PATENT SPECIFICATION

718,017

Date of Application and filing Complete Specification Jan. 30, 1951.
No. 2269/51.
Application made in Germany on Jan. 31, 1950.
Application made in Germany on Feb. 16, 1950.
Application made in Germany on May 5, 1950.
Application made in Germany on Dec. 9, 1950.
Complete Specification Published Nov. 10, 1954.

Index at acceptance:—**Classes 1(1),** F3B1; **2(3),** B1(F: 1); **and 91,** C(1E: 2E).

COMPLETE SPECIFICATION

Improvements in and relating to the production of Paraffin Wax

We, RUHRCHEMIE AKTIENGESELLSCHAFT, of Oberhausen-Holten, Germany, a German Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to improvements in the production of commercial paraffin waxes. It relates particularly to the production of commercial paraffin waxes from industrial mixtures of high boiling hydrocarbons, such as the products obtained in the catalytic hydrogenation of carbon monoxide.

Hitherto slab paraffin wax having a pour point of 50°/52° C. and containing hydrocarbons having about 20 to 30 carbon atoms in the molecule, and hard paraffin wax having a pour point of 90° C. or higher and containing hydrocarbons having more than approximately 28—30 carbon atoms in the molecule, could only be isolated in commercial quality from high-boiling products obtained in the catalytic hydrogenation of carbon monoxide by the use of cumbersome processing methods, because the fatty acids, esters, alcohols and aldehydes which are present in small quantities in such products have a disturbing influence. Similar difficulties also occur with many high-boiling petroleum distillates which are used for the manufacture of commercial grades of paraffin wax. Furthermore, it has not always been possible, by the process hitherto customary, to eliminate completely the usual yellow to yellowish-brown colour of the starting material.

It has now been found that these difficulties may be largely eliminated in a simple manner.

According to the invention, paraffin waxes are obtained from a hydrocarbon mixture containing constituents boiling above 320° C. by a process which comprises distilling the hydrocarbon mixture to obtain a residue boiling above 320° C., treating the residue with hydrogen at a temperature within the range 200°—260° C. and at a pressure of at least 5 kg. per sq. cm. in the presence of a hydrogenation catalyst comprising a metal and/or a metal oxide and thereafter separating paraffin wax fractions of given melting points from the treated residue by solvent extraction.

The process according to the invention is applied with particular advantage to paraffin wax mixtures which have been produced by the conversion, at medium pressure and in the presence of iron catalysts, of gas mixtures containing carbon monoxide and hydrogen. The process, may, however, also be applied to paraffin wax containing fractions obtained from other sources, such, for example, as those obtained by the low temperature carbonisation of lignite or by the distillation of shale, particularly shale of Scottish origin, as well as to fractions obtained from petroleum.

The hydrogen pressure most suitable for the hydrogenation of the starting material depends, among other factors, on the initial boiling point of the starting material. In general, a hydrogen pressure of 5 kg. per sq. cm. and over is sufficient in order to obtain commercial paraffin waxes in accordance with the invention. At gas pressures of between 30 and 60 kg. per sq. cm., substantially complete hydrogenation takes place and the end product has at suitable temperatures an iodine number, a neutralisation number, a saponification number and a hydroxyl number of zero.

The hydrogen absorption is comparatively slight as, in general, only small quantities of oxygen-containing compounds or unsaturated compounds are to

[*Price* 2/8]

be hydrogenated. For this reason it is generally sufficient to limit the treatment with hydrogen to a period of about 60—90 minutes.

The most suitable catalysts for the refining hydrogenation of the paraffin wax starting material are those containing nickel and magnesium oxide precipitated on kieselguhr, such as are used, for example, in the methanisation of industrial gases. About 10% by volume of the catalyst may be admixed with the starting material. Cobalt-containing catalysts, such as are commonly used in the catalytic hydrogenation of carbon monoxide, as well as other metal and/or metal oxide hydrogenation catalysts, may also be used. The hydrogenated product obtained under mild hydrogenation conditions, particularly under low hydrogenation pressures, is of yellow colour. When pressures of 30 kg. per sq. cm. and over are used, white products consisting essentially of saturated paraffin hydrocarbons are obtained.

