

[72] Inventors **Lorne W. Sproule;**  
**Donald W. Murray, both of Sarnia,**  
**Ontario, Canada**  
 [21] Appl. No. **794,688**  
 [22] Filed **Jan. 28, 1969**  
 [45] Patented **Dec. 14, 1971**  
 [73] Assignee **Esso Research and Engineering Company**

3,481,863 12/1969 Donaldson et al. .... 208/211  
 3,095,366 6/1963 Schieman ..... 208/19  
 3,224,955 12/1965 Anderson ..... 208/28  
 3,438,887 4/1969 Morris ..... 208/28

Primary Examiner—Delbert E. Gantz  
 Assistant Examiner—G. J. Crasanakis  
 Attorneys—Pearlman and Schlager and Wayne Hoover

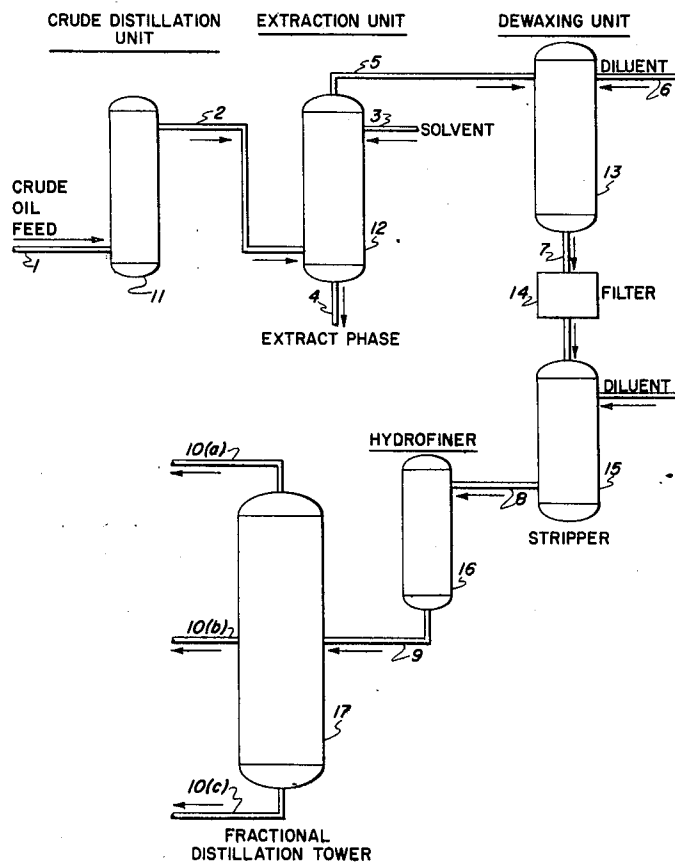
[54] **PROCESS FOR PRODUCING LOW-POUR POINT TRANSFORMER OILS FROM WAXY CRUDES**  
 11 Claims, 2 Drawing Figs.

