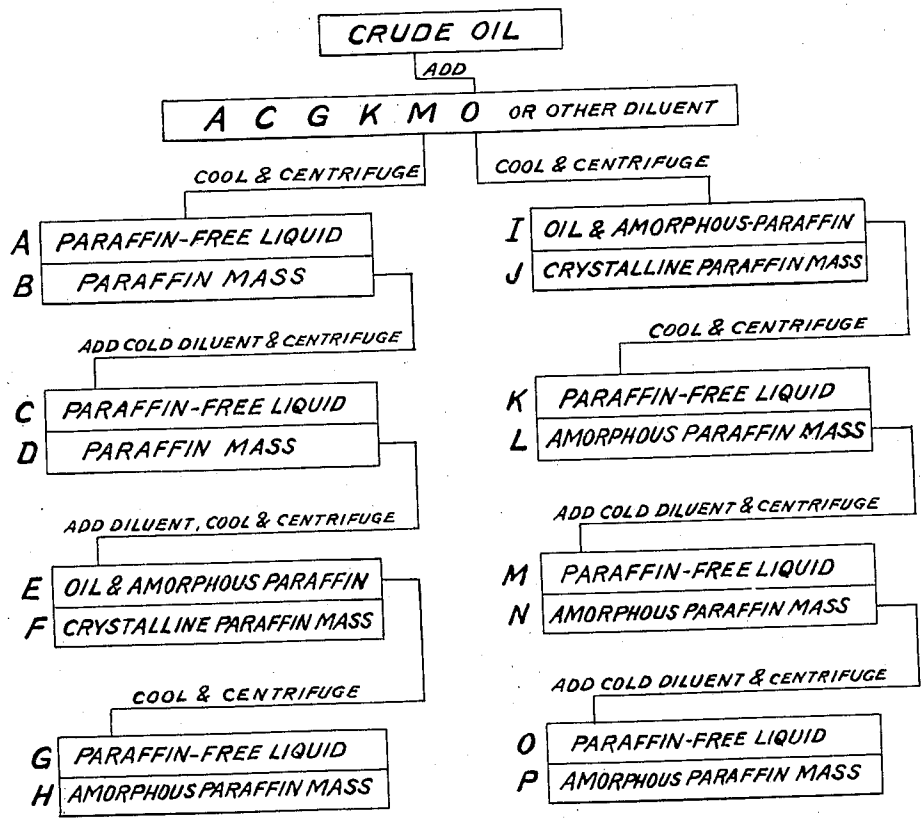
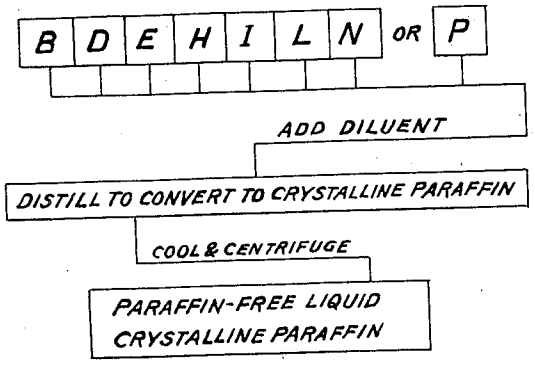
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PROCESS OF RECOVERING PARAFFIN FROM PARAFFIN CONTAINING MATERIALS

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UNITED STATES PATENT OFFICE

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PROCESS OF RECOVERING PARAFFIN FROM PARAFFIN-CONTAINING MATERIALS

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The present invention relates to the treatment of materials containing paraffin, such as (1) crude oils or tars containing paraffin, in order to produce lubricating oils with a low cold test and paraffins of high quality; (2) amorphous paraffin containing liquid hydrocarbons, natural paraffins containing oils, and other materials containing paraffin, in order to produce either (*a*) crystallized paraffins, or (*b*) refined amorphous paraffin with a high melting point; (3) distillates or residuum (containing paraffin) from crude oil or tar, in order to produce high grade paraffin of uniform composition.

Our process is adapted to the treatment of all the above specified materials for securing all the above specified products; and while the way of carrying out the process is substantially the same in all cases, the procedure, as applied to each of the specified base materials, is herein fully described.

Treatment of crude oils and tars containing paraffin, to produce lubricating oils with a low cold test and paraffins with a high melting point

The usual process, as heretofore practiced, involves the following procedure: The crude oil is distilled, and benzine or gasoline, kerosene, gas oils and paraffin oils are distilled over in the order specified. The residue in the stills is usually "cracked", i. e. subjected to destructive distillation, in which procedure certain quantities of paraffin oils are obtained in addition to large quantities of gas and coke or asphalt. By cooling this paraffin oil the paraffin crystallizes and can be filtered off by means of filter presses. The oil relieved of paraffin is then redistilled in order to obtain lubricating oils. This process is objectionable, because the preceding distillation must be effected in such a way that the viscosity of the oil is highly reduced, as paraffin crystallizes only with difficulty out of oils with high viscosities and is then very difficult to filter off.

