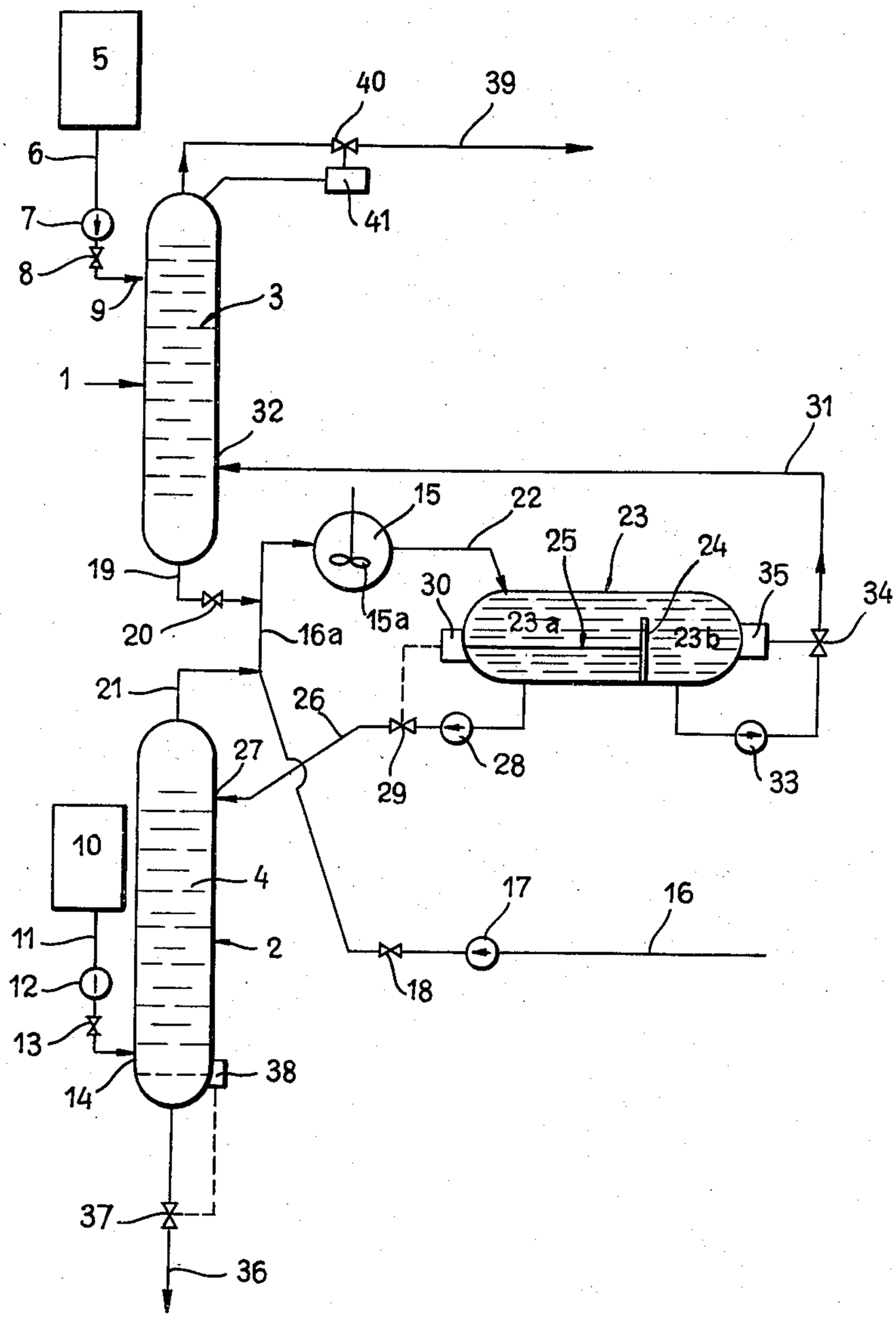
Sept. 20, 1960 T. MIGNONE 2,953,501

APPARATUS FOR EXTRACTION BY THE DOUBLE SOLVENT METHOD

Filed Jan. 22, 1958



United States Patent Office

2,953,501

Patented Sept. 20, 1960

2,953,501

APPARATUS FOR EXTRACTION BY THE DOUBLE SOLVENT METHOD

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Filed Jan. 22, 1958, Ser. No. 710,496