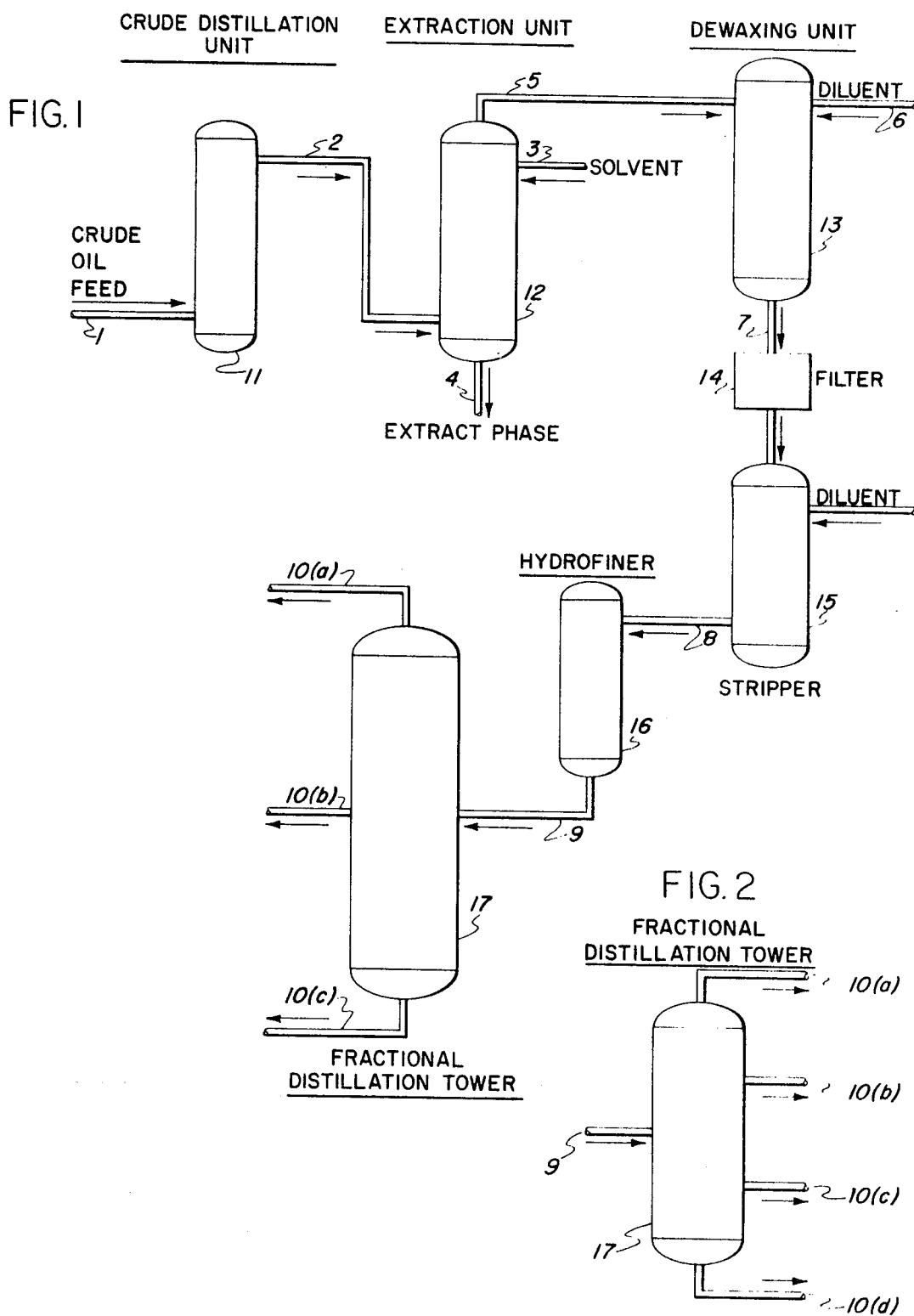
[52] U.S. Cl. .... 208/211,  
 208/311  
 [51] Int. Cl. .... C10g 23/00,  
 C10g 31/14, C10g 21/00  
 [50] Field of Search ..... 208/211,  
 209, 33, 28, 34, 19, 18, 311

[56] **References Cited**  
**UNITED STATES PATENTS**

3,014,859 12/1961 Royder ..... 208/33  
 3,382,168 5/1968 Wood et al. .... 208/211

**ABSTRACT:** A high-stability, low-pour point insulating oil is prepared by a process wherein a broad waxy vacuum gas oil fraction is first dewaxed and then fractionated to yield the desired insulating oil fraction. The broad vacuum gas oil fraction may be treated to remove aromatics and polar components. Likewise, the dewaxed oil may be hydrofined before fractionation to improve color, stability and sulfur content. The insulating oil fraction is obtained from the dewaxed oil as a heart cut. The bottoms fraction may be used as a lubricating oil, and the pour point of this fraction can be improved with further fractionation. The high-boiling waxy contaminants picked up during processing are removed as bottoms, thus yielding a product having an improved pour point when compared with an oil prepared by fractionation prior to dewaxing.





L. W. Sproule  
D. W. Murray

Inventors

By *Wayne H. Hoover* Attorney

# PROCESS FOR PRODUCING LOW-POUR POINT TRANSFORMER OILS FROM WAXY CRUDES

## BACKGROUND

This invention relates to a method of preparing high-stability insulating oils. More particularly, this invention relates to an improved method for preparing high-stability insulating oils having high-flash points and low-pour points from paraffinic distillates. Still more particularly, this invention relates to a method wherein high-stability insulating oils are cut with a high degree of fractionation from a previously dewaxed, broad hydrocarbon oil fraction from a paraffinic crude.

High-stability insulating oils are known in the prior art and are used in transformers and other electrical equipment such as circuit breakers. These oils are 2by (1) a relatively low viscosity, (2) a high-dielectric strength, (3) a relatively high-flash point, and (4) a low-pour point. In addition, these oils must be low in corrosive agents such as acid, alkali, and sulfur and resistant to oxidation and sludge formation.

Several methods for preparing insulating oils are known in the prior art. In general, they are produced from wax-free naphthenic crude oils which are not native to many parts of the world and consequently command premium prices and involve high transportation costs. Although these crudes permit production of exceptionally low-pour point insulating oils without the need for dewaxing or special attention to the degree of fractionation or distillate cut width, they also contain high percentages of sulfur and nitrogen which must be removed in order to satisfy the stringent stability requirements of insulating oils.