If a fraction boiling over 340° C. is used as starting material, the end product consists of a mixture of hydrocarbons having more than 18—19 carbon atoms per molecule. In addition, small quantities of oily constituents are also present.

Where the hydrocarbon mixture employed as starting material in the process accordng to the invention is one produced by the hydrogenation of carbon monoxide, there is a relationship between the operating conditions of the hydrocarbon synthesis and the hydrogenation temperature for the hydrocarbon fractions boiling above 320° C.—340° C. With hydrocarbons produced at medium pressure in the presence of an iron catalyst from gas mixtures containing carbon monoxide and hydrogen, the most favourable temperature for hydrogenation lies within the range 240°—260° C. and is advantageously 250° C., whilst with hydrocarbons of the same boiling range obtained in a hydrocarbon synthesis with the use of cobalt catalysts, the optimum effect is obtained with hydrogenation temperatures of 210°—230° C. preferably 220° C.

The crude paraffin wax treated in accordance with the invention can be separated comparatively easily by extraction into substantially oil-free, slab paraffin wax containing C_{26}—C_{30} paraffin hydrocarbons and into substantially oil-free, hard paraffin wax containing paraffin hydrocarbons above C_{30}. This extraction can, for example, be carried out with a benzene-isopropanol mixture from which a part of the solvent is distilled off after each crystallisation and filtration stage. When so operating the solvent mixture consists of two components capable of forming an azeotropic mixture, one of the components being a good solvent and the other a poor solvent for high boiling paraffin hydrocarbons above C_{30}.

The component in which the high boiling paraffin hydrocarbons are relatively insoluble should be present in excess of its proportion in the azeotropic mixture. The solution obtained by extraction with this solvent mixture is cooled several times in succession, and after each cooling is freed from the paraffin wax which separates out in solid form. A given portion of the solvent mixture is distilled off each time from the solution which remains after elimination of the solid material. This method can be carried out with numerous solvent mixtures if the boiling point of the azeotropic mixture is above 70° C. and if the boiling point of the second component, present in excess, is at least 10° C. higher so that a good separation of the two solvent components is possible by distillation. For use as the component in which the high boiling hydrocarbons are relatively less soluble, that is at the higher boiling component which is present in excess, lower aliphatic alcohols, preferably isopropanol and normal propanol, are particularly suitable. A solvent which consists of equal parts by volume of benzene and isopropanol and is composed of three parts by volume of a benzene-isopropanol azeotropic mixture (66.6% benzene and 33.3% isopropanol) and one part by volume of isopropanol is particularly suitable. Instead of isopropanol, normal propanol may be used. Mixtures containing carbon tetrachloride or trichlorethylene instead of benzene are also very suitable for the process. The ratio, in the mixture, of the component which is a poor solvent for paraffin wax to the component which is a good solvent for paraffin wax depends on the starting material which is to be treated and on the end products desired.

By the use of these mixtures it is possible to separate the high boiling hydrocarbon fractions into hard paraffin wax, slab paraffin wax and oily constituents. In such a case, the components of the solvent mixture which are good solvents for paraffin wax and form the azeotropic mixture, are completely distilled off after the first cooling. The solvent which then remains consists only of the component which is a poor solvent for paraffin wax, that is to say, it consists in general of propyl alcohol. After the second crystallisation, that is to say, after the separation of the slab paraffin wax, the solvent contains only soft paraffin wax and oily constituents, and these are separated by distillation before the solvent is recycled.

The treatment of these hydrocarbon mixtures may, with the use of a single solvent, be carried out in a form which is simpler than the extraction method described. In this case lower alcohols, preferably propanol and isopropanol, are used as extraction agents, advantageously in such quantities that the oily portions of the hydrocarbon mixture are well dissolved after cooling has been effected, while the solid constituents remain practically undissolved in a suspension which can readily be pumped and filtered. Depending on the nature of the hydrogenated crude product, lower alcohols, preferably propanol or isopropanol, in quantities by weight equal to or greater than that of the hydrogenated crude product may be used. In practical operation they are heated together with the crude product, generally under a reflux condenser, and the mixture thereupon cooled to at least 20° C. and possibly even lower.