Claims priority, application Italy July 18, 1957

2 Claims. (Cl. 196—14.52)

This invention relates to the technique of separation of homogeneous liquid mixtures of two components by the use of two solvents, and more specifically to the extraction by the use of two solvents of a mixture of mineral oils of which one component consists of paraffin oils and the other of aromatic i.e. non-paraffin oils. It is to be understood, however, that, selecting suitable solvents, the invention may also be applied to other homogeneous mixtures (solutions) as for example a mixture of acetone-acetic acid (using chloroform as a solvent for the acetone and water as a solvent for the acetic acid), or a mixture of parachloronitrobenzol - orthochloronitrobenzol (in which the first is dissolved with heptane and the latter with methane). This specification, however, is particularly concerned with mineral lubricating oils.

As is well-known it is frequently necessary in the production of lubricating oils to separate the paraffin hydrocarbons from the aromatic and asphaltic hydrocarbons, and as the viscosity of the former is much less affected by temperature than is the case with the latter, paraffin oils present far superior properties as lubricants.

The term "paraffin" as used in this connection is not to be interpreted literally, but is intended to imply a phase composed of hydrocarbons in which aliphatic chains largely preponderate; the same applies by analogy to the terms "aromatic" and "asphaltic" respectively.

The separation of a crude oil into its "paraffin" and "non-paraffin" phases is effected industrially by means of solvent extraction operations. A large number of chemical compounds are known to be suitable solvents, each under certain conditions of application and for specific types of operations. Some of these solvents have a highly polar molecule e.g. phenol, cresols, furfural, sulphurous acid, nitrobenzol and aniline: these solvents present good solubility with non-paraffin hydrocarbons, but are barely soluble in paraffin hydrocarbons. A crude oil, if mixed with one of these solvents, will separate out into a layer composed of the solvent with incorporated non-paraffin hydrocarbons, and a layer composed of paraffin hydrocarbons containing a small quantity of the solvent.

In the case of certain crude oils, e.g. all oils rich in asphaltic products and composed of vacuum distillation residues, treatment with a single solvent (as referred to above) is no longer adequate, as it is first necessary to eliminate the asphalt content of the oil. This is generally done by precipitating out the asphalt with a mixture of liquefied propane and propylene.

According to the method as practised at the present time, a crude oil containing asphaltic components is first subjected to treatment with propane to remove the asphalt, and then to successive extraction with one of the solvents mentioned above for the separation of the paraffin oils on the one hand, and of the aromatic oils on the other. As will be seen in the following, the yields resulting from such a process are unsatisfactory, and one of the principal purposes of this invention is to provide a process and apparatus affording better yields.

Another disadvantage of the processes employed up till

now resides in the fact that neither the refined product (paraffin oils) nor the extract (non-paraffin oils) are obtained in a sufficiently pure state and their coloration reveals the existence of impurities that can be eliminated only by the use of decolorising earths. Accordingly, another purpose of the invention is to provide means for remedying these disadvantages.

A further purpose of this invention is to simplify the technique of extraction by the double solvent method, independently of the type of the crude mixture to be separated into its components, so as to afford a process and apparatus of general application.

According to one form, the present process for the extraction of a homogeneous mixture of two liquid components by means of two solvents is principally characterised by the operations of: providing a refining zone and an extraction zone, both extending in a substantially vertical direction and each capable of bringing about a descending flow of one liquid in contact with a rising flow of a second liquid having a specific gravity lower than that of the first liquid, feeding in a refining solvent at an upper part of the refining zone and producing a descending flow of the said refining solvent through the said refining zone; feeding in an extraction solvent at a lower part of the extraction zone and producing a rising flow of the said extraction solvent through the said extraction zone: taking from the bottom of the refining zone the bottom product of such zone, taking from the top of the extraction zone the top product of such zone and mixing the said bottom and top products with the aforementioned homogeneous liquid mixture to obtain a non-homogeneous liquid mixture; decanting by gravity the non-homogeneous mixture, separating it into a heavy phase and a light phase; feeding in the light phase at a lower part of the refining zone to produce a rising flow of the light phase through the refining zone in refining contact with the descending flow of the refining solvent: feeding in the heavy phase at any upper part of the extraction zone to produce a descending flow of the heavy phase through the extraction zone in contact with the rising flow of the extraction solvent; taking from the top of the refining zone a product predominantly consisting of one component of the aforementioned homogeneous mixture and of the extraction solvent, and taking from the bottom of the extraction zone a product predominantly consisting of a second component of the aforementioned homogeneous mixture and of the refining solvent.

