

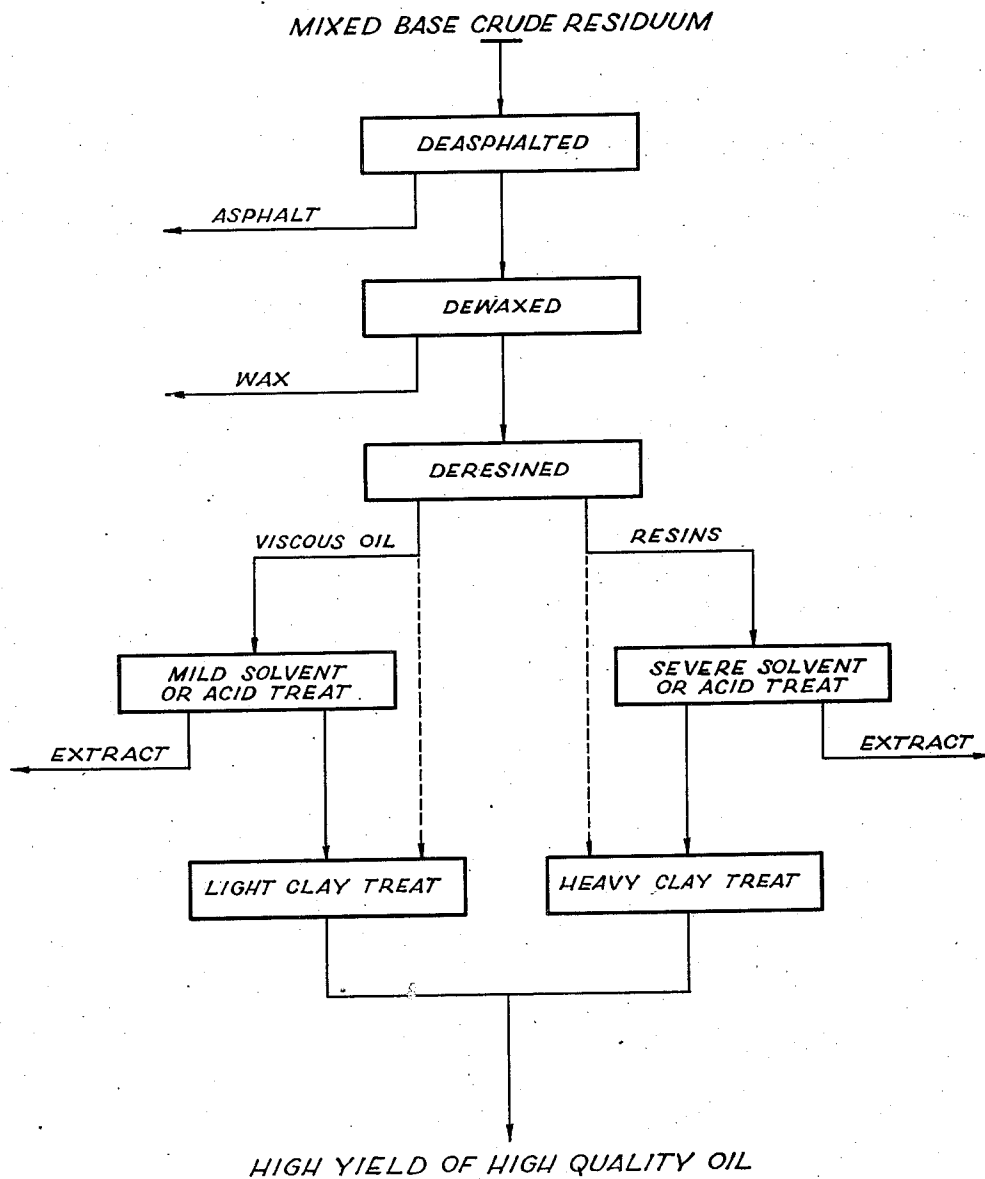
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REFINING OF HEAVY OILS

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REFINING OF HEAVY OILS

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This invention relates to the refining of heavy oils, and it pertains more particularly to an improved combination process for obtaining maximum yields of high quality lubricating oils from residual or resin-containing lubricating oil stocks.

The object of the invention is to provide a new and improved combination of refining steps for the processing of residual stocks or heavy distillate stocks containing an appreciable quantity of resins, whereby maximum yields of high quality lubricants are obtained, which lubricants are characterized by stability against oxidation, discoloration, sludge formation and carbon depositing tendencies, and which lubricants are further characterized by a high viscosity index (V. I.) and a low viscosity gravity constant (V. G. C.). A further object is to produce maximum yields of a heavy lubricant in which the Conradson carbon, or carbon residue is less than 1% of the Saybolt viscosity of the lubricant at 210° F. A further object is to provide a more efficient and effective refining system than has heretofore been available, and to correlate the steps of the processes and the treating conditions in each respective step, so that at no stage in the process will there be a loss of valuable lubricating oil. A further object is to correlate and integrate a lubricating oil refining process so that each step of the process may be carried out at minimum expense and with maximum efficiency, and so that the combination of steps results in larger yields and higher qualities than can be obtained by any process or combination of processes heretofore known.