The suspension formed can then be filtered, the filtrate obtained being again separated by distillation into oil and propanol and the latter returned to the process. The filter cake obtained is freed by distillation from entrained traces of alcohol and it may then be poured and cast into plates. The solvent thus removed by distillation may also be returned to the process. This manner of operation is particularly suitable for the separation of hydrocarbon mixtures into fractions the molecules of which contain not more than 20 carbon atoms, from fractions containing the molecules with more than 20 carbon atoms. The fraction the molecules of which contain more than 20 carbon atoms can in principle, again be split into slab paraffin wax and hard paraffin wax with the use of a single lower alcohol. In this case, however, additional operations are necessary. For the separation of hydrocarbon mixtures into: 1. Oil, 2. Slab paraffin wax, and 3. Hard paraffin wax, it is therefore advisable to use benzene isopropanol solvent mixtures of the kind hereinbefore described.

The invention is illustrated by the following examples:—

EXAMPLE I.

The starting material used was a synthetic product boiling above 340° C. and it was obtained from water gas by direct passage of the gas at pressures of approximately 10—20 kg. per sq. cm. over a precipitated iron catalyst supported on kieselguhr. This starting material was grayish-yellow to light brown in colour and had the following properties:—

Pour point measured on a rotating thermometer (ASTM D_{938-49}) - -	94.0° C.
Melting point measured in a closed capillary (ASTM D_{87-42}) - - - -	104° C.
Penetration number (ASTM D_{5-52}) - - - -	17.0
Iodine number - - -	3.0
Neutralisation number (ASTM D_{974-48}) - -	0.9
Saponification number (ASTM D_{94-48}) - -	2.3
Hydroxyl number - -	6.0
Oil content - - -	10.1%
Distillation:	
up to 340° C. - -	1.2%
from 340°—460° C. -	40.2%
above 460° C. - -	58.6%

3000 grams of this crude paraffin wax were mixed into molten condition with 300 cc. of a nickel-magnesiumoxide-kieselguhr catalyst consisting of 100 parts nickel, 12 parts magnesium oxide and 50 parts kieselguhr. The mixture was poured into a pressure-resistant reaction vessel having a cubic content of 5000 cc. and was heated to 250° C. A gas mixture consisting of 85 parts by volume of hydrogen and 15 parts by volume of nitrogen was then passed into the vessel at a pressure of up to 50 kg. per sq. cm. This pressure was maintained for 90 minutes whilst the contents of the vessel were subjected to continuous agitation. A small absorption of the hydrogen took place. After termination of the hydrogen treatment, the reaction mixture was discharged from the pressure vessel and separated from the catalyst in a heated filter press. 3000 grams of a completely white or colourless end product were obtained. All the fatty acids, esters, alcohols and aldehydes originally present in the starting material had been converted into paraffin hydrocarbons. Only the ketones, present in small amount, remained unchanged. The hydrogenated product had the following properties:—

Pour point measured on rotating thermometer (ASTM D_{938-49}) - -	94° C.
Melting point measured in a closed capillary (ASTM D_{87-42}) - - - -	105° C.
Penetration number (ASTM D_{5-52}) - - - -	8.0
Iodine number - - -	0.0
Neutralisation number (ASTM D_{974-48}) - -	0.0
Hydroxyl number - -	0.0
Oil content - - - -	12.7%
Initial boiling point - -	319° C.

Distillation:	
up to 340° C. - -	3.1%
from 340°—460° C. -	42.1%
above 460° C. - -	54.8%

These figures show that as a result of the hydrogen treatment, there was a slight increase in the lower boiling constituents. For the same reason, the oil content also increased somewhat. The penetration number decreased because the softening constituents in the starting material, that is the esters and alcohols, had been hydrogenated to paraffin hydrocarbons. After the hydrogen treatment, the molten material had to be carefully protected from the action of air as it is very sensitive to oxygen.