According to a further form, applied to a homogeneous mixture of mineral oils, the process according to the invention is characterised by the operations of: providing a refining and an extraction zone, both extending in a substantially vertical direction and each capable of bringing about a descending flow of one liquid in contact with a rising flow of a second liquid having a specific gravity lower than that of the first liquid; feeding in at an upper part of the refining zone a refining solvent, capable of dissolving the non-paraffin oils, and producing a descending flow of the said refining solvent through the refining zone; feeding in at a lower part of the extraction zone an extraction solvent capable of dissolving the paraffin oils, and producing a rising flow of the said extraction solvent through the extraction zone; taking from the bottom of the refining zone the bottom product of such zone, taking from the top of the extraction zone the top product of such zone, and mixing the said bottom and top products with the homogeneous mixture of paraffin oils and non-paraffin oils to obtain a non-homogeneous liquid mixture comprising a light phase and a heavy phase; decanting by gravity this non-homogeneous mixture, separating the said light and heavy phases; feeding in the light phase at a lower part of the refining zone to produce a rising flow of the light phase through the

refining zone in refining contact with the descending flow of the refining solvent; feeding in the heavy phase at an upper part of the extraction zone to produce a descending flow of the heavy phase through the extraction zone in contact with the rising flow of the extraction solvent; taking from the top of the refining zone a product predominantly consisting of the paraffin oils and of the extraction solvent; and taking from the bottom of the extraction zone a product predominantly consisting of the non-paraffin oils and of the refining solvent.

Furthermore, according to this invention, an apparatus for the extraction of a homogeneous mixture of two liquid components by means of two solvents is characterised by the feature of comprising in combination: a refining column and an extraction column; means in both columns capable of producing an intimate contact between a liquid rising and a liquid descending through the column; means for feeding in a refining solvent at an upper part of the refining column; means for feeding in an extraction solvent at a lower part of the extraction column; a mixing receptacle in communication with a bottom zone of the refining column and with a top zone of the extraction column; means for feeding the aforesaid homogeneous mixture into the mixing receptacle; a separator receptacle capable of being fed from the said mixing receptacle and having a lower zone in communication with an upper zone of the extraction column, and an upper zone in communication with a lower zone of the refining column; and means branching from the top of the refining column and from the bottom of the extraction column for taking from the said columns the refined and extracted products respectively.

Further details of the invention will be evident from the description given below with reference to the accompanying drawing which represents diagrammatically one form of application of the invention for the extraction of lubricating oils.

According to the drawing, the refining column **1** and the extraction column **2** which extend in a vertical direction contain means, **3** and **4**, capable of producing an intimate contact between a descending liquid and a rising liquid respectively. Such means, as far as column **1** is concerned, preferably consists of a series of plates and, as regards column **2**, of a large number of horizontally disposed angular sections, whereby in both columns the descending liquid may be intimately mixed with the rising for the necessary reciprocal transfer of the extracted and refined products respectively, in a manner well-known per se in the art.

A tank **5**, which contains a refining solvent well-known in the art and composed of equal parts of phenol and cresol, is connected by means of a pipe **6**, incorporating a pump **7** and a control valve **8**, with an upper part **9** of column **1** through which the refining solvent aforementioned can be fed into the column under volume and pressure control.

A tank **10** containing an extension solvent comprising liquid propane is connected by means of a pipe **11**, incorporating a pump **12** and a control valve **13**, with a lower part **14** of the extraction column **2** through which liquid propane may be fed into the column under volume and pressure control.

A mixing apparatus **15**, comprising a unit separate from the two columns **1** and **2**, is fed with crude oil through a pipe **16** which incorporates a pump **17** and a control valve **18**. A tube **19** leading from the bottom of the refining column **1** and incorporating a control valve **20** joins pipe **16** at section **16***a* which is located between the valve **18** and the mixer **15**, whereby, subject to appropriate regulation of valves **18**, **20**, the bottom product in column **1** may be brought into the mixer **15** together with the crude oil. Furthermore, the top product in the extraction column **2**, the top of which communicates with section **16***a* of pipe **16** through a tube **21**, is also fed into the mixer **15**.

