This consists of hard pitch, alone or in association with distillation residues from petroleum or ozokerite, or any other suitable pitch which is miscible with paraffin and contains a mineral or other added oil. The object of the method is to produce a paint without any drying oil or volatile solvent, which paint can be liquefied by heat, is of low viscosity, and is just sufficiently tacky to adhere to the metal, so that it can be easily applied with a brush.

ANTICORROSIVE COMPOSITION.

Fats and oils form good anticorrosives for a time, but not permanently when exposed to atmospheric influences. Their effect is due to the formation of an impervious protective layer on the iron; but this layer is readily injured by mechanical influences, and therefore is only effective when the coated articles are to be stored indoors and not used.

When prolonged storage is in question, the fats or oils selected for this purpose must be such as have only a slight affinity for oxygen, and therefore only a small tendency to form fatty acids, since these acids attack the iron and form compounds of a rusty brown colour. Tallow is often used for coating ironware, but the layer of tallow soon changes into a greasy, rust-coloured mass, and the underlying iron gets rusty. Liquid fats, such as olive oil, are still more liable to form rust, though this tendency can be counteracted to some extent by boiling the oil for some time along with anhydrous sodium carbonate, which imparts a slightly alkaline reaction. Better results, however, are obtained with mineral oils, either alone or associated with ozokerite (ceresine); and one of the best anticorrosives known is paraffin dissolved in benzine or oil of turpentine, since paraffin is a neutral body, which does not give any acid reaction and therefore cannot set up any rusting action on the iron.

CERESINE NON-DEPOSIT PAINT.

An addition of paraffin or ceresine is recommended for the purpose of preventing the separation and deposition of red lead in paints prepared from this pigment with linseed oil or varnish, about 0·2 part of ceresine being used to 13½ parts of red lead and 3½ parts of oil or varnish. The gloss of the paint may be enhanced by adding 0·2 part of amber varnish. The resulting paint always keeps soft, and merely requires stirring up before use; and it gives coatings that do not become brittle, even after a considerable lapse of time.

PRESERVATIVE COMPOSITION FOR SOLE LEATHER.

Experience has shown that, when applied in a liquid form, substances of a fatty or waxy character will penetrate deeply into the pores of sole leather and fill up the interstitial spaces. Solutions of paraffin in volatile solvents are less suitable, because so many applications are necessary to secure the desired result. On the other hand, if the paraffin be simply melted, it chokes up the superficial pores and does not penetrate to the requisite depth. A better result is obtained with mixtures of fatty oils and paraffin, both of which will penetrate deeply into the leather, whilst the paraffin impregnates the external layers completely and preserves the soles from wear.

A composition of this kind is prepared by melting 10 parts of paraffin in an iron pan and stirring in with it 10 parts of linseed oil, the whole being then diluted with 2 parts of oil of turpentine. The mixture is applied to the warmed soles whilst in a liquid state, or else the boots are dipped in the mixture up to the welts. The leather takes up as much as it can absorb, and after the mass has set hard the surplus is scraped off.

PARAFFIN FOR IMPREGNATING CHROME LEATHER BELTS.

When once absorbed by leather, paraffin can only be expelled by heat, and being impervious to the action of acids, alkalis, water, etc., preserves the leather. Its waxy character improves the feel of the leather and also adds to the weight—a point that is sometimes of importance. Chrome-tanned leather belting can absorb up to 40 per cent. of its own weight of paraffin, though as a rule about 20 per cent. is sufficient to impart the desired properties. The paraffin (m. p. between 35 and 70° C.) is melted over a water bath, and applied to the dry leather with a brush or rag until it begins to exude from the other side. The leather is then left in cold water for 2–3 days and is finished by hammering and rolling.

PARAFFIN LEATHER GREASE.

One hundred parts of paraffin are melted in an enamelled pan and stirred up with an equal quantity of thick vaseline oil, specific gravity 0·900, followed by 10–15 parts of train oil. The boots, harness, belting, etc., to be impregnated are placed in a warm room (about 104° F.) for 8–12 hours, until all the contained moisture has been expelled, after which they are laid in the hot impregnating composition until all the air has been driven out of the pores. Boots and shoes are placed in a shallow vessel with depressions for the heels, so that the soles stand in about an inch of the liquid composition. In a few minutes they will be thoroughly impregnated, leaving the uppers porous to allow of the escape of perspiration from the feet. Hence there is no risk of the boots being injurious to health, whilst the soles may be immersed in water for hours at a time without taking up a trace of moisture. In addition they are proof against the action of acids, alkalis, or any other liquids, including liquid manure. The grease may also be applied with a soft brush.

INSTANTANEOUS BLACK STAIN FOR TANNERS.

Ceresine	9	parts by weight.
Crude wool fat	22·5	,,
Tallow	10	,,
Commercial olein . . .	23·5	,,
Vaseline oil (sp. gr. 0·885) .	70	,,
Nigrosine (sol. in oil) . .	15	,,

The ceresine, wool fat, and tallow are melted together, and the olein and vaseline are stirred in by degrees. When well mixed, the mass is taken away from the fire, and the nigrosine is dissolved by stirring till cold. The product is packed in tins.

LEATHER PRESERVATIVE.

Ten parts of ceresine are melted along with an equal weight of best Greenland seal oil, and mixed, by stirring, with 400 parts of refined mineral oil of specific gravity 0·805.

PISTON-ROD GREASE.

Ten parts of ceresine are melted with 40 parts of neutral wool fat, 25 parts of mineral oil (specific gravity 0·805) being added, and finally 25 parts of graphite are incorporated with the hot mass.

DRY LUBRICANT FOR BEARINGS.