A further object of the invention is to provide a refining process for heavy oils in which the by-products consist essentially of high melting point asphalts, low gravity extracts or combinations thereof, and wax. The amount and quality of material rejected from the process is one measure of the efficiency of the refining operations and the object of the invention is to obtain maximum efficiency.

Known refining processes for heavy lubricating oil stocks include propane deasphalting, propane deresining, propane fractionation for separating high molecular weight from low molecular weight components, solvent extraction, acid treating, clay treating, etc., and applicant claims no novelty in any of these particular processes per se. The present invention correlates these

processes in a novel manner and interrelates the operating conditions in the several processes and the stocks charged thereto so that the combination of processes leads to results which could not be accomplished by any combination of the processes heretofore used. In other words, the operating conditions in each process are dependent upon the operating conditions in other processes and the stocks charged to and produced by said other processes. Overall savings in amounts of treating agents and in treating costs are thereby effected.

It is recognized in the industry that separate treatment of light distillates and residuals is advantageous for obtaining maximum yields and equal product quality from both stocks. It has been generally considered that a residual stock, the lubricating oil content of which is normal bright stock, is one fraction and the lighter distillates are the other fractions. One object of this invention is to adapt the step of propane deresining to separate the residual stock itself into two fractions, one having a viscosity of less than 300 S. S. U. at 210° F. and the other being a heavier fraction. By subsequent severe treatment of the heavier, or resin, fraction a high quality, high viscosity lubricating oil can be obtained. This material, per se, is a new product of high viscosity, but even more important is the fact that it may be blended with the less viscous fraction to give a product in which the fractions of different viscosities have separately controllable quality. It has been the usual procedure to treat the entire cylinder stock as one fraction with inadequate quality improvement on the heavier portion, but the step of propane deresining, followed by severe treatment of the heavier and more viscous fraction, enables the refiner to obtain any desired quality on both this fraction and the less viscous oils.

Heretofore refiners have endeavored to minimize the amount of resins produced because they were not deemed suitable as components of high quality lubricants. In practicing my invention either a small or a large resin fraction is purposely separated from a heavy lubricating oil stock, either in the presence or absence of asphaltic materials, and this resin fraction is separately refined by acid treating, solvent extraction, clay treating or by a combination of two or more of

said processes, so that extremely valuable components are separated from these resins for blending with the deresined lubricant. The acid treating and the claying of the resins may have to be effected in the presence of a light gasoline or naphtha diluent since the resins are of such high molecular weight materials that they cannot be treated by the corresponding conventional propane processes.

Phenol is the preferred solvent for extraction. Heretofore phenol has been considered unsuitable for the refining of resins which are in fact the heaviest portion of the ordinary residual stocks, but I have discovered that under severe conditions, i. e. high temperature and large solvent ratios, phenol extraction is not only adequate but exceptionally effective. The invention is not limited to the use of phenol, however, since other solvents such as cresylic acid, cresols, chlorphenols, mixtures of such phenolic solvents with small amounts of water, dichlorethyl ether, furfural, nitrobenzene, etc. may be used either on the resins or on the deresined oil, as will be hereinafter described.

In practicing the invention the heavy residual or resin-containing stock is first fractionated on the basis of molecular weight to give a viscous oil fraction having a viscosity less than 300 seconds Saybolt at 210° F. and a resin fraction having a viscosity of about 300 seconds Saybolt to 1000 seconds Saybolt at 210° F. This initial fractionation is important because it removes from the resin fraction certain viscous oils which, if allowed to remain with the resins, would be severely over-treated with a consequent loss in yield under the conditions required for adequate solvent extraction, acid treating or clay treating of said resin fractions. The resin fraction is then acid-treated, extracted with phenol or clay treated under severe conditions, while the separated viscous oil may require little or no subsequent refining. A mild treatment of the viscous oils, as distinguished from the heavy treatment of the resin fraction, prevents the destruction of large amounts of the viscous oil.

An inherent and important advantage of my process lies in the fact that the blending of the separately clay treated resins with the viscous cylinder stock produces a final product characterized by a highly desirable cast. The general appearance and cast of a bright stock is sometimes impaired during the acid treating or solvent extraction thereof and the problem of preserving or obtaining the desired cast has baffled oil refiners for years. I have found that by separately clay treating the resins in accordance with my invention and then blending the separately clay treated resins with a bright stock having poor cast characteristics the finished product is characterized by an excellent cast with an excellent stability.

