

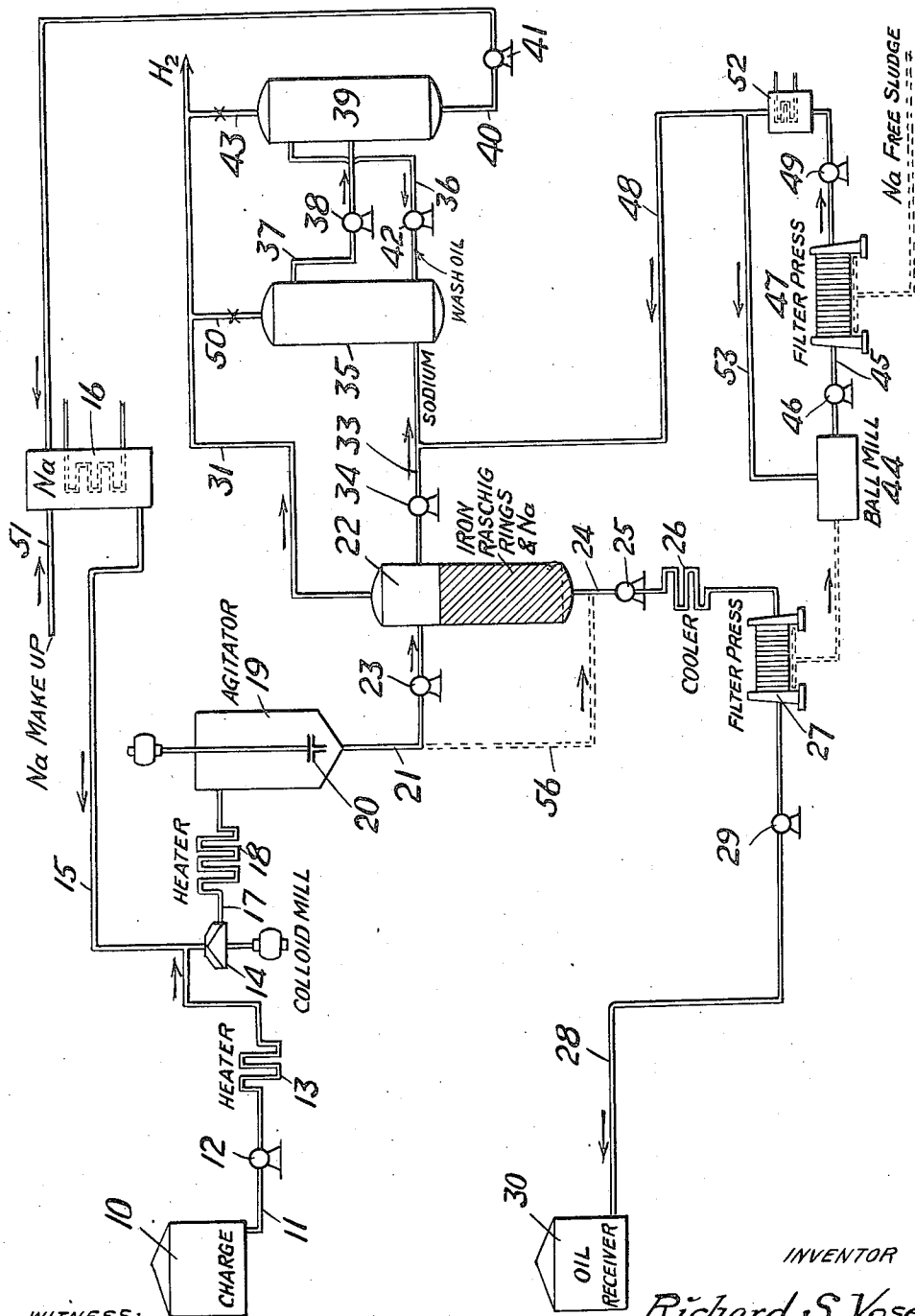
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LUBRICATING OIL

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LUBRICATING OIL

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9 Claims. (Cl. 196—151)

My invention relates to improved lubricating oils having a combination of qualities, comprising improved color, low organic acidity, good demulsibility, and particularly low Conradson carbon content and low Sligh oxidation numbers. These oils are preferably produced by specially treating distilled lubricating oils with metallic sodium, or by specially treating a residuum, or mazoot, with metallic sodium and then, by controlled methods such as, for example, as disclosed in Pew Patent No. 1,761,153, distilling my improved oils from the sodium treated mazoot. The herein described processes of producing my improved oils, which are described and claimed in Patent No. 2,055,210, issued September 22, 1936, on my co-pending application Serial No. 685,994, filed August 21, 1933, and of which this application is a continuation in part, are applicable to the treatment of any lubricating oils. The residual oil from which my improved lubricating oil distillates are distilled is believed to have a novel combination of qualities irrespective of the nature of the crude oil. It is particularly advantageous, however, to apply the processes to the treatment of naphthenic base oils (by which I mean to include mixed base oils), since when a residual oil of such a base is treated by such processes for the purpose of effecting a substantial dehydrogenation of the unsaturates, it changes their temperature-viscosity characteristics to approximate those of a paraffine base distillate, besides imparting to them a combination of qualities (including high fire and flash points, a negligible organic acidity definitive of an absolutely neutral oil, an excellent color, a very low Conradson carbon content and a zero Sligh oxidation test) which have never heretofore characterized distillates derived from crude oil of such a base. I herein claim said residual oil broadly as well as applied to naphthenic base oils as above defined, and claim specifically the novel range of distillates derived from naphthenic base oils.