If the crude oil contains only very small quantities of asphalt, the residues left after distilling off benzine, burning oils and paraffin oil are treated directly for producing lubricating oils with high viscosities. These residues, however, contain the paraffin as "wax" (amorphous paraffin), which is precipitated in a form that cannot be filtered, inasmuch as the mass will block the filters at once. Therefore, such residue is usually diluted with gasoline or naphtha, then cooled and caused to stand in large tanks, and on account of its higher specific gravity the amorphous paraffin settles by gravity. There is thus obtained an upper layer of a relatively paraffin-free oil which is used for the manufacture of highly viscous oil, and a lower layer of a heavy mass rich in paraffin. This method certainly avoids impairing the viscosity of the heavy oil by cracking incidental to ordinary fractional distillation, but the paraffin is incompletely precipitated and the oil obtained has a rather high cold test. Further, the method is very time-consuming, and large quantities of gasoline and oil are always tied up in the manufacturing procedure.

It has also been proposed to treat this residue, after dilution and cooling, by means of centrifuges, whereby the separated paraffin is caused to deposit at the periphery of the separator bowl, from where it is removed in some way or other continuously, if possible.

It has been also proposed to dilute the residue obtained with gasoline when only benzine and lamp oil have been distilled off, to cool and then to centrifuge. Although in this case the material does not suffer so much as from cracking, temperatures are used that cause decomposition.

Methods that involve dilution by gasoline (naphtha), have the disadvantage that they require large quantities of gasoline and two distillations; benzine and kerosene being first

distilled off from the crude oil and then about the same quantity of gasoline being added to dilute the residue. Consequently, large distilling plants and large quantities of fuel are required.

The usual methods for refining the paraffin are also objectionable. It is generally believed that the paraffin is contained in the crude oil mainly in amorphous form, and therefore distillation is used to convert the amorphous paraffin to crystallized paraffin, which can then be freed from the oil in several ways. The drawback of this method is that the paraffin, which exists in the residue in a highly viscous solution, must be exposed to an intentionally destructive distillation, in order to obtain it in the form of sufficently large crystals for the subsequent refining process. This procedure produces paraffin of relatively low melting point and oil of relatively low viscosity. Inasmuch as the market price of paraffin decreases as its melting point is lowered, a loss to the manufacturer is involved.

The present invention comprises a method of manufacture by which a material free from or poor in paraffin is obtainable for the manufacture of lubricating oils, and by which a material rich in paraffin, and with a low viscosity, may be obtained for the manufacture of paraffin, and wherein the double distillation above mentioned is avoided; whereby the viscosity of the oil, as well as the structure of the paraffin contained in the crude oil, can be completely preserved.

These advantages are obtained by separating the paraffin directly out of the crude oil. When the latter has been relieved of water and solid impurities—by centrifugation or by some other means—it is cooled so that the paraffin contained in it is precipitated in a solid state and can be separated by centrifuges. If the process is applied to crude oils that contain relatively small quantities of light components, a diluting agent, for instance gasoline, may be required to make the oil sufficiently free flowing to allow centrifugation.

The invention does not exclude the primary treatment of the crude oil with chemicals or in some other way, in order, for example, to free it from any asphalts contained therein, or from ingredients or unsaturated hydrocarbons similar to asphalt.

If the crude oil itself contains crystalline paraffin, it is possible to let the same crystallize by slowly cooling it while preventing disturbing effects as far as possible, and in this case a fractional separation of the paraffin can be effected by two centrifugations. For this purpose so much material per unit of time is passed through the separator in the first centrifugation that mainly the paraffin crystals already formed and harder paraffins are thrown against the bowl wall, while oil carrying the larger part of the amorphous paraffin is discharged from the central or inner zone of the bowl. The second centrifugation is performed after another cooling to a lower temperature, and with a relatively slow flow of the liquid into the separator, so that the amorphous paraffin is thrown against the bowl wall, while oil free from or poor in paraffin is discharged from the separator.

The paraffin-free oil, or the oil poor in paraffin, as obtained by one or several centrifugation operations, and which—as above pointed out—retains all the characteristics of the crude oil except the paraffin constituents, can then be treated, in order to obtain oil products inclusive of lubricating oils, in accordance with any known treatment of a natural paraffin-free crude oil. The output of paraffin-free crude oil obtained in this way, i. e. without any dilution with gasoline, or, at least, with relatively small quantities of a dilution liquid, is about 80 to 90%, according to the content of paraffin in the original crude oil.

The mass which, in the centrifugal operation, is thrown on the bowl wall contains a considerable proportion of highly viscous oil. A further step in the process involves a substantial reduction in the proportion of this viscous oil in order to render the paraffin more suitable for subsequent refining. To this end, the centrifugally separated paraffin mass is diluted with any material (for instance, gasoline) that will dissolve the oil, and the mixture of paraffin and solution of viscous oil and gasoline is centrifuged. As the paraffin mass obtained in the first centrifugal operation is already well cooled, it is advisable to cool the dissolving or diluting liquid separately, and to add it in a cooled condition. For certain kinds of crude oil, however, it may be of advantage to dissolve the paraffin mass in gasoline at a higher temperature, and to precipitate the paraffin by another cooling process.

If the quantity of viscous oil in the paraffin mass obtained in the last described centrifugation is still too high, the mass may be diluted and centrifuged once more. The procedure is repeated until a product usable for the subsequent refining process is obtained. As the paraffin-free liquid obtained by repeated centrifugations naturally becomes more and more pure, i. e. contains after each centrifugation, a smaller proportion of heavy hydrocarbons than is contained in the product of the preceding centrifugation, it is advisable to use, for instance, the paraffin-free liquid from the third centrifugal operation (the second centrifugation of the diluted paraffin mass) as a diluent for the second centrifugal operation (the first centrifugation of the diluted par-

affin mass) in order to economize with the diluting liquid and distillation.