The drawing which is annexed hereto and which forms a part of this specification shows a flow diagram of my improved system for refining resin-containing lubricating oil stocks, the details of the several steps thereof being set forth in the tables which also form a part of this specification.

The invention is not limited to any particular type of crude, although it is particularly useful in connection with residuums from Pennsylvania and mixed base (Mid-Continent) crudes. The invention is only applicable, however, to the refining of heavy oils which contain viscous high molecular weight compounds which are com-

monly referred to as resins. I have found that when residual oils in the cylinder stock range are fractionated with propane, that fraction having a viscosity below about 290 to 300 seconds Saybolt at 210° F. is substantially free from resins, while the fraction ranging in viscosity from about 300 to 1000 seconds Saybolt at 210° F. contains large amounts of the resins. The first step of my process consists in fractionating the heavy residual stock (which may have been previously dewaxed) into a viscous oil fraction (about 50 to 300 viscosity at 210° F.) and a resin fraction (about 300 to 1000 viscosity at 210° F.), the fractionation preferably being effected with a 10:1 propane-oil solution at a temperature of about 180° F. It should be understood, however, that this propane fractionation may be effected with various propane ratios and at various temperatures, depending on the particular stock undergoing treatment, the essential feature of this step being the separation of viscous oil from resin fractions, as herein defined. While propane deresining is my preferred method of separating resin fractions from viscous oil, it should be understood that any other fractionating may be employed that will produce the desired separation.

The viscous oil fraction in the case of Pennsylvania oils may require no further treatment whatsoever. In the case of mixed base crudes the viscous oil fraction may be given a light acid treatment but it is preferably extracted with a selective solvent such as phenol under mild operating conditions, i. e. at temperatures below 200° F. and at solvent ratios from about 1:1 to 3½:1. It is desirable to dewax this deresined oil prior to phenol treating when the dewaxing is effected in the absence of viscous oil components initially removed therefrom in the original fractionation step.

The resin fraction in the case of Pennsylvania or Pennsylvania type stocks is given a severe acid treatment or solvent refining treatment such as extraction with phenol at temperatures higher than 200° F. and solvent ratios of about 2:1 to 4½:1. With these high solvent ratios and high treating temperatures an extract may be removed having an A. P. I. gravity of about 8 or 9 and a raffinate can be obtained which has an A. P. I. gravity of about 23.5 and a V. G. C. of about .810 to .816.

After the raffinate is freed from solvent it may be percolated in naphtha solution through Attapulugus clay or other clay equivalent thereto at a temperature of about 180° F. The clay yields may range from about 3 to 5 barrels per ton, but since it is only the resin fraction or resin raffinate which has to undergo this clay treatment, a considerable savings in clay is effected. The overall clay yield in one example of my process was about 26 barrels of blended oil per ton of clay. If the oil had been percolated through clay before fractionation, in accordance with processes heretofore used, the clay yield would have been less than 11 barrels per ton. Since clay treating is the most expensive step in the refining of these viscous lubricating oils, the enormous savings effected by the use of this invention will be obvious.

It would be expected that the use of such a small amount of clay would result in a blend of doubtful color stability, oxidation stability and sludge and carbon forming tendencies. The fact is, however, that the finished blend is characterized by excellent color and color stability. The

blend is stable against oxidation and sludge-forming and it has a carbon residue which is less than 1% of the viscosity of the finished blend at 210° F. The efficiency of the process is evidenced by the fact that the only materials removed from the oil are the low gravity extract (about 8° or 9° A. P. I.) and a 116° to 117° F. melting point wax.

I have shown in Table I a summary of the process applied to Allegheny cylinder stock, a heavy residuum from a dark Pennsylvania crude. In this case it will be noted that the separated asphalt and resins were blended prior to the

phenol treating step, and that after clay percolation of the phenol raffinate of the resin fraction, the blend with deresined oil gave a yield of 92.1% with an A. P. I. gravity of 26.2, a viscosity at 210 of 167, a carbon residue of 1.47% and a V. G. C. of .809. On dewaxing the yield was reduced to 78% with a gravity of 25.3; the viscosity was increased to 176.7, the carbon residue to 1.63 and the V. G. C. to 1.814.

In Fig. 1 the letters "T. R." refer to Tag Robinson color, "C. R." indicates carbon residue and "S. P." stands for solid point. The remaining indicia are believed to be self-explanatory.