It will be understood, also, that my invention is not limited to lubricating oils produced by the herein described process, nor necessarily to lubricating oils produced by treatment of the oils, or of the stock (residuum or mazoot) used to produce them, with metallic sodium, but said processes are herein fully described in order that those skilled in the art may have full and exact knowledge of practicable methods whereby they can be produced without the necessity of experiment.

The use of metallic sodium for the purpose of improving the qualities of, and particularly for

desulphurizing, gasoline is old and well known. The amount of sodium required for such purpose is so small as not to involve a prohibitive expense. But it is impossible to treat lubricating oil with metallic sodium in an amount at all comparable with that required for the treatment of gasoline and secure any substantial improvement in the quality of the oil. It is known, however, to treat naphthenic base lubricating oil with metallic sodium in much greater amounts and substantially improve some of its qualities, but the main objects of my invention, particularly dehydrogenation of unsaturates and change of temperature-viscosity relationship to approximate that characteristic of paraffine base lubricating oils, have not been achieved.

The preferred process for treating lubricating oil distillates, or oil containing lubricating oil fractions before distillation of the lubricating oils, involves the use of a comparatively large amount of metallic sodium at a temperature above its melting point. It involves, also, the formation by the sodium of a colloidal or quasi-colloidal suspension in the oil to give a maximum ratio of contact surface to weights of sodium. The process also involves a method for securing a nearly quantitative recovery of the sodium, without which the process is uneconomical.

Briefly, the process consists in mixing oil with the proper amount of sodium, at a temperature above the melting point of the sodium, and then passing the oil-sodium mixture through a colloid mill, or other apparatus capable of breaking the molten metallic sodium into colloidal or quasi-colloidal form, and dispersing it throughout the oil. After passing through the colloid mill, the oil, which now contains the sodium in colloidal or quasi-colloidal form, is passed through a heater wherein it is heated to the temperature at which it is desired to have the reaction take place. This temperature is usually in the neighborhood of 450° F. Within the reaction chamber the oil-sodium mixture is maintained in a constant state of agitation, the length of time for treatment being varied according to the type of oil being treated, and also according to the purity of the oil being treated. After the oil-sodium mixture has remained in the reaction chamber for a sufficient length of time to insure completion of the reaction, the mixture is drained from the chamber and passed either to a device for coagulating the unreacted sodium which is in colloidal form or through a cooler and then to a filter press for separation of unreacted sodium and sludge produced from the oil. The sludge is then passed

to a further stage in the process wherein the unreacted sodium may be removed therefrom; as will hereinafter be more completely described. With some oils having a high degree of purity the reaction chamber may be dispensed with, as sufficient contact is obtained during the passage of the oil-sodium mixture through the colloid mill.

The sodium apparently acts as an inductor or promoter for the reaction rather than a catalyst. The reaction is in the nature of a polymerization and/or dehydrogenation of the unsaturated portions of the oil, reducing them to a form in which they may be removed from the oil by filtration or other means. Certain sulphur compounds in the oil, and of course any water present, will react with a very small proportion of the sodium before the sodium begins its role as inductor or promoter. However, this phase of the treatment is not novel per se, as the desulphurization and dehydration of oils by the use of very small amounts of sodium is old, as heretofore set forth. It is generally recognized that the degree of concentration of a catalyst in a reaction mixture does not affect the reaction and that inductors or promoters do affect a reaction mixture by their degree of concentration. Since, therefore, it has been observed that small amounts of sodium do not promote the desired reaction in the oil, it is believed that the conclusion above referred to is correct, namely, that the sodium, after it acts as a direct desulphurization or dehydration reagent, becomes a promoter or inductor for the dehydrogenation reaction.

It is necessary, in order to secure substantial dehydrogenation, that the sodium shall be in colloidal or quasi-colloidal suspension in the oil. It is also admissible, if not necessary, in order to secure the maximum desired dehydrogenation, to use not less than from 170 to 220 grams of sodium per liter of oil, dependent on the oil being treated. It is also necessary to use this minimum proportion of sodium if it be desired to recover anything approaching a quantitative recovery of sodium. As the quantity of sodium is reduced, the proportion possible to recover is reduced. Metallic sodium being comparatively expensive, as compared, for example, with reagents such as sodium hydroxide, its recovery is essential to an economical practice of the process. For the reasons stated, it is desirable, if not necessary, to use a larger proportion of sodium than has heretofore been utilized, as well as to use it in colloidal form.

Alloys such as sodium-potassium, sodium-calcium, sodium-barium, sodium-tin, sodium-lead, and sodium-ammonium may be used in the process instead of pure sodium. Sodium amalgam may also be used. It is only necessary to carry out the reaction at temperatures above the melting point of the alloy or amalgam, and reduce the amalgam to colloidal or quasi-colloidal form. It is of course known that ammonia when in combination with sodium acts as a metal, the combination being, to all intents and purposes, an alloy.