The following is a specific example of a process conducted in accordance with the general description hereinbefore given:

It may be assumed that we start with crude oil capable of being treated without dilution. Any traces of paraffin that may be contained in the "paraffin-free" liquid obtained by centrifugation are ignored. It may be further assumed that, in the operation of centrifuging the crude oil, the paraffin mass obtained contains 40% of paraffin, although, in practice, this percentage will vary. Two examples of treating the paraffin mass thus obtained in the centrifugation of crude oil may be given:

(1) Dilute once and centrifuge. This treatment involves two centrifugal operations in the entire process.

(2) Dilute and centrifuge twice. This treatment involves three centrifugation operations in the entire process.

In the second example, the first dilution is effected with paraffin-free liquid taken from the third centrifugation. In order to render the results obtained in the two instances comparable, such predetermined quantities of diluting liquid are added that in both cases the same quantities of material will be centrifuged.

Starting with crude oil of the following composition:

	Per cent
Benzine	14
Paraffin	3
Other hydrocarbons	83

The compositions of the separated constituents after the first centrifugation are as follows:

	(a) Paraffin-free liquid		(b) Paraffin mass	
	Kg.	%	Kg.	%
Benzine	13.35	14.4	0.65	8.7
Paraffin			3.00	40.0
Other hydrocarbons	79.15	85.6	3.85	51.3
	92.50	100.0	7.50	100.0

Example of single centrifugation of paraffin mass.—The paraffin mass (*b*) is diluted with 45 kilograms of benzine, the whole being then subjected to centrifugation. Constituents of the following compositions are obtained:

	(c) Paraffin-free liquid		(d) Paraffin mass	
	Kg.	%	Kg.	%
Benzine	41.50	92.2	4.15	55.3
Paraffin			3.00	40.0
Other hydrocarbons	3.50	7.8	0.35	4.7
	45.00	100.0	7.50	100.0

Example of multiple centrifugation of paraffin mass.—The paraffin mass (*b*) obtained in the first centrifugation is diluted with 18.75 kilograms of the paraffin-free liquid obtained by the third centrifugation mentioned below. This liquid contains 18.05 kilograms of benzine and 0.70 kilograms of other hydrocarbons. The thus diluted paraffin mass mentioned has the following composition:

	Kilograms	Per cent
Benzine	18.70	71.5
Paraffin	3.00	11.4
Other hydrocarbons	4.55	17.1
	26.25	100.0

After centrifugation the separated constituents have the following compositions:

	(e) Paraffin-free liquid		(f) Paraffin mass	
	Kg.	%	Kg.	%
Benzine	15.07	80.5	3.63	48.5
Paraffin			3.00	40.0
Other hydrocarbons	3.68	19.5	0.87	11.5
	18.75	100.0	7.50	100.0

The paraffin mass (*f*) is diluted with 18.75 kilograms of benzine and centrifugated, this being the third centrifugation.

The separated constituents have the following compositions:

	(g) Paraffin-free liquid		(h) Paraffin mass	
	Kg.	%	Kg.	%
Benzine	18.05	96.25	4.33	57.75
Paraffin			3.00	40.00
Other hydrocarbons	0.70	3.75	0.17	2.25
	18.75	100.00	7.50	100.00

If the benzine is distilled off from the paraffin masses (*d*) and (*h*) paraffin masses of the following compositions respectively are obtained:

	First example		Second example	
	Kg.	%	Kg.	%
Paraffin	3.00	89.5	3.00	94.6
Other hydrocarbons	0.35	10.5	0.17	5.4

If the crude oil contains too small a proportion of light components to be directly centrifugated, it is advisable to use a paraffin-free liquid obtained in a subsequent operation as a diluting agent, gasoline being saved in this way and, consequently, the distilling costs reduced.

The invention is not limited in its application to crude oils, but can also be used advantageously for treating paraffin-containing tars, mineral coals and lignites, shales and other bituminous materials.

The applicability of the method is not dependent on the use of coolers and centrifugal

separators of special construction, the method being workable with coolers and centrifuges that have been heretofore used in the mineral oil industry. However, certain types of coolers and centrifuges are preferable.

For the cooling procedure, regenerative apparatuses are especially to be recommended, for if such be used, the low-cooled liquids running from the separator may be used to pre-cool the base material or the diluting means.

Bowls with disks and bowls without disks may be used for the centrifugation, the first-mentioned bowls being preferred on account of their higher efficiency. It is of course advantageous to use centrifuges from which the paraffin mass as well as the paraffin-free liquids may be discharged continuously; but it is also practicable to use centrifuges wherein the paraffin mass accumulates on the wall of the bowl, whence it is discharged periodically, if necessary after stopping the bowl.