This composition, introduced in England and America under the name "Metalline," is a graphitic mass nearly as hard as lead; it gives off a tarry smell when strongly heated, but does not melt, and exhibits little alteration from its original consistency when recooled. Microscopical examination reveals the presence of graphitic flakes, metallic particles, and amorphous white bodies. Chemical examination shows it to consist of—

Paraffin	4·98	per cent.
Carbon (graphite)	18·89	,,
Silica	6·44	,,
Lime	3·96	,,
Magnesia	1·99	,,
Ferric oxide	3·94	,,
Alumina	2·53	,,
Lead	32·40	,,
Zinc	20·07	,,
Tin	1·55	,,
Copper	2·75	,,

PARAFFIN LUBRICATING POWDER.

Lime or China clay is intimately mixed with paraffin, spermaceti, wax, or the like, the product being a solid lubricant which differs from others containing graphite in being white and being particularly suitable for lubricating looms for weaving tulle and other white fabrics.

OZOKERITE CART GREASE.

Forty parts of crude ozokerite are melted in a cast-iron pan with 220 parts of settled heavy blue resin oil. When melted, the mixture is stirred until it thickens, and is then transferred to another pan containing 20 parts of the following mixtures: (1) 100 parts of hot crude resin oil and 50 parts of dry slaked lime; (2) 150 parts of dry slaked lime in powder, 50 parts of blue mineral oil, and 50 parts of tar oil, heated together for several hours.

These two masses are well mixed, and the ozokerite and resin oil mixture is stirred in by degrees. Finally, 35 parts of caustic soda lye (containing 2 parts of turmeric) and 160 parts of water are added. For winter use, this grease may contain an addition of 20–30 parts of heavy coal-tar oil, free from creosote, this oil making it as soft as butter; but for summer use this oil would make it too fluid and liable to leak out of the kegs.

PARAFFIN AND BLACKLEAD STOVE POLISH.

Paraffin	120	parts by weight.
Japan wax	100	,,
Oil of turpentine . . .	1000	,,
Lampblack	120	,,
Finest levigated graphite .	100	,,

The paraffin and wax are melted and stirred until homogeneous. The oil of turpentine is added after removal from the fire, and when the mass is half cold, the lampblack and graphite, mixed with a little oil of turpentine, are stirred in.

PARAFFIN DRESSING FOR TEXTILES.

Paraffin, ceresine, and animal and vegetable waxes play an important part in the finishing of textile fabrics. The paraffin and ceresine used must be distributed throughout the dressing in an extremely finely divided condition, whereas the animal and vegetable waxes are saponified by the alkali present. Paraffin and ceresine belong to the soft-dressing group, and are always used in conjunction with stiffening and loading materials, as well as with starch for glossing, in which case they bring up a gloss on the fabric when put through suitable processes, especially calendering in the hot press. For this purpose they are also used with soap and other softeners, to make the fabrics glossy and supple. Paraffin is also indispensable for imparting a high gloss and smooth appearance to collars, cuffs, and shirt fronts in laundry work. The quantities of paraffin and ceresine required in textile dressings are relatively small, a very little being sufficient to produce the desired result.

RECIPES.

German Dressing.

Water	55 gals.
Potato flour	16½ lb.
Wheat or rice starch	7¾ ,,
China clay	22 ,,
Mineral white	11 ,,
Paraffin	1¼ ,,
Marseilles soap	½ ,,

Dressing for Calico Shirtings.

Wheat starch	11 ,,
Potato starch	11 ,,
White clay	11 ,,
Mineral white	8¾ ,,
White glue or gelatine	½ ,,
White wax	1¾ ,,
Paraffin or ceresine	1¾ ,,
White soap	1¼ ,,
Soda crystals	½ ,,
Water	55 gals.

Stiff Dressing for Imitation Linens.

Water	22 ,,
Wheat starch	6½ lb.
Potato flour	22 ,,
China clay	22 ,,
White soap	3¼ ,,
White ceresine	1 ,,

Tallow	1 lb.
White glycerine (28° B.)	7 pints
Common salt	2¼ lb.
Magnesium chloride	3 ,,

Dressing for Coloured Bed Covers.

Water	22 gals.
Dextrin	6¾ lb.
Wheat starch	6¾ ,,
Magnesium sulphate	4½ ,,
Paraffin	10 oz.

Dressing for Black Cotton Goods.

Water	66 gals.	
Potato starch	3½ ,,	
Wheat starch	4½ ,,	
Glue	5½ lb.	
Yellow ceresine	2¼ ,,	These are boiled together till emulsified, and then added to the solution of starch and glue.
Tallow	2¼ ,,	
Palm oil	2¼ ,,	
Soda crystals	1¾ ,,	

Dressing for Silks.

Gelatine	½ lb.
Water	4½ gals.
Paraffin	3¼ lb.
White wax	1 ,,
Castor oil	2½ ,,
Soap	2 ,,

PARAFFIN GUN-COTTON.

Gun-cotton is very difficult to transport in a dry state, being liable to explode under the influence of shock, percussion, etc., but may be rendered safe by immersing it in melted soft paraffin and squeezing out the surplus of the latter between rollers or by wringing. The gun-cotton has then a silky brown appearance and burns quietly, but is insensitive to shock or percussion.

PREPARATION FOR CLEANING GUN-BARRELS AND OTHER METALLIC OBJECTS.

This preparation is made by dissolving commercial caustic soda in alcohol and mixing the solution with a mixture of paraffin (or other suitable hydrocarbon) and vaseline oil. The caustic soda is the active ingredient, this substance combining with the acid products left by the powder, and enabling them to be wiped out of the gun along with the alcohol and hydrocarbon vehicle. Caustic potash is unsuitable owing to its hygroscopic properties, which make it a rust-producing rather than anticorrosive agent, whereas caustic soda is converted into carbonate under the influence of moisture and the carbon dioxide in the air, the product absorbing the moisture to serve as water of crystallisation. According to German Patent No. 171,232, a saturated alcoholic solution of caustic soda is incorporated with a mixture of paraffin and vaseline oil in sufficient proportions to furnish a uniform mass of the consistency of ointment, which can be spread on a plug of tow and drawn through the gun-barrel twice, this being sufficient to clean the weapon thoroughly. A better result is said to be obtained in a shorter time than with other preparations for the same purpose.