The product of the hydrogen treatment was finely ground and treated with 15 litres of a solvent mixture which consisted of 3 parts by volume of pure benzene and 2 parts by volume of normal propyl alcohol. The hot extraction solution was cooled to 20° C. and separated from the solid constituents in a filter press. After the crystallised mass had been freed by distillation from the retained solvent, 2100 grams of hard paraffin wax of a melting point of 99.5° C. were obtained.

From the cold extraction solution remaining after separation of the hard paraffin wax 9.5 litres of solvent were distilled off. Thereupon the solution was again cooled to 20° C. and the crystallised constitutents were separated from the liquid phase in a filter press. The filter cake was freed by distillation from the solvent contained therein and gave 60 grams of slab paraffin wax of a melting point of 50°/52° C. The remaining solvent was freed from the dissolved oily constituents and was recycled to the process.

EXAMPLE II.

As starting material, a crude paraffin wax fraction which had been obtained from the products of the low temperature carbonisation of lignite and which had been hydrogenated at 280°—300° C., was used. The fraction had the following properties.

Initial boiling point - -	108° C.
Distillation:	
up to 340° C. - -	6.3%
from 340°—460° C. -	87%
over 460° C. - -	4.8%
Pour point measured on a rotating thermometer (ASTM D_{938-49}) - -	51.0 C.
Melting point measured in a closed capillary (ASTM D_{87-42}) - - - -	51° C.
Oil content (measured at 0° C. by the acetone method with the use of a quantity of acetone equal to 40 times of that of the sample tested) - -	20.84%
at 21° C. - - -	13.65%
Sulphur - - - -	traces
Iodine number - - -	0
Neutralisation number (ASTM D_{974-45}) - -	1.0
Saponification number (ASTM D_{94-45}) - -	0.2
Ester number - - -	5.2
Hydroxyl number - -	43.0
Carbonyl number - -	80.0
Aniline point - - -	108

1000 grams of this material were hydrogenated, in the presence of 100 cc. of a nickel-magnesium catalyst, at a temperature of 250° C. and at 50 kg. per sq. cm. hydrogen pressure in an autoclave provided with an agitator for a time of reaction of 90 minutes.

After termination of the hydrogen treatment, the reaction mixture was discharged from the autoclave and separated from the catalyst in a heated filter press. The filtered hydrogenated product was mixed, at a temperature of about 70° C., with an equal quantity by weight of propanol. The mixture was then cooled to 20° C. and stirred into a thin sludge or suspension. The oil-alcohol mixture was filtered off and the filter cake obtained was washed again with the same quantity of fresh propanol. After the alcohol still present in the filter cake had been distilled off, the distillation residue (slab paraffin wax) was poured into plates. The oil-alcohol mixture obtained as filtrate was separated into oil and propanol by distillation. The propanol so recovered can again be fed to the process together with the propanol distilled off from the filter cake. At the conclusion of this treatment, 850 grams of an oil-free, transparent thoroughly typical slab paraffin wax, having a pour point (measured on a rotating thermometer of 54.3° C. and characteristic values (iodene number, saponification number, neutralisation number, hydroxyl number, ester number and carbonyl number) of 0, were obtained, in addition to 150 grams of oil having a pour point of 20° C.

EXAMPLE III.

The starting material used was a synthetic product boiling above 340° C. obtained from water gas by direct passage of the gas at a pressure of approximately 10—20 kg. per sq. cm. over a precipitated iron catalyst containing kieselguhr as support. This starting material was grayish-yellow to light brown in colour and had the following characteristics:—

Iodine number - - -	3.0
Neutralisation number (ASTM D_{974-48}) - - - -	0.9
Saponification number (ASTM D_{94-48}) - - - -	2.3
Hydroxyl number - - -	6.0

The starting material was hydrogenated in a column having a length of 5 metres and an inner diameter of 59 mm. in the presence of a nickel-magnesium oxide-kieselguhr catalyst of the same composition as that used in Example I. The hydrogenation was effected in continuous manner, at a temperature of 250° C and at a pressure of 5 kg. per sq. cm., by passing the starting material and a gas mixture consisting of 85 parts of hydrogen and 15 parts of nitrogen in the same direction through the column.