On account of the consistency of the paraffin mass, its continuous discharge from the bowl does not generally offer any difficulties. In some cases it may be advantageous, however, when working continuously, to have recourse to special means in order to facilitate the discharge of the paraffin mass. For instance, mechanical auxiliary means may be used in order to scrape off the mass from the bowl wall and to bring it toward the center, whence it may be led away without difficulty. It is also possible, however, to float out the mass or to wash it out. In the floating operation, a liquid, cooled down to a suitable temperature, is introduced at the outer wall of the bowl. The liquid must have a higher specific gravity than the paraffin mass. A traveling cushion is then formed on which the mass floats and together with which it is discharged. This procedure, however, has the drawback that it may be difficult at times to separate the paraffin mass from the cushioning liquid without impairing the quality of the former.

This inconvenience can be avoided if the paraffin mass is sludged out by utilizing the paraffin mass already obtained by centrifugation as a sludging liquid. It will be understood that it is desirable, where the paraffin is being continuously discharged, to so constrict the outlet for the paraffin relative to the outlet for the lighter oil, that the paraffin mass will be as rich in paraffin as possible. In such case, the quantity of the paraffin mass may be so small and, consequently, the velocity thereof so low, that large paraffin crystals, if there are any present, may stick to the outer wall of the bowl and accumulate thereon. If, however, the velocity of flow is then increased by the addition of a large quantity of the paraffin mass that has been already centrifugated, any paraffin crystals adhering to the bowl wall will be torn off or they will be prevented from depositing thereon. In order not to reduce the capacity of the bowl, the sludging liquid is introduced separately, and not admixed with the liquid mixture whose constituents are to be centrifugally separated. In bowls with disks, it is therefore advantageous to introduce the sludging liquid between the outer bowl wall and the disks. If this space adjacent the bowl wall be divided into a number of axial channels, the transverse section of the partition walls and, consequently, the quantity of the sludging liquid, may be reduced.

The discharge of the paraffin mass can also be facilitated by reducing the viscosity of the mass. This may be obtained by the arrangement of a heating surface effective to melt the paraffin mass, wholly or partly. This method can also be used in conjunction with a sludging liquid, which in this case could be introduced at a somewhat higher temperature than that of the separated paraffin mass. It is, however, always necessary to regulate the application of heat in such a way that the annular wall of paraffin immediately adjacent the inner or central zone of oil will not be so raised in temperature as to go into solution with the oil with which it contacts. For example, the temperature of the sludging liquid may be just sufficiently high to melt the layer of paraffin adjacent to the sludging liquid but not high enough to melt the layer of paraffin adjacent to the oil.

The bringing of the separated paraffin mass to and out the center of the bowl is facilitated if the same be required to flow through a number of radially or conically arranged channels at a comparatively high velocity. The said channels may also be used to advantage when the above-mentioned space is not divided into axial channels. Such devices to increase the velocity of the paraffin mass offer advantages in cases where no sludging liquid is used.

Treatment of amorphous paraffin materials, oil-containing waxes, and other materials containing paraffin, to produce refined crystallized paraffin

In the process, as heretofore practiced, the amorphous paraffin is converted to crystallized paraffin by destructive distillation, and afterwards it is freed from adhering oil by means of filter presses. If, among the oils contained in the materials, there is oil of a high viscosity which renders the growing of crystals and, consequently, the filtering and sweating of the masses difficult, this distillation must take place at a high temperature, with a partial destruction of the highly viscous oil, which causes the paraffins to be partly destroyed and their melting points to be lowered.

In the process involving our invention the highly viscous oil is washed out of the paraffin

masses, permitting subsequent and very careful distillation of the masses, thereby avoiding a reduction of the viscosity of such oil through destructive distillation.

The process involving our invention is also applicable to paraffin masses in which the paraffin has been changed to a crystallized form.

In accordance with our process, the paraffin masses are first freed from water and solid impurities, by centrifugation, or in some other way.

The paraffin masses are then diluted with a light hydrocarbon liquid capable of dissolving the liquid hydrocarbons in the paraffin mass, for instance, with gasoline (naphtha), whereupon the masses are cooled. The paraffin is thus precipitated in a solid state, and is removed from the liquid mixture by means of centrifuges. In certain cases it may be useful to separately cool the paraffin mass and the diluent liquid and to mix them in a cooled condition.

If the mass contains crystallized as well as amorphous paraffin, it may be preferable to use fractional centrifugation. For this purpose the diluted paraffin mass is first moderately cooled and centrifugated, in which operation substantially all the crystallized paraffin is separated. Then the material from which the crystalline mass has been separated is cooled to a lower temperature and the remaining paraffin is separated by centrifugation. In the first operation the centrifuge is run at a relatively high capacity, and the second operation at a relatively low capacity.

It is obvious that the greater the amount of the added diluent hydrocarbon, the greater the reduction in the content of highly viscous oil, and as the paraffin mass centrifugated out at the bowl wall contains an oil of the same composition as does the diluted mass before centrifugation, the content of highly viscous oil in the separated paraffin mass will be correspondingly reduced.

If necessary, the crystalline mass obtained after the first centrifugation, is again diluted, cooled and centrifugated. The second centrifugation (of the remaining paraffin) may also be fractional. In each case the centrifugation may be repeated until the paraffin mass is sufficiently freed from highly viscous oil to allow of further refining.

The amorphous paraffin is then distilled in order to convert it to crystals. In order to facilitate this distillation and to better protect the paraffin against splitting (which it is desired to avoid), an oil of a viscosity and of a boiling point lower than those of the paraffin may be added to the paraffin mass before distillation. The added oil then dissolves the paraffin, and the solution distils at a lower temperature than that at which the paraffin alone will distil.