The mixer **15** is connected by a pipe **22** with a gravity separator, or decanter **23**, of any suitable type. As is well-known in the art, a classical form of separator comprises a bottom zone in which a "heavy" phase is deposited and a top zone in which a "light" phase is collected. The separator shown in the accompanying drawing is of a horizontal type comprising a vertical partition **24** extending from the bottom wall up to a point at close proximity to the upper wall of the separator, so that the interior of the separator is subdivided into two chambers **23***a*, **23***b*, the end of the first chamber, remote from the partition **24**, being fed from the mixer **15** through tube **22**. The heavy phase collects on the bottom of chamber **23***a*, while the light phase passes over the partition **24** and collects in chamber **23***b*, the separation level between the two phases being shown at **25**. Therefore, considered in relation to a vertical separator of the classical type, the chamber **23***b* of separator **23** may be defined as the top zone of the separator and that part of chamber **23***a* situated below the level **25** may be defined as the bottom zone of the separator.

It will further be seen from the drawing that the bottom zone of separator **23** is connected via pipe **26** with an upper part **27** of extraction column **2**, the pipe **26** aforesaid incorporating a pump **28** and a control valve **29**. This valve **29** is automatically controlled by an apparatus **30** which is associated with chamber **23***a* of the separator and sensitive to variations in the interphase level **25**. Apparatus of this kind is well-known in the art and does not therefore call for specific description.

The top zone **23***b* of separator **23** is connected via pipe **31** with a lower part **32** of the refining column **1**. The pipe **31** incorporates a pump **33** and a valve **34** which is automatically controlled by an apparatus **35** sensitive to the pressure obtaining in chamber **23***b*, in such a manner that valve **24** is largely opened or largely closed respectively, according to the increase or diminution, as the case may be, of the pressure in the separator **23**. The extract, comprising in this case non-paraffin oil, is taken from the bottom of the extraction column **2** through a pipe **36**, incorporating a control valve **37**, which is automatically controlled by an apparatus **38** associated with the bottom zone of column **2** and sensitive to variations in the level of the non-paraffin oil which is collected in this zone of column **2**.

Finally, the refined product, comprising in this case paraffin oil, is taken from the top of column **1** through a pipe **39** incorporating a control valve **40** which is automatically controlled by an apparatus **41** associated with the top zone of column **1** and sensitive to the pressure in that zone.

As will be readily understood from this description of the apparatus according to the invention, the apparatus in question necessarily operates under super-atmospheric pressure in order to maintain the propane in the liquid state. It will also be understood that in using solvents which are liquid under ordinary temperature and pressure conditions, the pressures inside the various parts of the apparatus will only be those necessary for maintaining the individual liquid phases in motion, and that they are therefore very substantially different from the pressures required for the liquefaction of propane which may amount to as much as 25–30 atmospheres effective pressure or more.

As already stated above, the crude oil, which consists of a mutual solution of various non-paraffin and paraffin oils, is fed through the pipe **16**. As is the case with all known processes, the purpose of this process is to transform this solution (homogeneous mixture) into a non-homogeneous mixture of two phases and to separate the two phases from one another by decantation. Finally, the solvents are recovered by a process which is known per se, but which is outside the scope of this invention.

In the normal operation of the apparatus here concerned, and considering primarily the two columns **1** and **2**, it is

clear that in each of these columns opposite flows of two liquid phases in contact with one another are stabilized, the lighter phase, comprising propane in which paraffin oils are dissolved, rising to the top of each column, and the heavier phase comprising the phenol-cresol solvent in which non-paraffin oils are dissolved, descending to the bottom. The bottom product of column **1** therefore contains a substantial proportion of phenol-cresol solvent, while the product at the top of column **2** contains a substantial proportion of propane. These two products are taken from the respective columns through pipes **19** and **21**, added to the crude oil in pipe **16***a* and conveyed to the mixer **15**, which as has been shown, constitutes a unit independent of the two columns, and is designed through agitating means **15***a* to effect a vigorous remixture of the contents which are then discharged into the separator **23**. The liquid fed into separator **23** is manifestly composed of a non-homogeneous mixture in which particles or droplets of phenol-cresol solvent with their non-paraffin oil content are finely dispersed among particles or droplets of propane with their content of paraffin oils. By force of gravity, therefore, the heavy phase containing a little paraffin oil will accumulate at the bottom of chamber **23***a*, while the light phase containing a little non-paraffin oil will accumulate in chamber **23***b*. The heavy, or bottom, phase is then conveyed to the top of column **2** through pipe **26**, while the light, or top, phase in the separator **23** is conveyed to the bottom of column **1** through pipe **31**.