PARAFFIN ROLLERS.

Paraffin rollers are used in the textile industry for imparting smoothness and increased strength to yarn, the threads being drawn over the rollers, which are kept in continuous motion. The rollers are moulded on a core of iron wire, about one-tenth to one-fifth of an inch thick, which runs in bearings and carries a small pinion operated from the driving mechanism. The rollers are supplied either with the iron core or else hollow, so that the user can insert the core himself. In either case the rollers are cast in moulds traversed by iron wire cores, the latter being coated with glycerine if the rollers are desired hollow. In the latter event the mould core must be rather thinner than the one to be used on the machine, in order that the machine core may fit friction-tight and keep the material of the roller from slipping, since otherwise the paraffin roller would be liable to stick without rotating and would fail to secure the desired result.

PARAFFIN BATHS.

In many cases a higher constant temperature is required than can be obtained with a water bath, the latter being also unsuitable for certain purposes on account of evaporation or of the action of the steam on the material under treatment. The difficulties of the water bath may be overcome by using paraffin, which has a range of melting-points between 38 and 76° C. and does not boil below 360° C., so that it can be used for the attainment of any desired temperature between those limits by regulating the source of heat accordingly. Another advantage is that paraffin does not diminish in volume, and therefore there is no need to keep replenishing the bath unless the highest permissible temperature be exceeded. For laboratory use, Lothar Meyer recommended a paraffin bath of the following dimensions: length, 10 inches; breadth, 9 inches; depth, $2\frac{3}{4}$ inches; made of sheet brass and fitted with a cast cover plate provided with 7 openings of different sizes and 2 larger ones with removable vessels, together with 5 hemispherical nickel-plated pans. A second bath measuring $5\frac{1}{4}$ inches by 4 inches by $2\frac{3}{4}$ inches was also recommended, fitted with drawers for drying glass plates. Both these baths are provided with detachable chimneys and with handles.

PARAFFIN COMPOSITION FOR LINING BEER CASKS.

Crude pine resin is melted in an open pan, and is then freed from mechanical impurities (wood, sand, etc.) by straining it through a wire sieve. It is next mixed with 5–10 per cent. of crushed juniper berries, and distilled in an apparatus connected with a condenser. The first runnings consist of water and acetic acid, after which the application of further heat to the still drives off oil of turpentine. The pitch then remaining in the still is taken out, neutralised with lime, etc., and mixed with about 10 per cent. of refined paraffin to make it sufficiently soft, the mass being strained through a wire sieve into boxes for storage. The object of adding juniper berries is to impart perfume and disinfectant properties.

PARAFFIN PENCILS FOR WRITING ON GLASS, PORCELAIN, AND METAL.

1. One hundred parts of paraffin, 75 of tallow, and 20 of ceresine are melted over a gentle fire and mixed with 150 parts of finely powdered red lead or other pigment by stirring, the mass being poured out into strips on a damp plate of stone or glass.

2. Black—

Lampblack	10 parts by weight.
Paraffin	40 ,,
Tallow	10 ,,

White—

White lead	40 ,,
Paraffin	20 ,,
Tallow	10 ,,

Pale blue—

Light Berlin blue	10 ,,
Paraffin	20 ,,
Tallow	10 ,,

Dark blue—

Dark Berlin blue	15	parts by weight.
Paraffin	20	,,
Tallow	10	,,

Yellow—

Chrome yellow	10	,,
Paraffin	20	,,
Tallow	20	,,

The pigments are incorporated with the warmed paraffin and tallow by thorough grinding, and the mass is dried in the air so that it can be pressed into round pencils in a hydraulic press (as in making blacklead pencils). It is finally redried in the air and put into wooden casings.

3. Schwarzwald's paraffin pencils—

One hundred parts of paraffin are mixed with 2–10 parts of gum, dammar, or shellac, 1–2 parts of potassium dichromate, 50–100 parts of gold leaf, bronze, or pigment, and 5–20 parts of naphthol. The latter, besides assisting the paraffin as a binding medium, prevents the oxidation of the mass and the bleaching of the pigments. An addition of 1–10 parts of mica may also be given, to improve the grading of the colours and to give a moiré or watered appearance to the marks made by the pencils. The ingredients are heated in a pan and mixed by stirring, the resulting mass being dried to a certain consistency and finally pressed into moulds.

CERESINE MARKING-PENCILS.

Ceresine	4	parts by weight.
Carnauba wax	1·2	,,
Japan wax	2·4	,,
Asbestos	5	,,

Colouring Matter.

Blue—1·25 part of finely powdered Paris blue or ultramarine.
Red—1½ part of vermilionette.
Green—1½ part of pale or dark chrome green.
White—1½ part of zinc white.
Yellow—1½ part of chrome yellow.
Black—0·8 part of lampblack or carbon black.

The ceresine and waxes are melted together, the asbestos and colour being afterwards stirred in, and the whole is heated for half an hour longer on the water bath, with continued stirring. The mass is then poured into glass or metal tubes 10 inches in length, from which it is pressed out when cold and cut into 4-inch lengths, the remainder being melted down along with the succeeding batch.

PARAFFIN-WAX COMPOSITION FOR ELECTROTYPE.

Paraffin	700	parts by weight.
Yellow beeswax	4900	,,
Thick turpentine	175	,,
Best graphite	1225	,,

are melted together.

PARAFFIN FOR SEALING BOTTLES.