Extremely stable-insulating oils produced either totally or partially from paraffinic crudes by conventional dewaxing techniques are also used in certain applications where moderate climatic conditions do not demand oils with especially low-cloud or pour points. When exceptionally low-pour points are required, however, deep dewaxing of paraffinic distillates at temperatures below  $-40^{\circ}\text{F}$ . cannot compete economically with the manufacture of these oils from naphthenic crudes.

A process which avoids deep dewaxing and produces competitively priced, low-viscosity oils with exceptionally low-pour points from paraffinic distillates was disclosed in U.S. Pat. No. 2,906,688. In this process a broad, wax-containing fraction is first sharply fractionated to yield a narrow heart cut of suitable viscosity which is then dewaxed at about  $0^{\circ}\text{F}$ . to yield a dewaxed oil having a pour point of  $-50^{\circ}\text{F}$ ., or lower. The narrow cut also may be solvent extracted prior to dewaxing without materially affecting the pour point of the product. The difficulty associated with this process is related to the nature of the low-pour point oils produced. Although they have low-pour points, their cloud points, which mark the onset of wax precipitation, are relatively high, approximating the dewaxing temperature used in making them. This, in turn, makes the pour point of the product extremely sensitive to waxy contaminants such as paraffin wax, which would certainly be encountered in process lines comprising phenol treating, hydrofining, and dewaxing units which normally operate on relatively high-pour point, wax-bearing streams. In fact, it has been found that the addition of as little as 0.5 percent of a paraffin wax to an insulating oil prepared by the aforescribed process and having a cloud point of  $-12^{\circ}\text{F}$ . will raise the pour point from  $-50^{\circ}\text{F}$ . to  $-5^{\circ}\text{F}$ ., whereas the addition of the same amount of paraffin wax to a wax-free, naphthenic oil having the same viscosity and pour point does not affect the pour point at all.

## BRIEF SUMMARY

It has been found that the foregoing problems can be avoided by the process of the present invention. Accordingly, it is an object of this invention to provide an improved process for preparing high-stability insulating oils. Another object of this invention is to provide a process for preparing high-stability insulating oils having improved sensitivity to waxy contami-

nants. Still another object of this invention is to provide a process for preparing high-stability insulating oils which meet the specifications for use in transformers. Other objects will be apparent from the following description.

In accordance with this invention, the foregoing and other objects are accomplished by separating the insulating oil fraction from a previously dewaxed oil fraction. The previously dewaxed fraction, which contains the desired insulating oil fraction as a heart cut, is treated to remove aromatic and polar components prior to the dewaxing step. The dewaxed fraction is then hydrofined to improve color and oxidation stability prior to the fractionation step.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a flow diagram of the complete insulating oil preparation process.

FIG. 2 is a detailed drawing of the fractional distillation tower used to separate both the insulating oil fraction and a low-pour point light lube from the broad, dewaxed hydrocarbon oil containing these fractions.

## DETAILED DESCRIPTION

Referring to FIG. 1, a wax-containing crude oil is fed through line 1, to a crude distillation unit 11, wherein the crude oil is fractionated into several cuts. The crude cut containing the insulating oil fraction is taken from the vacuum portion of the crude still through line 2, and transferred to an extraction unit 12, wherein the aromatic and polar components content of the vacuum oil fraction is reduced by selective extraction. The extraction solvent enters the extraction unit 12 through line 3, and leaves through line 4. The raffinate goes overhead through line 5 to the dewaxer 13. In the dewaxer 13 the wax content of the vacuum oil fraction is reduced to the desired level by cooling to a predetermined temperature. As is well known in the art, the cooling causes precipitation of the wax, which is then removed by filtration. In conventional operations the wax-containing oil is diluted with a suitable solvent prior to the dewaxing step. When this expedient is employed the dewaxing solvent will enter the dewaxer 13 through line 6. The diluted oil containing precipitated wax leaves the dewaxer through line 7. The wax is then separated from the diluted oil by conventional means such as filtration, 14, and the diluent is flashed off by the use of suitable means, 15. The dewaxed oil then enters a hydrofiner, 16, through line 8. The hydrofined oil fraction is then transferred through line 9 to a fractional distillation tower 17 wherein the insulating oil fraction is separated as a heart cut, leaving the distillation tower through line 10(b). The lighter components which are unsuitable for use as an insulating oil pass overhead and leave the fractional distillation tower through 10(a). The heavier fraction which may be used as a lubricating oil contains the high-boiling wax contaminants and is removed from the fractional distillation tower as bottoms through line 10(c). As is shown in FIG. 2, the heavier fraction can be further separated into two streams. In this embodiment the high-boiling, waxy contaminants are removed as bottoms through 10(d). The remaining portion of the heavy fraction is removed through line 10(c) and is suitable for use as a low-pour point lubricating oil.