After discharge from the hydrogenation column, the reaction product was collected in a pressure vessel, the pressure was then released and the reaction product was separated from the catalyst in a heated filter press. The end product obtained was of a yellow colour and had the following characteristics:—

Iodine number - - -	0.0
Neutralisation number (ASTM D_{974-48}) - - - -	0.5
Saponification number (ASTM D_{94-48}) - - - -	1.6
Hydroxyl number - - -	2.0

As can be seen from these values, the fatty acids, esters, alcohol, aldehydes and olefines present in the starting material have, in greater part, been converted into paraffin hydrocarbons. The rate of flow was 1—1.5 kg./hour.

The end product obtained was then separated by extraction, as in Example I and/or Example II, into the desired hydrocarbon groups.

EXAMPLE IV.

The starting material used was a synthetic product boiling above 340° C. and which had been obtained from water gas by direct passage of the gas at pressure of approximately 10—20 kg. per sq. cm. over a precipitated cobalt catalyst supported on kieselguhr. This starting material was grayish-yellow to light brown in colour and had the following properties:—

Iodine number - - -	4.0
Neutralisation number (ASTM D_{974-48}) - - - -	2.1
Saponification number (ASTM D_{94-48}) - - - -	3.9
Hydroxyl number - - -	7.5

The starting material was hydrogenated in a column having a length of 5 metres and an inner diameter of 59 mm. in the presence of a nickel-magnesium oxide-kieselguhr catalyst of the same composition as that used in Example I. The hydrogenation was effected at a temperature of 220° C. and at a pressure of 50 kg. per sq. cm., the starting material being passed through the column concurrently with a gas mixture consisting of 85 parts hydrogen and 15 parts nitrogen.

After emergence from the hydrogenation column, the reaction mixture was collected in a pressure vessel, the pressure was then released and the reaction product was separated from the catalyst in a heated filter press. The product obtained was colourless and had characteristic numbers (iodene number, neutralisation number, saponification number, hydroxyl number, ester number and carbonyl number) of 0.

It will thus be understood that the whole of the fatty acids, esters, alcohols, aldehydes and olefines present in the starting material, had been converted into paraffin hydrocarbons. The rate of flow was 5—7 kg./hour.

The end product obtained was thereupon separated, as in Example I, by extraction into the desired hydrocarbon groups.

The nickel-magnesium-kieselguhr catalysts which were used in the four examples, were prepared by heating a solution of nickel nitrate and magnesium nitrate and adding the solution as quickly as possible and with stirring to a sodium carbonate solution containing 100 grams of Na_2CO_3 per litre. When the two solutions had been thoroughly intermixed, kieselguhr, which had previously been roasted at 700° C., was added to the mixture. The mixture was stirred until the evolution of carbon dioxide had practically ceased, after which the precipitated mass was separated from the solution as quickly as possible in a filter press. The filtered mass was washed with distilled water until 100 cc. of the water draining from the mass required less than 5 cc. of decinormal hydrochloric acid for neutralization. The washed mass was dried in known manner and then moulded, after which it was reduced at 380° C. with a gas mixture containing 75 vol.% of hydrogen and 25 vol.% of nitrogen. In order to remove any water that might still be adherent to the moulded catalyst particles, the hydrogen/nitrogen mixture was passed over the particles at high rate of flow of approximately 1 metre/second for the first 10 minutes of the period of reduction. The reduction was then continued for a further period of 45 minutes with the gas mixture at a rate of only 0.2 metres/second. The finished, reduced catalyst contained 75% of metallic nickel.