Paraffin may be used with advantage for protecting from the action of air the contents of bottles, glasses, etc., which are closed by means of corks, parchment, etc., and also for protecting corks from drying. For these purposes it is superior to sealing-wax, the poorer grades of which in particular have a tendency to crack off, whilst at the same time it does not soften. Moreover, a very thin layer of paraffin is sufficient, and its fluidity enables it to stop up all cracks and depressions completely. There is no risk of the contents of the bottle, etc., being contaminated with coloured material when the vessel is opened, as is the case with sealing-wax; and even if some of the paraffin should be accidentally swallowed, no harm will result, this substance being perfectly inert.

The method itself is simple in the extreme. Paraffin is melted in a vessel of sufficient size, and the necks of the bottles, glasses, etc., are dipped into the liquid so as to envelop the cork, etc., with paraffin, and are withdrawn slowly after a short time. If a single dipping does not give a thick enough coating, the operation may be repeated once, or even twice. Of course the bottle and cork, etc., must be perfectly dry before immersion, otherwise the paraffin would not stick, but would peel off and no longer act as a seal.

INSULATING COMPOSITION FOR ELECTRIC WIRES.

Paraffin	15	parts by weight.
Caoutchouc	40	,,
Powdered carbon . . .	15	,,
Silver sand or micaceous sand .	10	,,
Naphtha	5	,,

The paraffin is melted first, the caoutchouc being added, followed by stirring in the dry ingredients and finally by the addition of the naphtha.

ANDERSON AND KULLMANN'S INSULATING COMPOSITION.

Castor oil or other fatty oil is mixed with 1·2–3 per cent. of concentrated sulphuric acid, with careful stirring and precautions to keep down the temperature, moistened cellulose or cotton being then added to the mixture, which is next heated to 100° C. and mixed with a small quantity of flowers of sulphur or powdered sulphur. The resulting energetic reaction raises the temperature of the mixture considerably and accelerates the process. According to the degree of viscosity required, more sulphur is added, and when the latter is dissolved, the mass is poured out into cold water to solidify. The next stage consists in kneading the mass in the ordinary way between heated rollers, to increase the homogeneity of the mass and press out any adherent water, the mass being afterwards mixed with paraffin, ceresine, caoutchouc, etc. A useful mass is obtained when oil and cellulose, mixed with sulphuric acid in similar proportions, are incorporated with 20–30 per cent. of paraffin, 10–20 per cent. of sulphur, and 5–10 per cent. of gutta-percha or caoutchouc. The method of preparation may be modified, for instance by melting, in the mass, some or other of the aforesaid ingredients after having incorporated all the requisite amount of sulphur with them.

BRUNNER'S INSULATING COMPOSITION.

One hundred parts of crude ozokerite, 90 parts of amber, and 10 parts of asphaltum are melted together.

ROBSON'S INSULATING COMPOSITION.

Hard pitch is mixed with a mineral wax (paraffin, ceresine, ozokerite, or mineral wax) and an oil or fat of sufficient consistency to render the mass durable. The product is either applied direct to the metallic surface or first coated on paper, felt, or other flexible material, the coated side of the latter being then placed in contact with the object to be insulated.

FLEXIBLE ELECTRICAL INSULATING COMPOSITION.

Mineral wax (paraffin, ozokerite, etc.)	1 part by weight.
Wood tar	20 parts by weight.
Shellac	32 ,,
Asbestos, cotton, flax, wood or paper (in a perfectly dry, finely ground state) . .	32 ,,

These ingredients are mixed together in a pan at a temperature of 38–100° C. For soft compositions the quantity of mineral wax may be considerably increased; whilst for harder compositions a portion of the asbestos is replaced by ground slate. The mass is stirred until a thick paste is formed, and is then pressed into moulds of the desired shape, or rolled into sheets. If to be used for electrotype moulds, the mass is poured or pressed whilst hot into the pattern to be reproduced. In other respects the mass can be used for the same purposes and in the same way as gutta-percha and other insulating compositions, for covering electric wires, etc. If required to be of very close texture, it is subjected to heavy pressure in the moulds. It is unacted on by water or atmospheric influences, it does not soften when heated to 54° C., and is quite insensitive to the action of dilute sulphuric acid (1·6). Any scraps and used pieces of the mass can be melted down again.

GUTTA-PERCHA SUBSTITUTE.

Two parts of paraffin are melted with an equal weight of pitch and stirred with 2½ parts of Chinese wood oil until a uniform mixture is produced, whereupon 1·1 part of sulphur chloride and 0·1 part of finely powdered sulphur are stirred in and the mass is heated to 160° C. for about an hour.

WATERPROOF ELECTRICAL INSULATING COMPOSITION.

The distillates furnished by coal-tar oil at 260–280° C. are heated with certain resins—namely, Singapore copal, bastard copal, or anime—and the product is rendered flexible by the addition of a non-drying oil, paraffin, and a finely powdered, non-conducting mineral substance. For example, a useful product is obtained from: tar oil, 1 part; resin, 5; non-drying oil, 1; paraffin, 1½; soap-stone, 2 parts.

PITCH STOPPING FOR LEAKY CASKS, ETC.

1.	Yellow wax	25	parts by weight.
	Dark resin	10	,,
	Paraffin	5	,,
	Lampblack	5	,,
	Powdered gum-arabic	3	,,
	Powdered sugar-candy	2	,,

The resin and wax are melted, and the other substances incorporated by stirring, the mass being afterwards run into leaden moulds immersed in water.

2.	Yellow ceresine	27·220	parts by weight.
	Ground resin	11·340	,,
	Stearine	4·450	,,
	Powdered gum-arabic	3·400	,,
	Powdered sugar-candy	1·135	,,
3.	Japan wax	40	,,
	Paraffin	40	,,
	Pale resin	10	,,
	Chalk	10	,,
	Powdered gum-arabic	5	,,
	Powdered sugar-candy	5	,,

VASELINE FROM CERESINE AND PARAFFIN.