A better understanding of practicable process for producing my improved lubricating oil may be had by reference to the accompanying drawing illustrative of one form of apparatus for carrying out such a process:

The single figure is a diagrammatic flow sheet of an apparatus for treating oil with an excess of molten metallic sodium and recovering unused sodium.

In the drawing: The numeral 10 designates a storage vessel or tank for the oil to be treated.

The oil is removed through line 11, and forced by means of pump 12 through heater 13 to colloid mill 14. Before entering the colloid mill, there is added, to the oil stream in pipe 11, a predetermined amount of molten sodium from line 15, which is in communication with a molten sodium storage vessel 16. On passing through the colloid mill, the sodium and oil are thoroughly mixed, and the molten sodium is reduced to an extremely finely divided condition.

Leading from the colloid mill 14 is line 17, in which is placed heater 18 for heating the oil-sodium mixture. This line 17 leads to an agitator 19 having therein a motor driven centrifugal agitating member 20. There may be provided three or more agitators such as 19 in order, if desired, to make the process a continuous one. The centrifugal agitating member 20 is rotated at an extremely high speed of the order of 9000 R. P. M. The agitating member itself consists of a disc having a number of radial passages therethrough, and a downwardly extending portion having a passage therethrough which communicates with the radial passages in the disc. By rotating the agitating member at high speeds, the oil-sodium mixture is drawn through the downwardly extending portion and is expelled through the radial passages by the centrifugal force created by the high rotative speed. The agitator 19 is provided with suitable heating means (not shown), to maintain the oil-sodium mixture at reaction temperature during its entire time of treatment. Any suitable heating means may be used, such as electrical resistance heaters or heating coils employing steam or hot oil.

It is practicable, in place of the specific agitator described, to employ agitators of other types commonly used in the treatment of oils. Indeed it may be possible, after the passage through the colloid mill, to dispense with agitation, since agitation is not required to maintain the sodium in suspension in the oil. Agitation, however, is desirable, if not required, to maintain the entire body of oil at a substantially uniform temperature. It is also possible, with the type of agitator shown, to add the oil and molten sodium directly and reduce the sodium to colloidal form by the action of the agitator alone. This procedure, however, consumes considerably more time than does the combination of the colloid mill and agitator.

From the bottom of the agitator 19 a valve line 21 leads to a tower 22. The treated mixture of oil, sludge and unreacted sodium is forced by means of pump 23 in line 21 into the tower 22. This tower 22 contains packing material 23, which may be iron Raschig or Lessing rings, filling the tower to approximately two-thirds of its capacity.

The oil, containing sludge and some sodium, drains from the bottom of the tower 22, through line 24, containing pump 25, and cooling coils 26, to a filter press 27. There may be provided a number of presses in order that the process may be carried on continuously. Line 28 containing pump 29 leads from a filter press 27 to storage tank 30 for finished oil. The sludge, together with the finely divided sodium, forms filter cakes on the filtering media within the press 27.

Leading from the top of tower 22 is a line 31 for the removal of hydrogen which is formed during the treating step and is adsorbed by the sodium.

From a point below the top of tower 22 there

extends a line 33 containing pump 34 for drawing off metallic sodium which is separated in the tower and accumulates to the level of line 33. This metallic sodium is passed to a washing tank 35, wherein it is mixed with naphtha entering through line 36. The mixture of naphtha and sodium is withdrawn from the top of the washing apparatus through line 37 containing pump 38. The naphtha-sodium mixture is thus pumped to a settling chamber 39, wherein the sodium is settled from the naphtha and is pumped out through line 40 by means of pump 41 and returned to sodium storage receptacle 16. The naphtha is removed from near the top of settling tank 39 through line 36 and is returned by means of pump 42 through line 36 to washing apparatus 35.

Lines 50 and 43 lead from the tops of tanks 35 and 39 respectively, to line 31 for the removal of hydrogen which is separated in washing tank 50 and settling tank 39.

The sludge and sodium which accumulate as filter cakes in press 27 are placed in ball mill 44, or in some other suitable grinding apparatus, and therein admixed with molten sodium admitted through line 53. After being thoroughly ground with molten sodium, the mixture is passed through line 45 by means of pump 46, and is forced through filter press 47. The filtering medium in press 47 is of a type which will be wetted by metallic sodium and will therefore permit the free coalesced metallic sodium to pass through, but will hold back the sludge, which collects on such filtering medium as a filter cake. The sodium which has passed through the press 47 is removed through line 48 and part of it is forced, by means of pump 49, to the sodium washing chamber 35, and the remaining portion is recycled through line 53 to the ball mill 44 for admixture with sodium-containing sludge removed from press 27. When filter cakes of the desired depth have been formed on the media in press 47, they are removed therefrom.