The paraffin mass obtained in the distillation process may be freed from the oil either by filtering or sweating (which hitherto has been the usual procedure) or preferably, by the above-described treatment comprising dilution, cooling, and centrifugation, which may be repeated until a salable product is obtained. As of course the paraffin-free liquid obtained by repeated centrifugations becomes more and more pure, i. e. contains after each centrifugation, a smaller proportion of heavy hydrocarbons than after the preceding centrifugation, it is preferable to use the paraffin-free liquid from a subsequent centrifugation as a diluent for a preceding centrifugation. Paraffin masses obtained in other ways may be treated in the same manner, but the distilling process is then of course omitted.

If the paraffin mass contains asphalt, compounds similar to asphalt, and unsaturated hydrocarbons, these ingredients can be removed by chemical reagents or by other methods used in the refining of crude oil, and such refining treatment may either take place before the first centrifugation, or at some other stage in the complete process above-described, according to the nature of the paraffin mass to be treated.

Treatment of amorphous paraffin masses, oil-containing waxes and other materials containing paraffin, to produce refined amorphous paraffin with a high melting point

It is believed that no method exists which is economically workable on a commercial scale for producing, from natural or artificial amorphous paraffin masses, refined amorphous paraffin with a high melting point. Therefore, such paraffin masses are at present mainly used as insulating masses, and for similar purposes, whereas natural oil-free amorphous paraffin has a high value on account of its high melting point, and in a refined condition it is much higher valued than paraffin.

In order to produce refined amorphous paraffin of high melting point, the process involving our invention is so applied that the amorphous paraffin in the oil-containing masses is freed from oil without altering the structure of the paraffin. The invention is also applicable to such paraffin masses that contain, in addition to amorphous paraffin, paraffin in crystals or in some other form.

The paraffin mass is first freed from water and solid impurities by centrifugation. If the paraffin mass contains asphalt, or substances similar to asphalt, or unsaturated carbohydrates, it is treated with chemicals or by any means known in the oil refining art in order to remove said substances.

The mass is then diluted with some light hydrocarbon liquid (for instance gasoline)

after which it is cooled. The paraffin is thus precipitated in a solid condition and may be removed from the diluted mass by centrifugation. In certain cases it is advisable to cool the paraffin mass and the diluent liquid separately and to mix them together in cooled condition.

If the paraffin mass contains crystallized paraffin, or amorphous paraffins of different hardness, it may be of advantage to use fractional centrifugation. For this purpose the diluted mass is first moderately cooled and centrifugated, in which operation the harder paraffins will be separated. Then the material from which the paraffins have been separated is further cooled and the remaining paraffin is separated by a second centrifugation.

If the paraffin mass separated out in the first centrifugation is not of the required quality, it is diluted, cooled and again centrifugated. A similar fractional centrifugal treatment may be applied to the softer amorphous paraffin separated out in the second centrifugation. In each case the operation is repeated until, after the mixture entrained has been ejected, a marketable product is obtained. As of course the paraffin-free liquid obtained by repeated centrifugations becomes more and more pure, i. e. contains, after each centrifugation, a smaller proportion of heavy hydrocarbons than after the preceding centrifugation, it is preferable to use the paraffin-free liquid obtained in a subsequent centrifugation as a diluent for a preceding centrifugation. In this way economy is attained in the use of diluting agents and also with respect to the distillation and the fuel consumption.

With paraffin masses containing large quantities of asphalt, it may be useful to divide the chemical refining into two or more operations. The work may, for instance, be performed in the following way.

The paraffin mass to be treated, after water and solid impurities have first been removed, is diluted, cooled and centrifugated, in order to remove a portion of the highly viscous oil contained in the mass, and thus facilitate the action of the reagents used. The paraffin mass obtained from the centrifuge is then subjected to chemical refining, whereby the main portion of the asphalt is removed, thus facilitating separation of the paraffin in the subsequent centrifugation process. If the mass has not been diluted in connection with the refining operation, it is diluted, and cooled and again centrifugated. According to the properties of the mass and the result aimed at, the mass obtained in this operation is either centrifugated again, or else subjected, at this stage, to stronger chemical refining, whereupon the procedure comprising dilution and centrifugation is continued until a marketable product is obtained.

Treatment of distillates or residuum (containing paraffin) from crude oil or tar, to produce high grade paraffin of uniform composition

When using the methods hitherto employed for obtaining paraffin out of some crude material, a mixture of the different paraffins contained in such material is obtained. As these paraffins have melting points varying within wide limits, a mixture is obtained having a melting point near that of the paraffin which melts at the lowest temperature, which of course means a loss to the manufacturer, in that the market prices of paraffins increase with their melting points.

By proceeding in accordance with the present invention, we secure, by means of fractional cooling and centrifugation, isolation of practically uniform paraffin out of distillates, as well as residues, from crude oil or tar.

We proceed by taking the material from which the paraffin is to be isolated and cooling it to a temperature at which only paraffins of the highest melting points are precipitated in the solid state. After these have been separated by means of centrifugation, the liquid, freed from solid ingredients, is cooled further, whereby new quantities of paraffins of lower melting points are precipitated, which, too, are separated by centrifugation. This procedure is repeated until all the paraffin has been separated out of the liquid. It is thus possible to divide the paraffin contained in the base material into any number of fractions of different melting points, according to the prevalent market conditions.