The so-called natural vaseline is a transparent fatty mass resembling ointment, white when bleached, inodorous or smelling slightly of petroleum, inert, and possessing a certain ductility. It is obtained, chiefly in America, in the process of refining petroleum, by heating light-coloured Pennsylvania crude oils at a low temperature, with the assistance of an air blast, until the specific gravity attains 0·865–0·875, whereupon the mass is decolorised by filtration through bone-black. Another method which is also applied to German and Galician crude oils consists in refining the residuum from lamp-oil fractions (usually diluted with benzine) by repeated treatment with sulphuric acid.

Owing to the comparatively high price of natural vaseline, artificial products have for some time been manufactured for certain technical uses. While suitable for the purposes in question, these artificial vaselines are always granular and short in texture, in contrast to natural vaseline, and they also liquefy suddenly when heated, whereas natural vaseline melts slowly.

The manufacture of artificial vaseline is a fairly simple process, and consists in melting ceresine (either alone or with paraffin) along with refined mineral oil. According to the price obtainable for the product, the oils used consist of ordinary mineral oils of greater or smaller fluidity (non-fluorescent oils, etc.) or the distillates known as vaseline oil or fluid paraffin. An artificial vaseline of this kind mixed with 1–2 per cent. of well-dried fatty soap will exhibit a ductility approximating to that of natural vaseline, whilst greater smoothness of character can be imparted by a slight addition of colophony.

Artificial vaseline intended for greasing leather, and consisting of special mineral oil rich in paraffin and semi-solid at the ordinary temperature, frequently requires merely an addition of 2–3 per cent. of ceresine or paraffin to form a suitable article.

In making these artificial vaselines, ceresine, alone or with paraffin, is melted in a suitable pan, and the mineral oil is added, followed by a little colouring matter, the whole being left on the fire for a short time longer. Soap and colophony are melted along with ceresine and paraffin, and mineral oil is added.

1. White or yellow ceresine .	25 parts by weight.
Colourless vaseline oil . .	75 ,,

2.	White or yellow ceresine	20	parts by weight.
	Paraffin	7	,,
	Dry soap	2	,,
	Vaseline oil	75	,,
3.	White or yellow ceresine	15	,,
	Paraffin	10	,,
	Colophony	5	,,
	Vaseline oil	70	,,
4.	White or yellow ceresine	5	,,
	Special mineral oil	95	,,

The colour may be imparted by vegetable or coal-tar dyes as in the case of ceresine. For black vaseline about 5 parts of fine lampblack are used.

Artificial vaselines are also prepared with natural vaseline as a basis, the paraffin and vaseline oil being added in certain definite proportions. Care is also necessary to prevent the temperature rising above 80° C. (176° F.), or the product is very liable to darken in colour. The paraffin is melted over a fire with a small portion of the vaseline oil, and the natural vaseline is added to the mixture by degrees. Owing to the liability of the mass to froth up, some of the vaseline oil is kept handy, for cooling the contents of the pan if necessary. The following proportions are said to furnish a suitable product:—

1.	Natural vaseline	60	parts by weight.
	Vaseline oil (sp. gr. 0·875)	40	,,
	Paraffin (m. p. 52° C.)	10	,,
2.	Natural vaseline	90	,,
	Vaseline oil (sp. gr. 0·880–0·885)	10	,,
	Paraffin (m. p. 52° C.)	4	,,
3.	Natural vaseline	70	,,
	Vaseline oil (sp. gr. 0·875)	30	,,
	Paraffin (m. p. 52° C.)	7	,,

4.	Natural vaseline . . .	80 parts by weight.
	Vaseline oil (sp. gr. 0·875) .	30 ,,
	Paraffin (m. p. 52° C.) . .	5 ,,

COSMOLINE

consists mainly of vaseline, the consistency of which has been modified in a greater or smaller degree by the addition of varying proportions of paraffin, ceresine, tallow, or vegetable wax.

COLLOIDAL PARAFFIN

is another name for natural or artificial vaselines (prepared from ceresine, paraffin, and vaseline oil).

VASOGENE.

Whilst it is not certain that the vasogene obtained by the following process actually forms the basis of commercial *vasogenum spissum* and the various liquid vasogene preparations, it certainly bears a great resemblance thereto.

A mixture of 100 parts of paraffin, 50 of vaseline oil, 50 of olein, and 25 parts each of 10 per cent. ammonia and alcohol is warmed and stirred. Eventually a clear, pale yellow oil is obtained, which forms a very stable emulsion when shaken up with water, mixes to a clear liquid with chloroform, oil of turpentine, etc., and dissolves iodine. If *unguentum paraffinii* or vaseline be treated in the same way, a product with the consistency of ointment and miscible with water is obtained.

VASELINE AS AN ADJUNCT TO OLEOMARGARINE.

In examining oleomargarine for the presence of extraneous substances, Geisler detected an unsaponifiable body, which further tests showed to consist of paraffin; and he afterwards found 5–11·76 per cent. of this substance in various kinds of edible fats.

On saponification with alcoholic alkali and dilution with water in the usual way, paraffin, in quantities down to 2 per cent., is revealed by a decided turbidity; but less than 2–3 per cent. cannot be detected with certainty. Geisler's opinion—which is certainly correct—is that this addition of paraffin is not made with the intention of adulterating the edible fat, but in order to improve the consistency and obtain a mixture more closely resembling butter. There is no doubt that the addition of paraffin to oleomargarine is not calculated to make it more appetising or more digestible.

HARDENING OZOKERITE.

Melted ozokerite or other hydrocarbon is treated with about 25 per cent. of oxidised oil and 3 per cent. of sulphur, the mixture being heated to 90° C. until the desired consistency is attained, whereupon it is poured into moulds or worked up in any other way. The mass forms a substitute for caoutchouc and other waterproof and insulating materials.