In operation, the oil removed from tank 10, containing the charging stock, is heated in heater 13 to a temperature somewhat above the melting point of metallic sodium (207° F.), and is then passed through line 15 leading to the colloid mill. Line 15, as heretofore explained, conveys molten metallic sodium from the storage receptacle 16 to the colloid mill. The sodium and oil are so proportioned that the charge entering colloid mill 14 is 70 pounds of sodium to 1 barrel of oil (equivalent to approximately 200 grams of sodium to 1 liter of oil). In passing through the colloid mill the sodium is reduced to an extremely finely divided state and thoroughly admixed with the oil. The oil-sodium mixture leaving the colloid mill is then passed through heater 18 wherein it is heated to a temperature within the range of 400°-450° F. Enough oil-sodium mixture is fed to the agitator 19 to fill it to approximately two-thirds of its total capacity. Due to the high rotative speed of the agitating member 20, the oil-sodium mixture is maintained in a state of agitation insuring equal temperature conditions throughout the whole mixture. The oil-sodium mixture is maintained in the highly agitated condition for a period of time ranging from half an hour to thirty-six hours, depending upon the type of oil being treated and the resistance to oxidation or change in temperature-viscosity relationship desired.

Exactly what chemical change takes place in the oil is not known, as the sludge which is re-

covered has so far resisted all attempts at analysis. However, it has been determined that both dehydrogenation and probably hydrogenation are effected by the sodium treatment. That dehydrogenation is effected is shown by the presence of adsorbed hydrogen in the unreacted sodium. That hydrogenation is effected may be inferred by the change in temperature-viscosity characteristics and the change in specific gravity. The presence of free hydrogen in an adsorbed condition in the unreacted sodium present in the sludge was first detected when analyzing the sludge to determine the amount of sodium which had been reacted. This analysis was carried out by adding alcohol to a weighed sample and determining the amount of hydrogen evolved. Since the amounts of hydrogen evolved were greater than the equivalent of the amount of sodium which was known to be present, it was apparent that free hydrogen must be present. This was also conclusively demonstrated when the sludge was later treated with a further excess of molten sodium, and then washed with naphtha. Since no chemical change was apparent in the naphtha or in the sodium, and since free hydrogen was evolved, there can be no doubt that a dehydrogenation of the original oil took place in the agitator. Since the temperature-viscosity characteristics and the gravity of the oil were also changed, it was evident that dehydrogenation and possibly hydrogenation of the original oil had been effected. It is well known that naphthenic base oils have a temperature-viscosity slope differing from the temperature-viscosity slope of paraffin base oils, and when the temperature-viscosity slopes of the untreated and the treated oils were compared, it was found that the treated oil more nearly approximated the temperature-viscosity slope of a paraffin base oil. This fact, coupled with the change in specific gravity, corresponding to increase in the percentage of hydrogen in the oil, leaves no doubt as to the occurrence of dehydrogenation and possibly hydrogenation. It is, of course, evident that if unsaturated components of the original oil are dehydrogenated by the action of the sodium and the dehydrogenated components are removed as sludge, the remaining components of the oil will be substantially saturated. It is also evident that the sodium treatment may dehydrogenate a portion of the unsaturates and hydrogenate the remaining unsaturates. There is concrete evidence of the dehydrogenation and removal of unsaturates but no irrefutable evidence of hydrogenation of a portion of the unsaturates.

The treated oil, containing unreacted sodium and sludge, is drained from the agitator 19 and pumped into the top of packed tower 22, which contains in addition to the packing, fresh molten sodium. The entering mixture may be considered as an oil-sodium emulsion with the oil in the external continuous phase. Obviously, with such an emulsion, the coalescence of the sodium particles is impossible until the phase is changed and the sodium becomes the external continuous phase, or interlacing phase. This change is accomplished by flowing the oil, sludge and sodium mixture downwardly through the tower 22 which contains Raschig or Lessing ring packing and metallic sodium. In flowing down through the tower, the oil sodium emulsion changes phase and the sodium in the emulsion becomes the external phase, due to the action of the large bath of sodium in the tower, which coalesces the finely

divided sodium that mixes with the sodium constituting the bath. In practice, all but about 17 or 18 per cent of the unreacted sodium will be recovered in the tower 22. When refining some light oils, the oil-sodium mixture may have a specific gravity less than that of the molten sodium at the temperature existent in tower 22. In such case the oil-sludge mixture would be admitted to the bottom of tower 22 and flowed upwardly through the sodium. The sodium free oil-sludge mixture would then be withdrawn from tower 22 near the top thereof, and the excess sodium at a point below that at which the oil sludge mixture is withdrawn.

The oil-sludge mixture, still containing a small proportion of unreacted sodium, is passed from the bottom of the tower through cooling coils 26 to the filter press 27. Due to the coalescence in the tower of the finely divided sodium present in the oil-sludge mixture, the amount of free sodium in the tower is increased, the excess being drawn off through line 33 and passed to the washing chamber 35.

In some cases it may be desired to eliminate the sodium recovery step in tower 22, in which case the oil-sodium mixture from agitator 19 will be passed directly to pump 25, cooler 26, and press 27 by by-pass 56 shown in dotted lines.