Where the separation of the paraffins out of oils of a high viscosity is not substantially complete, it may in many cases be of advantage to split the paraffin, first into one or a few fractions, in the way described, from the starting material, and then to separate the rough fractions obtained by the following treatment. The paraffin masses obtained are diluted with a suitable liquid, such as gasoline, which dissolves liquid hydrocarbons, and then are fractionally cooled and centrifugated. It is thereby possible to separate the paraffins, with regard to the melting points, to the degree desired; the advantage of the procedure being that the highly viscous oils are washed off, allowing refining of the paraffin to be readily effected in accordance with known methods.

The method described above has, especially in the treatment of distillation residuums of high viscosity, the advantage over ordinary methods that part of the paraffin is removed at higher temperatures, and when lower temperatures are made use of, only a relatively small proportion of paraffin remains in the liquid from which the paraffin has been largely separated, and consequently the ma-

terial remaining to be centrifugally treated is maintained in a relatively fluid condition and therefore requires little or no dilution. Otherwise, the distillation of large quantities of diluting liquid would be comparatively costly.

In order to diagrammatically show two complete processes comprising different alternative embodiments of our invention, containing essential as well as non-essential but preferential features, a drawing is attached hereto which, in connection with the foregoing description, requires no special description.

What we claim as new and desire to secure by Letters Patent is:—

1. The process of separating paraffin from hydrocarbon material containing paraffin and other hydrocarbons, which comprises diluting the material with a liquid hydrocarbon, cooling the diluted material to a temperature adapted to precipitate a part of the paraffin, centrifugally separating the paraffin mass containing said precipitated paraffin from a lighter specific gravity constituent containing the remaining paraffin and the bulk of the other hydrocarbons, cooling such remaining material to precipitate additional paraffin, centrifugally separating from such remaining material the paraffin mass containing the paraffin precipitated by the second cooling from a lighter specific gravity constituent comprising a substantially paraffin-free liquid, adding a diluent to the second paraffin mass and centrifugally separating paraffin from a lighter specific gravity constituent comprising a substantially paraffin-free liquid, and in the course of the foregoing operation utilizing the lighter specific gravity constituent separated out in a later centrifugation as a diluent preparatory to an earlier centrifugation, and utilizing the paraffin mass separated out in a later centrifugation to facilitate, in an earlier centrifugation, the removal of the centrifugally separated paraffin mass from the locus of centrifugation; distilling one of said paraffin masses containing amorphous paraffin at the temperature required to convert amorphous paraffin to crystalline paraffin, diluting the crystalline paraffin mass and cooling to precipitate the crystalline paraffin, and centrifuging to separate the crystalline paraffin.

2. The process of separating paraffin from material containing such paraffin in both amorphous and crystalline form and other hydrocarbons, which comprises cooling the material to a temperature adapted to precipitate crystallized paraffin, centrifugally separating such crystallized paraffin, cooling the remaining material to a lower temperature to precipitate the remaining paraffin, and centrifugally separating the paraffin precipitated in the second cooling.

3. The process of separating paraffin from material containing such paraffin in both amorphous and crystalline form and other hydrocarbons, which comprises diluting the material with a liquid hydrocarbon, cooling the diluted material to a temperature adapted to precipitate crystallized paraffin, centrifugally separating such crystallized paraffin, cooling the remaining material to a lower temperature to precipitate the remaining paraffin, and centrifugally separating the paraffin precipitated in the second cooling.

4. The process of separating paraffin from material containing paraffin and viscous hydrocarbons, which comprises cooling said material to precipitate paraffin, centrifugally separating a paraffin mass, comprising precipitated paraffin and some of said viscous hydrocarbons, from the bulk of said viscous hydrocarbons, adding a diluent capable of dissolving hydrocarbons to the centrifugally separated paraffin mass to reduce its proportional content of said viscous hydrocarbons, and centrifugally separating, from the diluted paraffin mass, paraffin containing a relatively small proportion of said viscous hydrocarbons.

5. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises adding a diluent and cooling to precipitate paraffin, centrifugally separating from the diluted material the paraffin mass containing precipitated paraffin and some of said other hydrocarbons, adding a diluent capable of dissolving hydrocarbons to the centrifugally separated paraffin mass to reduce the proportional content of said other hydrocarbons in the resultant mixture, and centrifugally separating from the diluted paraffin mass paraffin containing a relatively small proportion of said other hydrocarbons; the material from which the paraffin is separated in the last mentioned centrifugation being used as a diluent for the material which is subjected to the first mentioned centrifugation.

6. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises cooling the material to a temperature adapted to precipitate paraffin, centrifugally separating the paraffin mass, comprising precipitated paraffin and some of said other hydrocarbons, from the bulk of said other hydrocarbons, adding a diluent capable of dissolving hydrocarbons to the centrifugally separated paraffin mass to reduce the proportional content of said other hydrocarbons in the resultant mixture, centrifugally separating from the diluted paraffin mass paraffin containing a smaller proportion of said other hydrocarbons than was contained in the first paraffin mass, adding a similar diluent to the paraffin mass separated out by the second centrifugation, and centrifugally separating from the last named diluted paraffin mass paraffin containing a

still smaller proportion of said other hydrocarbons.

7. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises cooling the material to a temperature adapted to precipitate paraffin, centrifugally separating the paraffin mass, comprising precipitated paraffin and some of said other hydrocarbons, from the bulk of said other hydrocarbons, adding a diluent capable of dissolving hydrocarbons to the centrifugally separated paraffin mass to reduce the proportional content of said other hydrocarbons in the resultant mixture, centrifugally separating from the diluted paraffin mass paraffin containing a smaller proportion of said other hydrocarbons than was contained in the first centrifugally separated paraffin mass, adding a diluent capable of dissolving hydrocarbons to the paraffin mass separated out by the second centrifugation to still further reduce the proportional content of said other hydrocarbons, and centrifugally separating from the last named diluted paraffin mass paraffin containing a still smaller proportion of said other hydrocarbons; the material from which the paraffin is separated in the last mentioned centrifugation being used as the specified diluent for the material which is subjected to the immediately preceding centrifugation.

8. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises adding to said material a diluent capable of dissolving hydrocarbons and cooling to precipitate paraffin containing a reduced proportion of said other hydrocarbons, centrifugally separating the paraffin mass, comprising a diluent and some of said other hydrocarbons, from the bulk of said hydrocarbons, adding a diluent capable of dissolving hydrocarbons to the centrifugally separated paraffin mass to further reduce the proportional content of said other hydrocarbons in the resultant mixture, centrifugally separating from the diluted paraffin mass paraffin containing a smaller proportion of said other hydrocarbons than was contained in the first centrifugally separated paraffin mass, adding a diluent capable of dissolving hydrocarbons to the paraffin mass separated by the second centrifugation to still further reduce the proportional content of said other hydrocarbons, and centrifugally separating from the last named diluted paraffin mass paraffin containing a still smaller proportion of said other hydrocarbons; the material from which the paraffin is separated in one of the later specified centrifugations being used as the diluent for the material which is subjected to an earlier centrifugation.

9. The process of separating paraffin from material containing paraffin in both amorphous and crystalline form and other hydrocarbons which comprises diluting the material with a liquid hydrocarbon, cooling the material to precipitate crystallized paraffin, centrifugally separating the mass comprising said precipitated paraffin from the remaining material, cooling the remaining material to precipitate amorphous paraffin, centrifugally separating the mass comprising the paraffin precipitated in the second cooling from the remaining material, and diluting one of said paraffin masses with a liquid hydrocarbon and centrifuging to recover the paraffin more nearly free of said other hydrocarbons.

10. The process of separating paraffin from material containing paraffin and other hydrocarbons which comprises diluting the material with a liquid hydrocarbon, cooling the material to precipitate crystallized paraffin, centrifugally separating the mass comprising said precipitated paraffin from the remaining material, cooling the remaining material to precipitate amorphous paraffin, centrifugally separating the mass comprising the paraffin precipitated in the second cooling from the remaining material, diluting one of said paraffin masses with a liquid hydrocarbon and centrifuging; and utilizing the constituent of lighter specific gravity separated in a later centrifugation as a diluent preparatory to an earlier centrifugation.

11. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises adding a diluent capable of dissolving hydrocarbons and cooling to precipitate paraffin, centrifugally separating the paraffin mass, comprising paraffin and some of said other hydrocarbons, from the bulk of said other hydrocarbons, thereby reducing the proportional content of said other hydrocarbons in the paraffin mass, repeating the diluting and separating operation on the separated paraffin mass, and utilizing a paraffin mass that has been so separated out in one operation to facilitate the removal in another operation of the centrifugally separated paraffin mass from the centrifuge.

12. The process of separating paraffin from material containing paraffin and other hydrocarbons, which comprises adding a diluent capable of dissolving hydrocarbons and cooling to precipitate paraffin, centrifugally separating the paraffin mass, comprising paraffin and some of said other hydrocarbons, from the bulk of said other hydrocarbons, thereby reducing the proportional content of said other hydrocarbons in the paraffin mass, repeating the diluting and separating operation on the separated paraffin mass, utilizing a paraffin mass that has been so separated out in one operation to facilitate the removal in another separation of the centrifugally separated paraffin mass from the centrifuge, and utilizing the lighter specific gravity constituent separated in a later centrifugation as a

diluent preparatory to an earlier centrifugation.

13. The process of separating paraffin from material containing paraffin in both amorphous and crystalline form and other hydrocarbons, which comprises diluting the material with a liquid hydrocarbon adapted to dissolve said other hydrocarbons, cooling the material to precipitate crystallized paraffin, centrifugally separating the precipitated paraffin from the remaining material containing the remaining paraffin, cooling the remaining material to precipitate amorphous paraffin, centrifugally separating paraffin precipitated in the second cooling from the remaining material, diluting the paraffin that has been centrifugally separated at the lower temperature with an oil of a viscosity and boiling point below that of the paraffin, distilling the solution thus formed at the temperature required to convert the paraffin to a crystalline state, and separating the last mentioned crystalline paraffin from the major part of any other hydrocarbons with which it is associated after the distillation.

14. The process of securing crystalline paraffin from material containing amorphous and crystalline paraffin and other hydrocarbons, which comprises adding a diluent capable of dissolving hydrocarbons, subjecting the diluted material to centrifugal treatment and chilling required to separate a paraffin mass of which the paraffin is mainly amorphous, distilling the paraffin mass at the temperature required to convert amorphous paraffin to crystalline paraffin, and separating the crystalline paraffin from the major part of other hydrocarbons with which it is associated after the distillation.