TABLE I

PROCESSING ALLEGHENY CYLINDER STOCK	
°API gravity.....	24.4
SSU Vis. 210° F.....	201
Color, T. R.....	1 1/4 DD
Car. res. percent.....	3.7
V. G. C.....	.818

PROPANE DEASPHALTING AND DERESINING	
Vol. percent C ₃ to asph. settler.....	1000
Vol. percent C ₃ to resin settler.....	500
Vol. percent C ₃ to resin washer.....	—
Temp. °F. asph. settler.....	120
Resin settler.....	180
Resin washer.....	—

Deresined oil	Resins	Asphalt
Vol. percent..... 68.0	Vol. percent..... 25.9	Vol. percent..... 6.1
°API..... 26.6	°API..... 21.7	°API..... 14.0
V/210..... 119.6	V/210..... 521	
T. R..... 1	T. R..... 300	
C. R. percent..... 1.20	C. R. percent..... 5.3	
V. G. C..... .812	V. G. C..... .827	

Blend	
V/210.....	712
C. R., percent.....	8.1

PHENOL TREATING	
Vol., percent solvent.....	345
Temp. °F. top.....	241
Btm.....	171
Vol., percent water injected.....	9.4

Raffinate	Extract
Vol., percent... 75.3 (24.1% based on charge)	Vol., percent... 24.7 (7.9% based on charge)
°API..... 23.8	°API..... 9.1
V/210..... 569	
C. R., percent... 4.2	
V. G. C..... .810	

CLAY PERCOLATION WITH NAPHTHA AT 180° F.	
Yield B/T.....	3.0
°API.....	25.5
V/210.....	469
T. R.....	1 1/4
C. R., percent... 1.90	
V. G. C.....	.799

Blend (92.1% based on charge)	
°API.....	26.2
V/210.....	167.0
T. R.....	1 1/4
C. R., percent... 1.47	
V. G. C.....	.809

PROPANE DEWAXING	
Vol., percent C ₃	300
Filt. temp. °F.....	-41

Dewaxed oil	Wax
Vol., percent... 84.7 (78.0% based on charge)	Vol., percent... 15.3 (14.1% based on charge)
°API..... 25.3	M. P. °F..... 116
V/210..... 176.7	
T. R..... 1	
C. R., percent... 1.63	
S. P. °F..... 0	
V. G. C..... 1.814	

Table II gives a detailed sketch of a similar refining process on the same stock, wherein the asphalt fraction was not blended with the resin fraction, but was removed from the process as such. It will be noted that the removal of the asphalt caused a considerable reduction in the volume of the extract from the extraction stage and caused a slight decrease in the yield of final dewaxed oil.

1.0537, a melting point of 147° F. and constituted 28.5% of the stock charged.

The resin fraction which constituted 25.2% of the stock charged was extracted with phenol under severe conditions, i. e. a temperature up to 211° F. and a solvent ratio of about 4:1. This resulted in the elimination of slightly more than half of the resin fraction (13.2% of the stock charged) as an extract whose API gravity was

TABLE II

PROCESSING ALLEGHENY CYLINDER STOCK	
°API gravity.....	24.4
SSU Vis., 210° F.....	201
Color, T. R.....	1 1/4 DD
Car. res., percent.....	2.7
V. G. C.....	.818

PROPANE DEASPHALTING AND DERESINING	
Vol. percent C ₃ to asph. settler.....	1000
Vol. percent C ₃ to resin settler.....	—
Vol. percent C ₃ to resin washer.....	500
Temp. °F. asph. settler.....	120
Resin settler.....	180
Resin Washer.....	100

Deresined oil	
Vol. percent.....	65.6
°API.....	26.6
V/210.....	113.8
T. R.....	2 3/4
C. R., percent.....	1.20
V. G. C.....	.812

Resins	
Vol. percent.....	28.4
°API.....	21.5
V/210.....	508
T. R.....	2 1/4 DD
C. R., percent.....	5.2
V. G. C.....	.829

Asphalt	
Vol. percent.....	6.0
°API.....	14.5

PHENOL TREATING	
Raffinate	Extract
Vol. percent... 88.8 (25.2% based on charge)	Vol. percent... 11.2 (3.2 based on charge)
°API..... 23.4	°API..... 8.0
V/210..... 451	
T. R..... 3 DD	
C. R., percent. 3.9	
V. G. C..... .816	

CLAY PERCOLATION WITH NAPHTHA AT 180° F.	
Yield B/T.....	4.7
°API.....	24.4
V/210.....	385
T. R.....	1 1/4
C. R., percent.....	2.33
V. G. C.....	.810

Blend (90.8% based on charge)	
°API.....	26.1
V/210.....	157.9
T. R.....	1 1/4
C. R., percent.....	1.65
V. G. C.....	.810