The light phase introduced at **32** into the refining column **1** will rise in that column in counter-current to the descending dispersed flow of phenol-cresol solvent, whereby the solvent will remove from the light phase its content of non-paraffin oils, while, on the other hand, the propane contained in the light phase will keep the paraffin oils sufficiently diluted so as not to be dissolved in the descending phase to any remarkable extent. Finally, a product predominantly composed of paraffin oils and propane will be collected at the top of column **1**.

The heavy phase introduced at **27** into column **2** will descend in counter-current to the rising dispersed flow of liquid propane, whereby the latter will remove from the heavy phase its content of paraffin oils. Finally, a product predominantly composed of non-paraffin oils and of phenol-cresol solvent will be collected at the bottom of column **2**.

The bottom product of column **1** is predominantly composed of phenol-cresol solvent which carries along with it comparatively small quantities of non-paraffin oil and propane, while the product at the top of column **2** is predominantly composed of propane which carries along with it comparatively small quantities of paraffin oils and phenol-cresol solvent. These two products are taken from the columns and, as stated above, added to and vigorously remixed with the crude oil in the mixer **15** whereby the entire amount of incoming crude oil in the separator **23** is rapidly and efficiently separated into two rough phases even before entering the columns. In this way each of the two columns operates only on a part of the crude oil, i.e. that part which has been pre-cleansed from the unwanted component. The mixer **15** and the separator **23** may therefore be regarded as an intermediate separation stage, outside the two columns and operating to provide through the pipes **31**, **32** a reflux for column **1**, and through the pipes **26**, **27** a reflux for column **2**. In their turn, the columns **1**, **2** with pipes **19**, **21** respectively, produce a kind of reflux for the mixer-separator group **15**, **23**. The interconnection between this group and the columns is regulable by means of the valves **20**, **29** and **34** incorporated in the pipes **19**, **26** and **31** respectively, so that the entire process of extraction may easily be controlled to afford the most favourable results.

The table below shows the composition in kilo-grammes per hour of the phases at the salient points of the installation, designated at the top of the individual columns by the same reference numerals as those used in the drawing.

Table I

Operation	Position Component	16	14	9	39	36	21	19	27	32
With crude oil type 1	Oil	1,000			281	719	230	170	949	451
	Propane		2,000		1,490	510	2,500	140	1,010	1,630
	Phenol-Cresol solvent			1,500	225	1,275	270	1,540	1,545	265
	Density at 15° C	1.003	0.508	1.050	0.576	0.863	0.553	0.92	0.800	0.595
With crude oil type 2	Oil	1,000			342	658	235	315	893	657
	Propane		2,000		1,640	360	2,300	230	660	1,870
	Phenol-Cresol solvent			1,000	350	750	290	1,210	1,040	460
	Density	0.998	0.508	1.050	0.592	0.865	0.549	0.916	0.811	0.619
With crude oil type 3	Oil	1,000			552	448	438	390	886	942
	Propane		1,800		1,510	290	2,080	110	570	1,620
	Phenol-Cresol solvent			1,800	350	1,450	320	1,740	1,770	290
	Density	0.931	0.508	1.050	0.619	0.915	0.681	0.980	0.870	0.631

From this table it will be seen, for example, that in the case of operation with crude oil of type 1 there are obtained 281 liters per hour of paraffin oil leaving the top of column **1** through the pipe **39** and 719 liters per hour of non-paraffin oil leaving the bottom of column **2** through the pipe **36**.

The characteristics of the crude oil, paraffin oil and non-paraffin oil respectively in relation to the table given above will be seen from the Table II below in which the columns marked A refer to extraction according to the present process and the columns marked B refer to one of the processes ordinarily employed, comprising a preliminary asphalt-removal phase using propane, followed by extraction with phenol, while the column marked C refers to the treatment of crude oil type 3 in an ordinary extraction plant using phenol. It should be noted that the crude oil type 3 is a vacuum-distilled oil.