As hereinbefore explained, the oil sludge mixture after being cooled in coils 26 to a temperature approximating 70° F., is passed through the filter press 27, wherein the oil is separated entirely from sludge and unreacted sodium and is passed out through line 28 to storage tank 30. From time to time the filtering media in press 27 are removed and the sludge cakes accumulated thereon are placed in ball mill 44, and ground with molten metallic sodium. When this grinding is complete, the sludge-sodium mixture is subjected to further pressing in filter press 47. This operation results in the removal of all unreacted sodium from the sludge. The free sodium is then partly recycled to the grinding operation in mill 44, and partly pumped to washing tank 35.

The washing and settling operations remove from the sodium the hydrogen which has been adsorbed therein. In the washing step, carried out in tank 35, a small amount of hydrogen may be released, which is from time to time vented through line 50 to line 31. The naphtha used for washing the sodium to remove adsorbed hydrogen is circulated continuously through the tanks 35 to 39, and is unchanged by the operations performed therein. The sodium is removed from settling tank 39 and is pumped to sodium storage tank 16. Sodium, to make up for that lost in the reaction, is added to storage tank 16 and may be added in the molten state through line 51.

It is to be understood that any low boiling metal may be used to dissolve the unreacted colloidal sodium, or mercury may be used to form a sodium amalgam which can be readily removed from the treated oil or sludge. Such metals as potassium, calcium, barium, tin and lead may be used to coalesce the colloidal sodium and would be equivalent to sodium used for the same purpose.

In all steps from its admission with the oil to the colloid mill to the final removal of all unreacted sodium, except in the step of separating the oil, the sodium is maintained in a molten condition. The temperature in agitator 19 is substantially 450° F. A like temperature prevails in tower 22.

Other methods of treatment with and recovery

of metallic sodium are described and claimed in my prior co-pending application Serial No. 685,994, filed August 21, 1933, but are not described herein as such further description would be superfluous.

Washing apparatus 35 and settling apparatus 39 are also maintained at temperatures above the melting point of sodium. The sodium which is separated with the sludge in filter press 27 has been cooled to a point below its melting point, but is subsequently heated by the addition of molten sodium to ball mill 44. Since the only sodium which circulates through ball mill 44 is that which is recovered, there is provided, preferably in line 48, a heating apparatus 52, to which heat may be supplied, preferably by means of superheated steam circulated through the coil therein, although mineral oil may be used as the heat transmitting medium. Since the melting point of sodium is below the boiling point of water, the handling of molten metallic sodium presents no difficulties whatever. It is, of course, understood that all sodium-carrying lines should be insulated.

The operations so far described have been directed to procedure for treating oil with sodium by a batch process. It is to be understood, however, that the treating and recovery methods hereinbefore described may be carried out in a continuous manner. For instance, oil and sodium in the proper proportions may be charged to agitator 19, (Fig. 1) and the mixture agitated until a sample of the oil upon withdrawal shows that it has reached the degree of purity or dehydrogenation desired. When the oil has reached such a state, fresh oil-sodium mixture is continuously charged to the agitator 19 at a slow rate and treated oil-sludge mixture is continuously withdrawn from the agitator at the same rate that the untreated oil-sodium mixture is charged. The treated oil-sludge mixture is then continuously passed through the tower 22 to coalesce most of the sodium contained in the sludge, and the largely sodium free mixture is withdrawn from tower 22 and passed through filter press 27. There will, of course, be provided a number of filter presses such as presses 27 and 47, and also several grinding devices, such as ball mill 44, in order that the final recovery of sodium from sludge may be continuous.

Numerous variations of the described preferred process are practicable. For instance, the oil treated may be a distilled lubricating fraction which has or has not been acid treated or subjected to other refining processes such as SO₂ treatment or contact clay treatment, or the oil treated may be a residual lubricating oil which has or has not been subjected to the usual refining steps. The third modification, and probably the most desirable, is the treatment of a reduced crude before distilling into the various desired lubricating fractions. If it is desired to treat a reduced crude, the fractions including all or light gas oil and all lighter fractions are removed by overhead distillation in any known manner, and the reduced crude or mazoot is treated as hereinbefore described by the addition of an excess of metallic sodium to the mazoot. The mixture is then subjected to the heating and reclaiming steps herein described. It has been stated that this modification is probably the most desirable, since the treating time is greatly lowered. For instance, with some impure lubricating oil fractions, the time of reaction may be as high as 36 hours, but when treating a mazoot with

sodium the reaction time is cut to one-half an hour.

The following specific examples will serve to illustrate the oil obtained when treating distillates and when treating reduced crudes and then distilling the desired lubricating fraction herefrom:

	Oil I (distillate)		Oil II (distillate)	
	Before Na treat- ing	After Na treating	Before Na treat- ing	After Na treating
15 Gravity, A. P. I.	21.1	24.1	19.7	23.2
Flash, Cleveland open cup, °F	340	335	400	395
Fire, °F	360	375	460	455
Viscosity, S. U./100° F.	152	144	533	440
Viscosity, S. U./130° F.	70	79	209	179
Viscosity, S. U./210° F.	39	43	54	54
Color - N. P. A.	134	1—	244	1—
Pour test, °F	-35	-35	0	-15
Demulsibility	1260	1620	1620	1620
Emulsibility	Good	Good	Good	Good
Steam emulsion	120	17	140	65
Conradson carbon	0.03	0.02	0.05	0.03
Ash	Trace	Trace	Trace	Trace
Reaction	Neutral	Neutral	Neutral	Neutral
Sligh oxidation	51.8	0.0	45.1	0.0
Sulphur, percent	0.23	0.01	0.24	0.01
Temperature of treatment, °F	410-440		410-440	
Yield of refined oil, percent	85		90	
Grams of metallic sodium per liter of oil	200		200	