PROPANE DEWAXING	
Vol. percent C ₃	300
Filt. temp. °F.....	37

Dewaxed oil	
Vol. percent... 85.3 (77.4% based on charge)	Vol. percent. 14.7 (13.4% based on charge)
°API..... 25.1	M. P. °F..... 117
V/210..... 166.0	
T. R..... 1 1/4	
C. R., percent. 1.50	
SP °F..... -2	
V. G. C..... .817	

Wax	
Vol. percent. 14.7 (13.4% based on charge)	
M. P. °F..... 117	

The application of my process to a mixed base crude is illustrated in Table III which shows the processing of a 7% Burma residuum. In this case the stock was first deasphalted with a 4:1 propane ratio at a temperature of 150° F. (washing temperature 155° F.) and the deasphalted oil was then deresined with a 2.1 propane ratio at a temperature of 180° F. (washing temperature 140° F.) with an additional 20% of propane. The separated asphalt had a specific gravity of

9.1. The raffinate, after removal of solvent, was propane dewaxed for the removal of 61.6% of wax (7.4% based on stock charged). This left 4.6% of the stock charged as a high quality oil to be combined with the viscous oil fraction.

The deresined oil was propane dewaxed and then phenol-treated under relatively mild conditions (with a top temperature of 176° F. and a solvent ratio of 3.23:1). The finished raffinate was blended with the high quality oil recovered

from the resins to yield a final blend of very high quality lubricating oil. Thus this relatively low grade residual stock is converted into 28.5% asphalt, 31.6% wax, 21.6% of very low gravity extract and 18.3% of high quality lubricating oil.

ditions which are required for the viscous oil. By fractionating with propane to separate an oil with a viscosity of about 50 to 300 seconds Saybolt at 210° F., and a resin fraction with a viscosity of about 300 to 1000 seconds Saybolt at

TABLE III

PROCESSING BURMAH 7% RESIDUUM			
°API gravity.....	17.3	SSU Vls., 210° F.....	235
		V. G. C.....	.874
PROPANE DEASPHALTING AND DERESINING			
Deresined oil		Asphalt	Resins
Vol. percent.....	46.3	Vol. percent.....	25.2
°API.....	24.8	Spec. grav.....	1.0537
V/210.....	87.9	M. P., °F.....	147
T. R.....	54D		
C. R., percent.....	2.16		
V. G. C.....	.831		
Propane dewaxing		Phenol treating	
Vol. percent.....	300	Vol. percent solvent.....	402
Filt. Temp. °F. Ave.....	-43	Temp. °F. top.....	211
		Btm.....	169
		Water injected percent.....	10.0
Dewaxed oil (22.1% of charge)		Raffinate	Extract
°API.....	18.7	Vol. percent—47.6 (12.0% of charge)	Vol. percent—52.4 (13.2% of charge)
V/210.....	183.8	°API.....	32.1
V/150.....	932	V/210.....	81.8
T. R.....	314D	T. R.....	2D
C. R., percent.....	2.80	C. R., percent.....	0.68
S. P., °F.....	18		
V. I.....	27.1		
V. G. C.....	.886		
Phenol treating		Propane dewaxing	
Vol. percent solvent.....	323	Volume, percent O ₂	300
Temp. °F. Top.....	176	Filt. temp. °F. Ave.....	-42
Btm.....	145		
Water injected, percent.....	9.8		
Raffinate		Dewaxed oil	Wax
Vol. percent.....	62.0 (13.7% of charge)	Vol. percent.....	38.4 (4.6% of charge)
°API.....	28.6	°API.....	27.9
V/210.....	28.6	V/210.....	123.3
V/100.....	11.76	V/100.....	1719
T. R.....	234	T. R.....	7D
C. R., percent.....	0.29	C. R., percent.....	0.62
S. P., °F.....	-4	S. P., °F.....	14
Flash.....	545	V. I.....	90.0
Fire.....	620	V. G. C.....	.801
V. I.....	92.2		
V. G. C.....	.801		
Extract		Final Blend	
Vol. percent—38.0 (8.4% of charge)	°API.....	74.9 vol. percent	25.1 vol. percent (18.3% of charge)
	4.9	°API.....	28.6
		V/210.....	101.7
		V/100.....	1327
		T. R.....	14
		C. R., percent.....	0.37
		S. P., °F.....	0
		V. I.....	93.2
		V. G. C.....	.800

From the examples hereinabove set forth it will be seen that my process is applicable to widely different types of viscous crude residuums or viscous oils containing resins. An important feature of the invention is the proper balance between the amount of resins removed from such a stock and the degree of treatment to which these removed resins are subjected. If the resin fraction is too large (i. e. if it contains too much of the viscous oil), then the overall yield will be low when sufficiently severe treating conditions are used to properly treat the heavy resins. If the resin fraction is too small (i. e. if some of the resins are separated with the viscous oil) the heavy fraction is not adequately treated and the color of the treated oil is not satisfactory, clay consumption is too great or the overall yield is impaired because of the more severe treating con-

210° F., it is possible to obtain maximum yields of high quality oils with minimum treating costs. An outstanding characteristic of the finished oils is their good color and color stability and their low carbon residue which is less than 1% of their viscosity at 210° F.