In the case of all the operations conducted in the apparatus as described above, all the valves shown in the diagram were so regulated as to afford the following internal pressures:

	Atm. effective pressure
At top of column **1**	24.5
At bottom of column **1**	26.0
At top of column **2**	23.0
At bottom of column **2**	24.0
At separator **23**	22.0

The temperature at which the process takes place ranges between 20° and 50° C., and amounts on the average to about 35° C.

The advantages of the invention will be immediately evident upon comparing columns B and C with columns A in Table II for each type of crude oil dealt with, and there is therefore no need to emphasize them further here.

It is also obvious that the invention is in no way restricted to the embodiment here described with reference to the accompanying drawing, but that various alternative modifications within the limits of technical equivalent may be applied thereto without exceeding the scope of the invention as defined in the following claims.

Table II

	Operation with crude oil type 1		Operation with crude oil type 2		Operation with crude oil type 3	
	A	B	A	B	A	C
Crude oil:						
Supply kg. per hr.	1000	1000	1000	1000	1000	1000
Density at 15° C.	1.0030	1.0030	0.9980	0.9980	0.9315	0.9315
Viscosity at 210° F. in centistokes	365	365	280	280	11.35	11.35
Paraffin oil:						
Supply kg. per hr.	281	218	342	273	552	425
Percentage yield by weight	28.1	21.8	34.2	27.3	55.2	42.5
Density at 15° C.	0.8990	0.8985	0.9050	0.9049	0.8652	0.8654
Viscosity at 100° F. in centistokes	408	421	464	473	--------	--------
Viscosity at 210° F. in centistokes	28.3	28.3	29.4	29.4	9.35	9.35
Viscosity index	102	100	97	96	115	115
Viscosity index after deparaffination	100	98.5	95	94	--------	--------
Carbonic residue acc. to Conradson (percentage by weight)	0.4	0.5	0.4	0.55	--------	--------
Color (Union)	3½	5	--------	--------	--------	--------
Non-paraffin oil:						
Supply kg. per hr.	719	782	658	727	448	575
Percentage yield by weight	71.9	78.2	65.8	72.7	44.8	57.5
Density at 15° C.	1.052	1.036	1.058	1.040	1.026	0.9861
Viscosity at 210° F. in centistokes	1700	850	1750	930	18.1	15.9

What I claim is:

1. An apparatus for the extraction by means of two solvents of a homogeneous mixture of two liquid components comprising in combination: a refining column and an extraction column; means in both columns for producing intimate contact between a liquid rising and a liquid descending through said columns; means for feeding a refining solvent into an upper part of the refining column; means for feeding an extraction solvent into a lower part of the extraction column; a mixing receptacle in communication with a bottom zone of the refining column and with a top zone of the extraction column; means

for feeding the aforementioned homogeneous mixture into the mixing receptacle; a separator receptacle connected to be fed from said mixing receptacle and having a lower zone in communication with an upper zone of the extraction column and an upper zone in communication with a lower zone of the refining column; and means branching from the top of the refining column and from the bottom of the extraction column for taking from the said columns the refined and extracted products respectively.

2. An apparatus for the extraction by means of two solvents of a homogeneous mixture of two liquid components comprising in combination: a refining column and an extraction column; means in both columns for producing intimate contact between a liquid rising and a liquid descending through said columns; means for feeding a refining solvent into an upper part of the refining column; means for feeding an extraction solvent into a lower part of the extraction column; a mixing receptacle in communication with a bottom zone of the refining column and with a top zone of the extraction column; means for feeding the aforementioned homogeneous mixture into the mixing receptacle; a separator receptacle connected to be fed from said mixing receptacle and having a lower zone in communication with an upper zone of the extraction column and an upper zone in communication with a lower zone of the refining column; valve means associated with the upper zone of the separator receptacle sensitive to pressure prevailing in the separator thereby to control light phase flow from said upper zone of the separator to the refining column; valve means associated with the separator receptacle sensitive to the phase separation level in the receptacle to control heavy phase flow from the lower zone of the separator to the extraction column, and means branching from the top of the refining column and from the bottom of the extraction column for taking from the columns the refined and extracted products, respectively.

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