DRAWN CERESINE CANDLES.

Most ceresine candles are made by drawing, the ordinary process of moulding being attended with many inconveniences. For this purpose a special apparatus is used, consisting of a jacketed trough fitted with a sinker, or fork, and draw plate. This is bored with a number of conical holes of progressively larger diameter. The wick is wound on a roller and is passed over the fork, through the melted ceresine and over the farther end of the pan to the draw plate. Here it is passed through the smallest bore and attached to the roller on the other side. By drawing the wick, impregnated with ceresine, through the smallest bore in the steel or brass draw plate first, a rope of wax is obtained, which is passed through the ceresine bath and drawn through the next larger hole in the plate, an operation repeated until the proper thickness for the candles has been obtained, which done, the rope is cut into proper candle lengths. For this purpose a long, narrow table is used, the upper edge of which is provided with an upwardly projecting metal plate. An adjustable, broad, hinged knife, similar to those used for cutting up loaf sugar, moves crosswise over the table, and when 32-inch candles are to be cut, this knife is fixed in position 33¼ inches from the metal plate, one or more ropes of wax being then laid on the table so as to touch the plate, and cut off by pressing down the knife. The candle lengths are then set in a long zinc-lined dish filled with lukewarm water, and as soon as they are flexible they are taken up and rolled on the table, with the left hand, against a small board held in the right hand, so as to form the conical top. When cold, the candles are hung on metal hooks attached to a small wooden disc fitted with a handle, by means of which they are dipped in a deep steam-jacketed pan containing melted ceresine and worked about to give them a smooth surface without having to be rolled like wax candles. The tips formed at the bottom ends by this dipping are afterwards cut off, and the candles are then ready for use. When a hole is to be provided in the bottom end to fix the candles on a spiked holder, this should be bored out with a wooden or ivory awl while the ceresine is still warm.

COMPOSITION FOR DRAWN CANDLES.

1. Ceresine, 30 parts; paraffin, 60 parts.
2. Ceresine, 30 parts; paraffin (m. p. 60–64° C.), 12 parts.
3. Ceresine, 30 parts; paraffin (m. p. 40–44° C.), 12; larch turpentine, 0·8 part.
4. Ceresine, 20 parts; paraffin (m. p. 42–48° C.), 35 parts.
5. Ceresine, 15 parts; beeswax, 15; paraffin (m. p. 36–38° C.), 18; larch turpentine, 0·8 part.
6. White beeswax, 30 parts; paraffin (m. p. 60–64° C.), 12; larch turpentine, 0·8 part.

MOULDED PARAFFIN AND CERESINE CANDLES.

Moulding is the best and simplest method of making candles, and in fact it is the only method practicable for paraffin candles, this material having only a low degree of plasticity. The principle of the process consists in passing a cotton wick through the centre of a cylindrical mould, fastening the wick at the two ends, and then filling the mould with melted paraffin or ceresine and leaving it to cool. The moulds and the moulding machine, *i.e.* the assemblage of a number of moulds into a battery, form the most important part of the equipment, and are made in various forms and of divers materials. Some moulds are made in one piece, others in two halves; and the material may be either tin, an alloy of tin and lead, glass, enamelled cast iron, or brass. The inside of the moulds must be perfectly clean and smooth, free from any inequalities of surface, and must always be kept in good condition. Glass moulds fulfil these conditions better than any others, but they are rather expensive to make, are too heavy to be easily handled, and are also fragile, so that they are not very popular. The most usual material for this purpose is tin, alloyed with about one-fifth of its own weight of lead; and the moulds are generally made in two parts, which fit together accurately and are held in position by three sliding rings. All moulds that are made in one piece must be tapered slightly towards the upper end, to facilitate the extraction of the finished candles; whereas moulds that take to pieces may be perfectly cylindrical. The lower end of the mould, *i.e.* the foot, is provided with a dished enlargement, so that it can be hung on a frame for pouring. In large works it is advantageous to arrange a number of moulds (24–30) in a battery mounted in a common frame, this facilitating the work considerably. Before pouring, the moulds are preferably greased with a little pure olive oil, to prevent the candles from sticking to the sides and to enable them to be drawn out with greater ease. Divided moulds are greased with an oily rag, undivided ones with a brush. After a batch of candles has been taken out of the moulds the latter are cleaned out with oil of turpentine, perfect cleanliness being essential to the production of a first-class article.

The wick, the end of which is doubled into a loop, is inserted in the bore at the lower end of the mould, and is engaged by a long hooked wire (the other end of which is formed into a ring handle), and is drawn right through the mould. The ends of all the wicks are then looped or tied over a stick which extends all across the mould, to stretch the wicks tight and prevent them from slipping into an eccentric position, which would cause the candles to burn unevenly. Another stick is passed through the looped wick at the lower end of the mould, to keep it perfectly tight. All the moulds having been threaded with wick in this manner, they are fixed on the frame of the

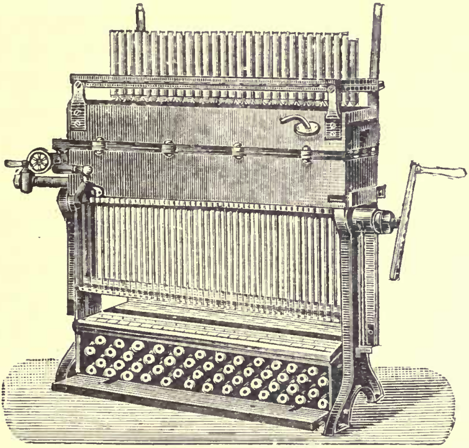


FIG. 28.—Candle-Moulding Machine.