	Oil III (distillate)
35 Temp. of treatment, °F	420-440
Time of treatment, hours	20
Temp. of filtration, °F	70
Grams of Na per liter of oil	244
Yield of refined oil, percent	82.5
Yield of "polymer", percent	17.5

	Characteristics of the oil		Before refining	After re- fining
45 Gravity, A. P. I.			18.3	22.1
Flash, Cleveland open cup, °F			450	430
Fire			515	510
Vis. Saybolt Univ. at 100° F.			2390	1354
Vis. Saybolt Univ. at 130° F.			672	461
Vis. Saybolt Univ. at 210° F.			88	82
Color, N. P. A.			34	13
Pour test, A. S. T. M., °F			+10	0
Demulsibility			300	1620
Emulsibility			Good	Good
Conradson carbon, percent			0.22	0.06
Ash			Trace	Trace
Reaction			Neutral	Neutral
Sligh oxidation			48.1	0.0
Sulphur, percent			0.35	0.02

	Oil IV (distillate)
Temp. of treatment, °F	430-440
Time of treatment, hours	20
Temp. of filtration, °F	70
Grams of Na per liter of oil	213
Yield of refined oil, percent	75.7
Yield of polymer (by difference), percent	24.3

Characteristics of the oil	Before refining	After refining
Gravity, A. P. I.	18.5	23.2
Flash, Cleveland open cup	520	500
Fire	605	585
Vis. S. U. at 100° F.	6113	2219
Vis. S. U. at 130° F.	1515	745
Vis. S. U. at 210° F.	152	109
Color, N. P. A.	-54	-134
Pour test, A. S. T. M., °F	+25	+5
Demulsibility	180	1620
Emulsibility	2 cc. Cuff.	Good
Conradson carbon, percent	0.89	0.12
Ash	Trace	Trace
Reaction	Neutral	Neutral
Sligh oxidation	65.6	0.0
Sulphur, percent	0.45	0.03

The following examples will serve as illustrations of the oils produced when mazoots or topped crudes (free of gas oil and lighter fractions) are treated with colloidal metallic sodium, and then distilled under vacuum into cuts suitable for use as "commercial lube oils":

Oil V—Mazoot from spindle top crude

Oil VI—Mixture consisting of mazoots from:

- 25% Esperson crude,
- 25% Vinton and Goose Creek crudes, and
- 50% Liberty crude.

	Oil V	Oil VI
Sodium/oil ratio used	200 g. Na/liter oil	200 g. Na/liter oil.
Temp. of treatment	425-450° F.	425-450° F.
Time of treatment	1 hour	1 hour
Temp. of filtration	70° F.	70° F.
Apparatus used	Centrifugal agitator 10,000 R. P. M. Suction filter.	Centrifugal agitator 10,000 R. P. M. Suction filter.
Yield of Na treat.	81.0%	73.0%
Mazoot from untreat. Mazoot.		
Amount of sodium consumed.	2.7%	0.86%
Hydrogen gas formed per bbl. untreat. Mazoot.*	163.0 cu. ft.	83.5 cu. ft.
Hydrogen gas formed per bbl. Na treat. Mazoot.*	201.2 cu. ft.	114.4 cu. ft.

*H₂ at 70° F. and 14.7 pounds pressure.

Distillates from oil V after treatment

	Temp., °F.		Mm. pres.	Per- cent chg.	A. P. I. grav.	Flash, °F.	Fire, °F.	Viscosity, °F.				Sligh oxid.	Color N. P. A.	Per- cent Con. carbon	Per- cent sulf.	Neut. No.
	Vap.	Liq.						100	130	160	210					
60 Orig. oil V					20.3	360	410	934	340		70			1.85	0.34	2.0 mg. KOH.
65 Na treated oil V					23.9	355	400	452	191		56	0.0	5½	0.16	0.09	Neut.
Cut 1	328	426	2	10	27.6	265	285	48	40	36		0.0	1—	0.01	0.04	Do.
2	360	460	2	10	26.5	320	360	88	58		38	0.0	1—	0.01	0.03	Do.
3	388	492	2	10	25.6	360	405	135	78		41	0.0	1—	0.01	0.04	Do.
4	414	522	2	10	24.6	395	440	265	123		47	0.0	1—	0.01	0.04	Do.
5	436	546	2	10	23.6	410	470	513	213		55	0.0	1—	0.01	0.06	Do.
6	460	575	2	10	22.8	440	510	803	304		65	0.0	1½	0.01	0.05	Do.
7	484	609	2	10	22.7	455	530	1130	404		73	0.0	1½	0.01	0.04	Do.
8	513	673	2	10	23.2	505	585		451	198	80	0.0	1½	0.02	0.05	Do.
9	556	719	1	10	24.1	530	610		596	264	99	0.0	1½	0.06	0.06	Do.
10	619	738	1	3	24.1	545	625	2215	789	340	122	0.0	3+	0.11	0.06	Do.
Btms.				7	22.5	655	725			1173	341	3.8	Dk. red	1.79	0.09	Do.