The iron catalyst used for the production of the starting materials used in Examples I and III, was produced in the following manner. 1000 litres of a hot solution containig 40 grams Fe per litre in the form $Fe(NO_3)_3$ and 2 grams Cu per litre in the form of $Cu(NO_3)_2$, were mixed with vigorous stirring with 1050 litres of a hot solution of sodium carbonate containing 100 grams Na_2CO_3 per litre. The mixture of the two solutions was then stirred and maintained at the boil until all the carbon dioxide evolved had escaped. The *p*H value upon termination of the precipitation was 7.

The precipitated, metal compounds were separated from the solution in a filter press and were then immediately washed with hot, distilled water for 30 minutes at a pressure of 3 kg./sq. cm. above atmospheric, in order to remove as much as possible of the alkali which was present. By maintaining a *p*H value of 7, it was readily possible to reduce the alkali (calculated as K_2O) content of the filter cake to 0.4 parts per 100 parts of the total amount of iron in the cake. If the precipitation had been carried out in the alkaline range, it would have been difficult or impossible, in spite of the long period of washing, to reduce the alkali content to below 1.5—2 part of K_2O per 100 parts of iron.

The washed filter cake was made into a paste with a little water in a mixer so as to obtain an as extensive as possible division of the precipitated mass; for this purpose, 30 litres of water were used for every 100 kg. of the moist filter cake. The paste was then mixed with a further 350 litres of water until a uniform suspension of an almost syrupy consistency was obtained. 17 kg. of potassium waterglass (potassium silicate), containing 8.1% K_2O and 20.5% SiO_2, were then added to the suspension.

The suspension, after impregnation with the potassium silicate, was mixed with 2.1 litres of nitric acid (48% HNO_3) per 100 kg. of the moist filter cake for the purpose of neutralisation, the acid being added in a thin jet with intensive stirring. The cake suspension was then filtered; the filter cake contained 4.6 parts K_2O, and 25 parts SiO_2 per 100 parts iron (Fe). The filter cake was moulded into small cylindrical particles having a diameter of from 2 mm. to 4 mm. and a length of from 3 mm. to 6 mm.; the pulverulent constituents were rejected and not returned to the moulding apparatus. Extremely hard catalyst particles were thus obtained. The moulded particles were reduced for 60 minutes with a hydrogen-nitrogen mixture at a temperature of 280° C., the rate of gas flowing being 1.5 metres per second (measured linearly and in the cold). In the finished reduced catalyst, 42% of the total iron was in elementary form.

The cobalt catalyst used for the production of starting material used in Example IV was prepared in the following manner.

A solution containing 40 grams of cobalt per litre, 2 grams of thorium oxide per litre and 4.5 grams of MgO per litre in the form of their nitrates, was run with continuous stirring and at 100° C. into an equal volume of hot sodium carbonate solution containing 104 grams Na_2CO_3 per litre. After thorough stirring for a short period, 200 parts of roasted kieselguhr per 100 parts of cobalt were added to the hot mixture with stirring. After stirring for a further half-a-minute, the suspension was filtered in a filter press and the filtered mass was immediately washed with hot distilled water at 70° C. The moist, washed mass was mixed with pulverulent particles from the moulding operation hereinafter referred to, pressed, moulded by means of extrusion press and then dried in a Buettner drier. The moulded material from the drier contained 7% water; it was broken into particles and sieved to size. The catalyst particles were then reduced at 400° C. in a nitrogen-hydrogen stream for one hour, thereby reducing 50% of the cobalt to the metallic state.

The carbonyl number, iodene number, hydroxyl number and the ester number of the materials used in the examples, were determined by the following methods:—

Carbonyl number	by oximation according to the method of Kauffmann and Leite ("Fette und Oele", 1938, pages 615—6).
Iodine number	by the method of Kauffmann Holde.
Hydroxyl number	by the method of Verley and Boelsing.
Ester number	the difference between the saponification number and the neutralisation number.