machine, and pouring is begun. The moulding table is of iron, the moulds of cast iron, and the former is arranged in such a manner that it is suspended from lifting tackle at the ceiling and can be lowered into a wooden or iron vat filled with hot water. When the candles have set and the moulds are cold, the latter are dipped into hot water, which causes the material of the moulds to expand and thereby allow the candles to drop out readily. In melting the ceresine and paraffin, gentle heat must be applied at first, a water bath being preferable for this purpose—that is to say, a jacketed pan heated by steam or boiling water from another vessel. The maximum temperature obtainable by this means is 212° F., which is more than sufficient to melt the candle material. When the charge is sufficiently fluid, the draw-off tap from the inner vessel of the jacketed pan is opened, and the pouring tub, placed underneath, is filled with melted material. This tub must be provided with a wooden handle, and must be warmed beforehand to prevent the wax solidifying around the sides. The pourer takes the tub and fills each mould in turn, taking care not to spill any of the contents; and the tub is refilled as often as required to fill all the moulds. Any material still remaining in the pan is left until the next occasion if the work is not carried on continuously. When the moulds are cold, they are dipped in hot water for a few minutes, to loosen the candles, which are then taken out and laid on a smooth table, the ends of the wicks being cut to the proper length and the candle bottoms trimmed off with a knife. Finally, the candles are made up to the proper weight and are rubbed between flannel cloths to smooth the surface and improve the gloss.

BELMONTINE CANDLES.

About sixty years ago the Belmont Works, Vauxhall, placed on the market a new type of candle, distinguished by great transparency and high illuminating power. Their novelty consisted in the use of a special raw material and the employment of a special manufacturing process; and as they appear to have been the first paraffin candles on the market, they possess a certain historical interest. The raw material in question consisted of a semi-fluid mass obtained from dug wells in the vicinity of the river Irrawady in Burma, the natives of which country employed this material for lighting purposes, protecting timber from the ravages of insects, for medicinal purposes, etc.

At the Belmont Works, this crude naphtha was first distilled by steam at 100° C., thus driving over about 25 per cent. of the whole, in the form of a mixture of various hydrocarbons which were difficult to fractionate. This was, however, accomplished to a certain extent by a twofold or threefold rectification, which furnished products ranging in specific gravity from 0·625 to 0·860, and in boiling-point from 26·7 to 200° C. They are all water-white liquids, which will dissolve caoutchouc and do not solidify at any temperature. The vapour of the most volatile fraction possesses anæsthetic properties; and the lighter grades, known as Sherwood's oil, are powerful detergents, being capable of removing grease from silk fabrics, etc., without injuring the most delicate shades. The heavy fractions form lamp oils, burning with a dazzling white flame. The residue from the distillation process formed about three-fourths of the total weight, and was refined by melting and treating it with sulphuric acid, the impurities being deposited as a black sediment. The clarified liquid was run off and distilled with superheated steam at 150–300° C.; and the product coming over above 220° C. contained a solid substance resembling paraffin and known by the name of Belmontine.

HARDENED PARAFFIN CANDLES.

Paraffin candles may be hardened by means of oxystearic acid, according to a German patent, the most suitable proportions being about 15–25 parts of oxystearic acid to $8\frac{1}{2}$ parts of paraffin. If the two ingredients be merely melted together, a kind of segregation process is observed to occur on cooling, with the result that the candles contain a progressively increasing quantity of oxystearic acid towards the upper end, so that the different parts of the candle vary in their power of resisting the softening influence of heat. To obviate this defect, which greatly militates against the utilisation of the cheap candle material, oxystearic acid, this substance is dissolved in a suitable solvent before being incorporated with the paraffin, the best solvent for this purpose being about 4 per cent. of stearic acid (calculated on the total weight). The latter may, however, be replaced by an equal quantity of benzoic acid or cerotic acid, by 10 per cent. of palmitic acid or crude tallow, or by about 7 per cent. of crude olein. As a rule the best results are obtained by taking 3 parts of oxystearic acid to 1 of solvent. The dissolved oxystearic acid is then melted with the paraffin and moulded into candles, which are found to be of uniform hardness throughout.

PARAFFIN NIGHT-LIGHTS.

Paraffin night-lights are generally made by pouring the paraffin into a mould of thin sheet metal containing a centrally arranged wick which extends to the bottom of the mould, the mass being then left to solidify. These night-lights, however, exhibit the serious defect, that if the wick be damaged or pulled out, they are spoiled; and in any case they give a very poor light. These drawbacks are removed by providing the night-light with an independent

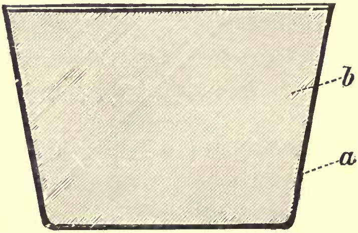


FIG. 29.—Paraffin Night-Light.

wick *c* carried in a holder *e*, which can be inserted into the paraffin mass *b* in the mould *a* and removed at will.

Fig. 29 shows the night-light without the wick.
Fig. 30 ,, a side view of the wick-holder.
Fig. 31 ,, a plan of same.
Fig. 32 ,, the finished night-light ready for use.

The wick-holder consists of a plate *d* provided with a bore *e* and with downward pointed lugs *f*, the latter being made by stamping V-shaped cuts in different parts of the plate and then turning down the triangular slips thus formed. For use, the plate *d* is pushed down so that the pointed lugs sink into the paraffin of the night-light and a wax light *c* is inserted through the bore *e* into the cavity thus formed in the mass. As the wax burns away and sinks, the wick remains in contact with the mass. When burnt out, the wick may be replaced by a new one. The wick-holder may, of course, be made in any desired shape.

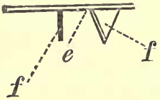


FIG. 30.

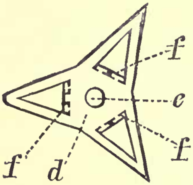


FIG. 31.