Distillates from oil VI after treatment

5	Temp., °F.		Mm. pres.	Per- cent chg.	A. P. I. grav.	Flash, °F.	Fire, °F.	Viscosity, °F.				Sligh oxid.	Color N. P. A.	Per- cent Con. carbon	Per- cent sulf.	Neut. No.	5	
	Vap.	Liq.						100	130	160	210							
10	Orig. oil VI				19.3	270	325	901	345		70			2.75	0.38	2.3 mg. KOH.	10	
	Na treated oil VI				24.4	285	320	348	156		52	0.0	3	0.11	0.08	Neut.		
	Cut 1	309	355	1	10	29.8	210	240	41.2	36.8	34	0.0	1-	0.01	0.04	Do.		
	2	346	388	1	10	26.8	270	295	56.4	44.4	38.8	0.0	1-	0.01	0.04	Do.		
	3	382	429	1	10	25.6	310	345	86	56		37	0.0	1-	0.01	0.02		Do.
	4	417	465	1	10	24.4	350	400	185	92		42	0.0	1	0.01	0.04		Do.
	5	445	496	1	10	23.4	390	440	380	159		49	0.0	1½	0.01	0.02		Do.
	6	478	532	1	10	22.4	405	480	798	283		60	0.0	1½	0.01	0.04		Do.
	7	517	582	1	10	21.7	460	525	1503	500		80	0.0	1½+	0.01	0.02		Do.
	8	565	648	1	10	22.1	505	580		694	287	100	0.0	2+	0.08	0.04		Do.
15	9	623	732	1	10	22.7	550	615		1007	427	138	0.0	3+	0.21	0.06	Do.	
	Btms.				10	22.1	655	715			1778	455	1.5	Dk. red	1.87	0.06	Do.	

20 Vacuum distillation of 8th distillate from oil V

25	Cut	Temp., °F.		Pres.	Chg.	Flash, °F.	Fire, °F.	Vis/210° F.	Color	25
		Vap.	Liq.							
				Mm.	Per-cent					
1	484	520	1	10	440	525	79	1¼		
2	489	528	1	10	450	540	82	1¼+		
3	495	536	1	10	470	545	83	1¼		
4	505	542	1	10	470	550	83	1¼		
5	520	560	1	10	475	550	84	1¼		
6	524	567	1	10	480	560	85	1¼		
7	533	575	1	10	485	570	85	1¼		
8	537	580	1	10	495	575	85	1¼		
9	543	589	1	10	505	585	86	1¼		
35	Btms.				10	530	615	101	4¼-	

Vacuum distillation of 8th distillate from oil VI

25	Cut	Temp., °F.		Pres.	Chg.	Flash, °F.	Fire, °F.	Vis/210° F.	Color	25
		Vap.	Liq.							
				Mm.	Per-cent					
1	514	562	1	10	470	540	84	1¼-		
2	528	575	1	10	475	545	90	1¼		
3	545	594	1	10	480	560	93	1¼		
4	548	598	1	10	485	570	96	1¼+		
5	555	603	1	10	500	585	99	1¼+		
6	556	606	1	10	505	590	102	1¼+		
7	562	618	1	10	510	595	105	2+		
8	580	632	1	10	540	605	111	2¼		
9	620	680	1	10	560	630	123	2½-		
	Btms.				10	575	650	169	5¼	

Vacuum distillation of 9th distillate from oil V

40	Cut	Temp., °F.		Pres.	Chg.	Flash, °F.	Fire, °F.	Vis/210° F.	Color	40
		Vap.	Liq.							
				Mm.	Per-cent					
1	535	602	1	10	480	560	82.2	1¼		
2	551	609	1	10	500	580	89.2	1¼		
3	564	617	1	10	510	595	92.1	1¼		
4	574	628	1	10	515	595	93.5	1¼		
5	580	642	1	10	525	605	94.5	1¼		
6	591	654	1	10	530	620	98.4	1¼		
7	602	670	1	10	540	630	103.9	2		
8	614	698	1	10	555	635	111.2	2¼		
9	620	720	1	10	560	650	123.7	2½+		
55	Btms.				10	610	675	177.2	Dark red	

Vacuum distillation of 9th distillate from oil VI

40	Cut	Temp., °F.		Pres.	Chg.	Flash, °F.	Fire, °F.	Vis/210° F.	Color	40
		Vap.	Liq.							
				Mm.	Per-cent					
1	558	630	1	10	500	595	114	2¼+		
2	567	640	1	10	505	600	114	2¼-		
3	580	650	1	10	525	610	121	2¼+		
4	590	661	1	10	530	620	123	2¼+		
5	602	672	1	10	540	630	130	2¼+		
6	610	681	1	10	545	635	136	2¼-		
7	623	695	1	10	550	640	143	3-		
8	634	711	1	10	555	650	152	3¼		
9	652	725	1	10	585	665	172	3¼-		
	Btms.				10	620	695	248	Dark red	50