15. The process of securing crystalline paraffin from material containing amorphous and crystalline paraffin and other hydrocarbons, which comprises adding a diluent capable of dissolving hydrocarbons, subjecting the diluted material to centrifugal treatment and chilling required to separate a paraffin mass of which the paraffin is mainly amorphous, distilling the paraffin mass at the temperature required to convert the amorphous paraffin of the mass to crystalline paraffin, diluting the paraffin mass with a solvent liquid having a viscosity below that of the paraffin, cooling to precipitate the crystalline paraffin, and centrifuging to separate the crystalline paraffin from the diluting agent and other hydrocarbons with which it is associated after the distillation.

16. The process of producing crystalline paraffin from hydrocarbon material containing amorphous and crystalline paraffin and other hydrocarbons which comprises diluting the material with a solvent liquid having a viscosity below that of paraffin, cooling the diluted material, centrifugally separating from said material a paraffin mass constituent containing some of the paraffin and a lighter specific gravity constituent containing the remainder of the paraffin, taking the lighter of said constituents and repeating the operation applied to the original material until there is separated a mass containing paraffin which is mainly amorphous, distilling said mass at the temperature required to convert amorphous paraffin to crystalline paraffin, cooling to precipitate the crystalline paraffin, and centrifuging to separate the crystalline paraffin.

17. The process of producing crystalline paraffin from hydrocarbon material containing amorphous and crystalline paraffin and other hydrocarbons, which comprises diluting the material with a solvent liquid having a viscosity below that of the paraffin, cooling the diluted material to a temperature adapted to precipitate paraffin, centrifugally separating a constituent containing said precipitated paraffin from a lighter specific gravity constituent, centrifuging one of said separated constituents and thereby separating a constituent containing amorphous paraffin from a different specific gravity constituent, centrifuging the last named constituent and thereby separating an amorphous paraffin mass from a relatively paraffin free liquid, utilizing the lighter specific gravity liquid constituent separated out in a later centrifugation as a diluent preparatory to an earlier centrifugation, and distilling the amorphous paraffin mass at the temperature required to convert amorphous paraffin to crystalline paraffin.

18. The method of separating paraffins from liquid hydrocarbons which comprises so subjecting oil to distillation and chilling as to produce liquid hydrocarbon fractions containing precipitated paraffins that to a substantial extent are in crystalline condition, subjecting the chilled crystalline paraffin-containing liquid hydrocarbons to centrifugal force to effect a separation of the bulk of the paraffin from the bulk of the liquid hydrocarbons and, while the centrifugal force is operative, subjecting the separated paraffin to a mechanical action adapted to facilitate its expulsion from the locus of centrifugal separation.

19. The method of separating paraffins from liquid hydrocarbons which comprises subjecting oil to fractional distillation and thereby producing liquid hydrocarbon fractions containing readily filterable paraffin and other liquid hydrocarbon fractions that contain paraffin not readily filterable, chilling the latter to precipitate the paraffin and subjecting the chilled paraffin-containing liquid hydrocarbon fractions to centrifugal force to effect a separation of the bulk of the paraffin from the bulk of the liquid hydrocarbons, and, while the centrifugal force is operative, subjecting the separated paraffin to a

mechanical action adapted to facilitate its expulsion from the locus of centrifugal separation.

20. The improvement in the treatment of petroleum oils for the removal of paraffin wax contained therein, comprising subjecting the oil to a cracking treatment to convert at least a portion of the wax contained therein to a crystalline form, subsequently chilling the oil and subjecting the cold oil to centrifugal force whereby the wax and wax free oil are separated into layers, mechanically discharging the wax in substantially solid form from the zone in which it is subjected to centrifugal force in a continuous manner at one point with movement toward the axis of rotation, and continuously discharging the wax free oil from said zone with movement toward the axis of rotation at another point.

21. The improvement in the treatment of petroleum oils for the removal of paraffin wax contained therein, comprising subjecting the oil to a cracking treatment to convert at least a portion of the wax to a crystalline form, subsequently chilling the oil in the presence of a diluent and subjecting the chilled mixture to centrifugal force whereby the wax and wax free oil and diluent are separated into layers, mechanically discharging the wax in substantially solid form from the zone in which it is subjected to centrifugal force at one point in a continuous manner with movement toward the axis of rotation and continuously discharging the wax free oil and diluent from said zone with movement toward the axis of rotation at another point.

22. The improvement in the treatment of petroleum oils for the removal of paraffin wax contained therein, comprising subjecting the oil to a cracking treatment to convert at least a portion of the wax to a crystalline form, subsequently chilling the oil in the presence of a low boiling naphtha and subjecting the chilled mixture to centrifugal force whereby the wax and wax free naphtha-oil solution are separated into layers, mechanically discharging the wax in substantially solid form from the zone in which it is subjected to centrifugal force at one point in a continuous manner with movement toward the axis of rotation and continuously discharging the wax free naphtha-oil solution from said zone with movement toward the axis of rotation at another point.

In testimony whereof we affix our signatures at Stockholm, Sweden, this 25th day of April, 1924.

HANS OLOF LINDGREN.
KNUT GUSTAF MALM.