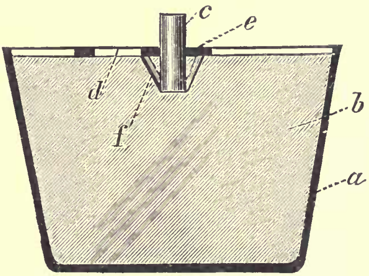


FIG. 32.

Paraffin Night-Light.

LEWY'S OPAQUE PARAFFIN.

Lewy's German patent relates to a method of manufacturing a composition of paraffin, mixtures of that substance with ceresine and stearine, or a mixture of palmitic and stearic acids. This latter is well known as the material of composite candles, whilst the product now in question is intended for making candles, wax matches, wax flowers, and so forth. The method consists in adding a naphthol, preferably β-naphthol, to the other ingredients, for the purpose of destroying the transparency of the mass and thus increasing its suitability for the purposes in question.

When β-naphthol is used, the mass acquires a fine white colour, whereas α-naphthol imparts a reddish tinge. The amount of naphthol added and the temperature of the molten mass vary with the nature of the material and the uses for which it is intended. A mass suitable for candle-making is obtained by heating 100 parts of paraffin and 2 parts of β-naphthol to 80–90° C. and stirring the mixture. The resulting candles have a handsome white appearance, in contrast to the greyish sheen of pure paraffin candles. Of course the mass may be coloured according to requirements, but the coloured candles will differ from those prepared without β-naphthol by their opacity.

BERGER'S CANDLE PARAFFIN.

According to German Patent No. 157,402, the addition of vaseline oil to paraffin, or mixtures of that substance with stearic and palmitic acids, renders the candles made from this material opaque. The addition of liquid hydrocarbons to candle material is not in itself novel, though as previously employed it produced an entirely different effect. The older method consisted in incorporating a large proportion of oils with the candle material, and necessitated the addition of an agent to prevent the candles becoming too soft. This agent, which formed an important feature of the method, consisted of a considerable addition of bone-black, which was intended to absorb and solidify the hydrocarbon. This black, however, necessarily imparted its colour to the candles; and it seems doubtful whether it would allow them to burn regularly, to say nothing of its effect on the illuminating power. Moreover, this addition of bone-black would prevent the hydrocarbon oils from removing the transparency of the material, the oils being absorbed by the particles of black. The quantities of oil, preferably vaseline oil, used in the present method may vary considerably, and the mixing is performed at a temperature selected in accordance with the material used in each case. For instance, a suitable mixture can be prepared with: paraffin, 70 parts; stearine, 13; and vaseline oil, 15 parts. The desired effect can, however, be obtained with a much smaller proportion of oil, the stearine content being at the same time reduced without affecting the result beyond lowering the cost. Such a mixture consists, for example, of: paraffin, 90 per cent.; stearine, 5 per cent.; vaseline oil, 5 per cent.

PETROLEUM WAX AND ITS USES.

The name petroleum wax has latterly been applied to distillation residues that are really asphaltum or of an asphaltic character. The composition of this material is variable. In the cold state it possesses a certain consistency and begins to soften between 30 and 35° C. It is very sticky, has none of the characteristics of fats, and is unsuitable for lubricating purposes. Dr. Berthier recently described a purpose for which this wax is eminently adapted.

It has long been known that the cracks in flooring, walls, etc., shelter nests of microbes, owing to the difficulty of cleaning out the dust accumulating in these cavities; and the attention of hygienists and architects has for some time been concentrated on means of filling up these recesses, but without any success. Boiled linseed oil, carbolineum, coal-tar, and even paraffin gradually lose their adhesive properties. Putties and lacquers crack and also gradually lose their elasticity; but Berthier is said to have now found a suitable material for the purpose, consisting of a hot mixture (85° C.) of

Petroleum wax	70 parts by weight.
Carnauba wax	30 ,,
Hydraulic lime	20 ,,

The flooring, etc., having been made thoroughly clean, the hot liquid is poured into the cracks and left to set hard, any surplus portions being afterwards scraped off with a hot spatula. The adhesive power and elasticity of the petroleum wax enable it to withstand the influence of vibration and slight flexion of the flooring. Experiments made with this preparation at the military hospital at Amelies-les-Bains gave highly satisfactory results, the filled cracks remaining perfectly sound for a period of four years.

In domestic life the proposed abolition of the dry broom has encountered great opposition, many housewives objecting to wet their floors. When the danger arising from dispersed dust is pointed out, they say that when polished floors are wiped over with a damp cloth, the wax must be renewed. Berthier, however, has introduced a preparation of petroleum wax for household use consisting of 20 parts of this wax and 100 parts of paraffin melted together, which, it is claimed, complies with all sanitary requirements. The mixture is applied cold, like beeswax, and the floors can be scrubbed and wiped with a woollen cloth. The wax adheres better when only a small quantity is used, and it need only be renewed about once a month. The floor can be washed at any time, and will recover its gloss completely when dry. The wax imparts a handsome colour to the wood, but if the ordinary appearance of a waxed floor is to be imitated, the proportion of petroleum wax in the mixture may be reduced to about 5 per cent.

Berthier also mentions cheap paints that can be made with petroleum wax and are suitable as a grounding colour in place of coal-tar, which gives a dark and dull appearance. The first coating consists of: petroleum wax, 2¾ lb.; coal-tar, 2¾ galls.; red ochre, ¾ lb.; and the second coating: petroleum wax, 5½ lb.; tar oil, 3¼ galls.; red ochre, 2¼ lb.; lampblack, 2½–5 oz. Unfortunately, these recipes do not contain paraffin, the waterproofing properties of which would certainly be advantageous; and even though it lacks antiseptic properties, it would keep out the air, and thus prevent fermentation.

THE END

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