The specific effect of my method of processing on the quality of the heavy resin fraction of the cylinder stock is shown by the results in Table IV obtained by processing the Allegheny residuum of Table I. The residuum was treated with 163% of phenol using a tower top temperature of 202° F. to produce a raffinate of .804 V. G. C. and 1.98 carbon residue. This raffinate was then deresined with 6.2 volumes of propane at a temperature of 148° F. to produce a resin fraction corresponding to 21.1% of the residuum and having a viscosity of 357 S. S. U. at

210° F., a carbon residue of 4.7 and a V. G. C. of .812. As shown in Table I, the comparable product from my process of deresining followed by phenol treating of the resins corresponded to 24.4% of the residuum and had a viscosity of 569 S. S. U. at 210° F., a carbon residue of 4.2 and a V. G. C. of .810. It will be noted that although the yield and viscosity are both considerably higher by my process, both the carbon residue and the V. G. C. are even better than for the alternate process. If the alternate product were reduced to give a viscosity of 569 S. S. U. at 210° F., the yield would be still lower and the carbon residue and V. G. C. even worse in relation to my product. These data illustrate the effectiveness of my method of processing on the quality of the resin fraction in the finished oil.

of 1000 seconds Saybolt at 210° F. from a Bradford cylinder stock were treated with a 3.4:1 phenol ratio at 240° F. in a continuous tower to yield 24% of an 8.8 gravity extract and 76% of a 23.1 gravity oil having a viscosity at 210° F. of 768, a carbon residue of 3.5% and a V. G. C. of .810. This raffinate when blended with the deresined cylinder stock markedly improved the yield of low V. G. C. oil.

I have already referred to the improved clay yields obtainable by separately claying the resin fraction into which substantially all of the color bodies are concentrated. To specifically illustrate this process, a partially refined 26.2° A. P. I. gravity lubricating oil having a viscosity of 148 seconds Saybolt at 210° F. and a Tag Robinson color of 2 dilute was separated by means of pro-

TABLE IV

EFFECT OF SEPARATE TREATMENT ON QUALITY OF HEAVY FRACTION

Allegheny cylinder stock		
°API gravity.....	24.4	
SSU vis., 210° F.....	201	
V. G. C.....	.818	
Phenol treating		
Volume, percent solvent.....	163	
Temp. °F., top.....	202	
Temp. °F., bottom.....	175	
Vol. percent water injected.....	10.3	
Propane deresining		
Phenol treating of resins (See Table I)		
Raffinate		
Vol. percent.....	86.9	
°API.....	28.7	
V/210.....	180.5	
C. R., percent.....	1.98	
V. G. C.....	.804	
Extract		
Vol. percent.....	13.1	
°API.....	10.7	
Propane deresining		
Vol. percent C ₃	620	
Temp., °F.....	148	
Deresined oil		
Vol., percent.....	75.6 (65.7 based on charge)	
°API.....	27.4	
V/210.....	141.2	
C. R., percent.....	1.22	
V. G. C.....	.802	
Resins		
Vol., percent.....	24.4 (21.2% based on charge)	
°API.....	24.6	
V/210.....	357	
C. R., percent.....	4.7	
V. G. C.....	.812	
Resin raffinate		
Vol., percent.....	24.1 (based on charge)	
°API.....	23.1	
V/210.....	669	
C. R., percent.....	4.2	
V. G. C.....	.810	

While phenol extraction has been described in my preferred examples, it should be understood that acid treating in propane or naphtha solution may be advantageous under certain circumstances. It should also be understood that other selective solvents than phenol may be used. The particular operating conditions for the acid treating or solvent extraction can readily be determined by preliminary experiments, but in all cases they will be more severe on the resin fraction than they would on the type of oils for which said processes have heretofore been employed. A feature of this invention is the use of these oil refining processes on resins under more severe conditions than they have heretofore been used in connection with heavy oils. It is difficult if not impossible to obtain high V. I. oils with reasonable yields by the solvent extraction of these heavy cylinder stocks, but by fractionating these cylinder stocks and treating the resins under more severe conditions than the oil fraction, I have succeeded in not only obtaining the desired high V. I.'s but have also obtained unexpectedly high yield V. G. C. relationships.