The method of Kauffmann Holde and the method of Verley and Boelsing are described in "Kohlenwasser-Stoffe, Oele und Fette, sowie die ihnen chemisch und technisch nahestehenden Stoffe" Holde, 1933, the former at page 711 and the latter at page 785.

What we claim is:—

1. A process for the production of paraffin waxes from a hydrocarbon mixture containing constituents boiling above 320° C. which comprises distilling the mixture to leave a residue boiling above 320° C., treating the residue with hydrogen at a temperature within the range 200°—260° C. and at a pressure of at least 5 kg. per sq. cm. in the presence of a hydrogenation catalyst comprising a metal and/or a metal oxide and thereafter separating paraffin wax fractions from the treated residue by solvent extraction.

2. A process according to Claim 1, in which the residue boils above 340° C.

3. A process according to Claim 1 or Claim 2, in which the treatment with hydrogen is effected at a pressure within the range 30—60 kg. per sq. cm.

4. A process according to any of the preceding claims, in which the hydrocarbon mixture is obtained by the hydrogenation of carbon monoxide at medium pressures in the presence of an iron catalyst.

5. A process according to Claim 4, in which the residue obtained from the specified hydrocarbon mixture is treated with hydrogen at a temperature within the range 240°—260° C., preferably at about 250° C.

6. A process according to any of claims 1 to 3, in which the hydrocarbon mixture is a product of the hydrogenation of carbon monoxide with the use of a cobalt catalyst, the hydrogen treatment of the residue obtained from this hydrocarbon mixture being effected at a temperature within the range 210°—230° C., preferably at about 220° C.

7. A process according to any of the preceding claims, in which the said hydrogenation catalyst contains nickel and/or cobalt.

8. A process according to any of the preceding claims, in which the said hydrogenation catalyst contains nickel, magnesium oxide and kieselguhr, the catalyst being used in an amount constituting about 10% by volume of the residue to be treated.

9. A process according to any one of the preceding claims, in which the solvent is a lower aliphatic alcohol.

10. A process according to Claim 9, in which the alcohol is isopropanol or *n*-propanol.

11. A process according to any one of the preceding claims, in which the solvent is present in an amount which, at a temperature of about 20° C., is sufficient to dissolve any oily constituents present in the treated residue whilst the wax constituents of the residue are substantially undissolved.

12. A process according to any one of Claims 1 to 8, in which the extraction is effected with a solvent mixture consisting of two components which form an azeotropic mixture, one of the components being a good solvent and the other a relatively poor solvent for paraffin hydrocarbons having more than 20 carbon atoms in the molecule, the component forming the poor solvent being present in an amount in excess of its proportion in the azeotropic mixture.

13. A process according to Claim 12, in which the treated residue is dissolved in the solvent mixture, the solution so formed being subjected two or more times to the following series of operations; the solution is cooled, the wax which separates out is removed and the remaining solution is then heated for removal of part of the solvent by distillation.

14. A process according to Claim 13, in which hard paraffin wax is removed from the solution at the first cooling and at the first heating the component forming the said good solvent is completely removed from the solution, slab paraffin wax being removed in the subsequent cooling operation or operations to leave a residual mixture comprising soft paraffin wax, oil and solvent.

15. A process according to any of Claims 12 to 14, in which the component forming the good solvent is benzene, trichlorethylene or carbon tetrachloride and the component forming the poor solvent is an aliphatic alcohol, preferably a propyl alcohol.

16. A process for the production of paraffin waxes according to any one of Claims 1 to 8, in which the waxes are extracted with a mixture of solvents in a manner substantially as hereinbefore described.

17. A process for the production of paraffin waxes according to any one of Claims 1 to 8, in which the waxes are extracted with a single solvent in a manner substantially as hereinbefore described.

18. A process for the production of paraffin waxes, substantially as hereinbefore described with reference to any one of the examples.

19. Paraffin wax whenever obtained by the process of any preceding claim.

Dated this 29th day of January, 1951.
EDWARD EVANS & CO.,
14—18, High Holborn, London, W.C.1.
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1954.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.