Vacuum distillation of 10th distillate from oil V

60	Cut	Temp., °F.		Pres.	Chg.	Flash, °F.	Fire, °F.	Vis/210° F.	Color	60
		Vap.	Liq.							
				Mm.	Per-cent					
1	567	624	1	10	480	555	83	3¼+		
2	572	628	1	10	545	635	107	3-		
3	584	637	1	10	555	635	116	2¼-		
4	592	648	1	10	555	635	118	2¼+		
5	610	660	1	10	560	645	126	2¼+		
6	618	672	1	10	570	650	128	2¼+		
7	624	677	1	10	575	650	135	2¼+		
8	631	692	1	10	585	665	141	2¼-		
9	657	720	1	10	615	685	158	3¼		
75	Btms.				10	630	700	229	Dark red	

It will be understood that the values given in the tables and in the appended claims refer, unless otherwise specifically noted, to the results of tests approved by the American Society for Testing Materials.

Where, in the tables, the values for sulphur content and Conradson carbon are given in terms of percentages, it will be understood that such percentages are approximate, in that they are expressed as accurately as possible, where the percentages are expressed in terms of hundredths of one per cent. Thus, in the tables entitled Distillates from Oils V and VI after Treatment, the Conradson carbon of a number of cuts is substantially less than the .01% specified, but is expressed by that percentage merely to indicate that the Conradson carbon content is not absolutely zero.

While, in the examples given in the tables entitled Distillates from Oils V and VI, the sodium treated long residua or mazoots from which the oils are distilled have respectively Conradson carbon contents of .16% and .11%, other sodium

treated long residua may have Conradson carbon contents varying from .05% to .3%.

What I claim and desire to protect by Letters Patent is as follows:

5 1. A residual petroleum oil produced by distilling from the crude gas oil and lighter constituents and which contains and from which can be produced a substantially full range of lubricating oil constituents varying from a distillate having a flash point not over 300° F. and a fire point not over 350° F. to a distillate having a flash point not less than 525° F. and a fire point not less than 600° F., characterized by the fact that its organic acidity as tested by the A. S. T. M. method does not exceed two-hundredths neutralization number, its Conradson carbon content is less than four-tenths of one per cent. and its Sligh oxidation test is zero.

20 2. A residual petroleum oil produced by distilling from the crude gas oil and lighter constituents and which contains and from which can be produced a substantially full range of lubricating oil constituents varying from a distillate having a flash point not over 300° F. and a fire point not over 50° F. to a distillate having a flash point not less than 525° F. and a fire point not less than 600° F., characterized by the fact that its organic acidity as tested by the A. S. T. M. method does not exceed two-hundredths neutralization number, its Conradson carbon content is less than one-fourth of one per cent and its Sligh oxidation test is zero.

35 3. A residual petroleum oil produced by distilling from the crude gas oil and lighter constituents and which contains and from which can be produced a substantially full range of lubricating oil constituents varying from a distillate having a flash point not over 300° F. and a fire point not over 350° F. to a distillate having a flash point not less than 525° F. and a fire point not less than 600° F., characterized by the fact that its organic acidity as tested by the A. S. T. M. method does not exceed two-hundredths neutralization number, and its Sligh oxidation test is zero and whose highest distillable lubricating oil portion has a Conradson carbon content less than four-tenths of one per cent.

4. An improvement in lubricating oils which are distillates of naphthenic base petroleum oils and which have flash points ranging from 210° to 550° F. and higher fire points ranging from 240° to 625° F. and whose organic acidity as tested by the A. S. T. M. method does not exceed .02 neutralization number, characterized by the fact that its Conradson carbon content ranges from about one one-hundredth of one per cent with oils in the low part of the range to less than .24 per cent with oils at the highest part of the range, that its color number ranges from less than one N. P. A. in the low part of the range to less than 4 N. P. A. in the highest part of the range, and that its Sligh oxidation is zero.

5. An improvement in lubricating oils which are distillates of naphthenic base petroleum oils and which have flash points within the range 210° to 460° F. and higher fire points within the range 240° to 530° F., and whose organic acidity as tested by the A. S. T. M. method does not exceed .02 neutralization number, characterized by the fact that its Conradson carbon content is less than one-fortieth of one per cent, that its color number is not over 2 N. P. A., and that its Sligh oxidation is zero.

6. An improvement in lubricating oils which are distillates of naphthenic base petroleum oils and which have flash points within the range 450° to 550° F. and higher fire points within the range 520° to 625° F. and whose organic acidity as tested by the A. S. T. M. method does not exceed .02 neutralization number, characterized by the fact that its Conradson carbon content is less than one-fourth of one per cent, that its color number is not over 4 N. P. A., and that its Sligh oxidation is zero.

7. A residual naphthenic base petroleum oil produced as defined in claim 1 and having the characteristics specified in claim 1.

8. A residual naphthenic base petroleum oil produced as defined in claim 2 and having the characteristics specified in claim 2.

9. A residual naphthenic base petroleum oil produced as defined in claim 3 and having the characteristics specified in claim 3.

RICHARD S. VOSE.