For instance, dewaxed resins having a viscosity

pane at temperatures of 160° and 145° F. into a light oil having the following characteristics:

A. P. I. gravity..... 27.3°
 Viscosity at 210° F..... seconds... 115.3
 Tag Robinson color..... 1 1/4
 Vol. percent of charge..... 86.3

and a heavy oil having the following characteristics:

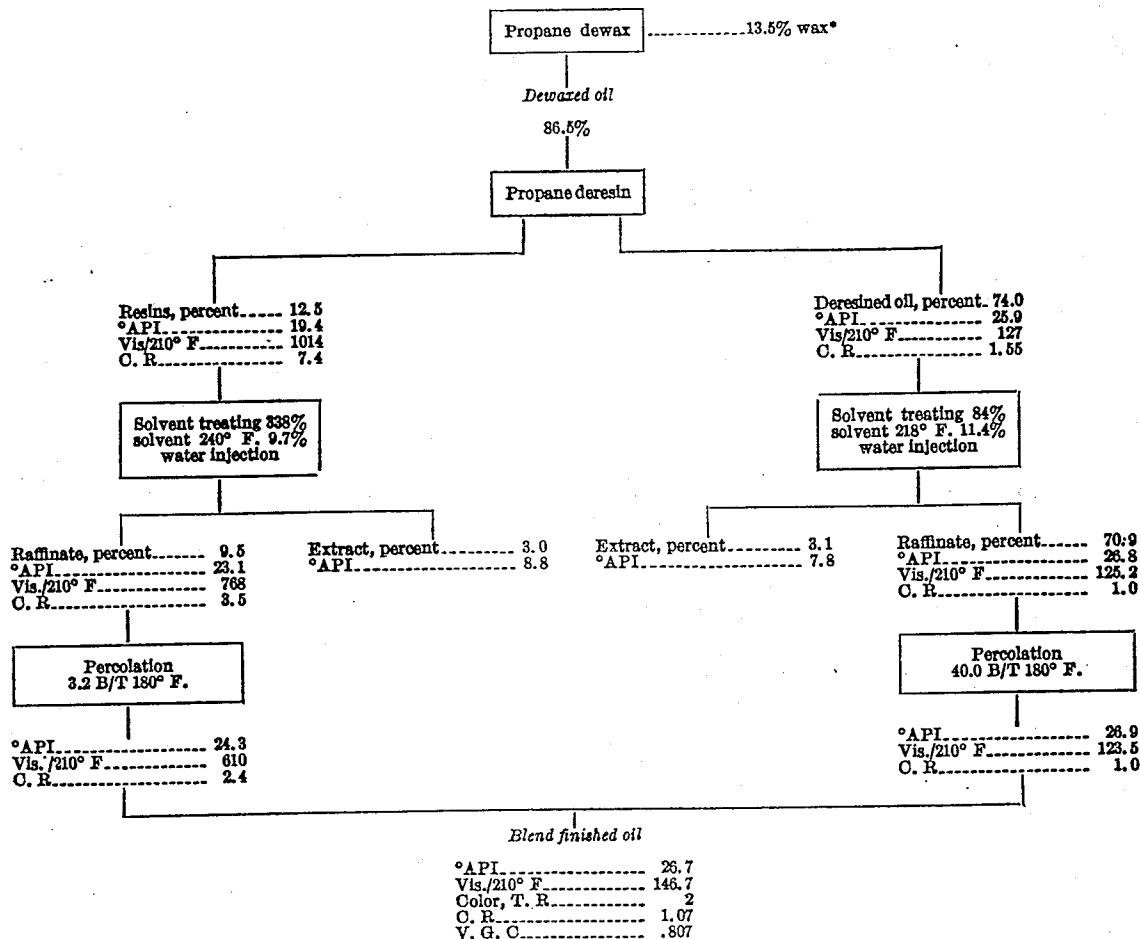
A. P. I. gravity..... 20.3°
 Viscosity at 210..... seconds... 993
 Color..... Very dark
 Vol. per cent of charge..... 13.7

When the original stock was percolated in naphtha through Attapulugus clay it was brought to a color of 1 Tag Robinson with a clay consumption corresponding to filtering 10.8 barrels of oil per ton of clay. When the resin fraction, i. e. the 20.3° A. P. I. gravity fraction, was filtered in naphtha solution through Attapulugus clay, with a clay yield corresponding to 3.6 barrels of oil per ton of clay, and when this clayed oil was blended back with the deresined oil, a color of 1 Tag Robinson was obtained on the blend. The yield of 3.6 barrels per ton of clay for filter-

ing the resin fraction corresponds to an over-all yield of 26 barrels of blended oil per ton of clay used. Thus by separating the original oil into a light fraction and a heavy resin-containing fraction in propane solution, it is possible to show an increase in yield on decolorizing the oil from 10.8 barrels per ton to 26 barrels per ton.

Table V presents data of a modification of my process wherein the dewaxing precedes the deresining and separate treatment of resins and deresined oil respectively.

TABLE V



*All percentages are volume percent.

The resin fraction was treated in a counter-current tower with 338% phenol and 9.7 water was injected at the bottom of the tower for re-fluxing. The raffinate from this process was 75.9% of the resin charge of 9.5% of the original cylinder stock. This raffinate had a viscosity of 768 seconds Saybolt at 210° F. and a carbon residue of 3.5. It was then percolated in naphtha solution through Attapulugus clay to a color of 1 Tag Robinson or about 8 N. P. A. with a clay yield of 3.2 barrels per ton. The finished resins then had a viscosity of 610 seconds Saybolt at 210° F. and a carbon residue of 2.4. Although this is an unusual product itself, it is of particular advantage when blended with the separately treated and percolated deresined oil.

The deresined oil was treated with only 84% solvent, as compared with 338% used on the resins, and the yield of 95.8% based on the charge, or 70.9% of the original cylinder stock. The viscosity was reduced only slightly (to 125.2 seconds Saybolt at 210° F.) while the carbon

residue was reduced to 0.96. This raffinate was then percolated through Attapulugus clay with a yield of 40 barrels per ton to give a color of 2½ Tag Robinson or about 6 N. P. A. and essentially no change in carbon residue.

When these two percolated oils were blended in the proportions in which they were produced, the resulting oil corresponded to 80.4% of the cylinder stock, the dewaxing loss having been 13.5% and the loss to extract by solvent treating 6.1%. The finished oil had a carbon residue

1.07, which is less than .75% of the viscosity at 210° F., and the V. G. C. was 0.807. It is particularly noteworthy that the heaviest 12% of the finished cylinder stock has a low carbon residue for the viscosity and a viscosity gravity constant which is better than that of the main portion of the bright stock.

While I have described my invention in detail, the specific examples hereinabove set forth are illustrative of my invention and will teach those skilled in the art how to practice the invention, but it should be understood that I do not limit myself to any details except as defined by the following claims which should be construed as broadly as the prior art will permit.

I claim:

1. The method of refining a petroleum oil for the production of improved lubricating oils which comprises fractionating a residual lubricating oil stock containing uncracked heavy paraffinic components and resins into at least one viscous oil fraction having a viscosity at 210° F. below 300

seconds Saybolt and a resin fraction having a viscosity at 210° F. or more than 300 seconds Saybolt, diluting the resin fraction with a hydrocarbon diluent heavier than propane but lighter than the removed viscous oil fraction, contacting the diluted resin fraction with clay in the absence of the separated viscous oil fraction, separately treating said viscous oil fraction for improving its color and quality and blending at least a portion of the clay contacted resin fraction with said separately treated viscous oil fraction in order to improve the quality of said viscous oil fraction.

2. The method of refining a petroleum oil for the production of improved lubricating oils which comprises fractionating a residual lubricating oil

stock containing uncracked heavy paraffinic components and resins into at least one viscous oil fraction having a viscosity at 210° F. below 300 seconds Saybolt and a resin fraction having a viscosity at 210° F. of more than 300 second Saybolt, diluting said resin fraction with naphtha, treating said diluted resin fraction with clay in the absence of said separated viscous oil fraction, treating said separated viscous oil fraction in the absence of the resin fraction to improve its color, stability and viscosity gravity constant, and blending at least a part of said separately clayed resin fraction with said separately treated viscous oil fraction for improving the quality thereof.

HENRY O. FORREST.

CERTIFICATE OF CORRECTION.

Patent No. 2,255,283.

September 9, 1941.

HENRY O. FORREST.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, Table II, under the heading "PHENOL TREATING" insert the following -

Vol. percent solvent-----	195
Temp. °F. top-----	207
Btm-----	170
Vol. percent water injected----	10.0

same table, under the heading "PROPANE DEWAXING", second item, for "37" read -- -37 --; same page 4, first column, line 4 from bottom, for "2.1 propane" read --2:1 propane--; page 5, Table III, lower left-hand side thereof, under the heading "Raffinate", for the third and fourth items

"V/210----- 28.6	read	--V/210----- 94.4
V/100----- 11.76"		V/100----- 1176--;

page 8, first column, line 2, claim 1, for "or" read --of--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 28th day of October, A. D. 1941.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.