By the method of the present invention, a high-stability insulating oil fraction can be recovered from any waxy paraffinic crude. Such crude oils are obtained from Western Canada, Saudi Arabia, Kuwait, the Panhandle, North Louisiana, Tia Juana, etc.

The vacuum gas oil fraction, which is taken from the crude distillation unit, will, in general, have a 50 to 95 percent boiling range of  $550^{\circ}$  to  $750^{\circ}\text{F}$ . The pour point of this fraction will, generally, be between  $35^{\circ}$  and  $70^{\circ}\text{F}$ . This cut will, obviously, contain the insulating oil fraction sought to be recovered in the present invention. It is important, however, that this fraction containing the insulating oil fraction as a heart cut; i.e., that the vacuum gas oil fraction contain both lower- and higher-boiling materials.

The aromatic and polar components are removed from the vacuum gas oil fraction by contacting this fraction with a solvent having a preferential selectivity for these constituents. Such selective extraction operations are well known in the art. Solvents such as phenol, furfural and the like are used in these processes. Generally, from about 0.5 to 4 volumes of solvent are utilized per volume of oil. The temperature employed is, generally, in the range of 120° to 250° F. while pressures are in the range of about atmospheric to 250 p.s.i.g. In general, the solvent flows downwardly and countercurrently contacts the upflowing oil under conditions wherein the more aromatic and polar-type constituents are dissolved in the solvent. The solvent extract phase is removed from the extraction zone and is processed to segregate the solvent from the aromatic and polar type compounds. The solvent is then recycled to the treating zone. Moreover, any solvent which may be entrained in the raffinate phase is separated prior to the dewaxing step.

The dewaxing step is accomplished by means well known in the prior art. For example, the vacuum gas oil fraction may be contacted with any one or more of a number of suitable dewaxing solvents such as liquid propane, methyl ethyl ketone, methyl isobutyl ketone, or mixtures thereof to secure a product having a pour point between the range of +20° to -25° F. In general, the temperature to which the solvent oil mixture is cooled will be between +20° and -40° F. It should be noted that the pour point of the final insulating oil product will depend upon both the dewaxing temperature and the particular solvent or solvents employed.

The hydrofining step may be accomplished by contacting the dewaxed oil with a suitable hydrofining catalyst containing the sulfides or oxides of such combinations of metals as cobalt and molybdenum, nickel and molybdenum, nickel and tungsten, etc. In general, the hydrofining will be accomplished by passing the dewaxed oil over a fixed bed at a space velocity of between 0.3 and 3.0 v./v./hr., a temperature between 350° and 650° F. and under a hydrogen pressure between 300 and 900 p.s.i.g. The purpose of this step is to improve color, oxidation stability and sulfur content of the vacuum gas oil. It should be clear, that this step may be omitted. The fractionation of the dewaxed vacuum gas oil fraction is most advantageously effected by an efficient fractional distillation carried out in such a manner so as to produce a relatively sharp separation between the fractions such as to separate the dewaxed vacuum gas oil fraction into at least three fractions. In general, the insulating oil fraction should be cut so as to have a 5 to 95 percent boiling range between 600° and 700° F. It will be appreciated that the exact boiling range of this fraction will depend upon the specifications sought to be met. The higher-boiling fraction will, in general, have a 5 to 95 percent boiling range between 680° and 770° F. This fraction will contain the high-boiling contaminants accumulated in the prior processing steps, but can be used directly as a light lube oil. In general, the pour point of this material will range between 0° and -20° F. If desired, the pour point of this material can be improved by further fractionation. For example, the pour

point can be brought within the range of -20° to -50° F. by taking a second side stream product from the tower and removing the high-boiling contaminants as the bottoms product. In this embodiment, the bottoms product will range between 3 and 20 percent of the total vacuum gas oil fraction and will have a boiling range between 700° and 1,000° F., the upper limit depending on the amount and type of contamination present.

#### PREFERRED EMBODIMENTS

The invention will be more apparent from the preferred embodiment, which is illustrated by example 1 and the comparative data set forth below.

#### EXAMPLE 1

With reference to FIG. 1, in this example, a waxy Western Canadian crude (Pembina) was fed through line 1, to a conventional refinery crude distillation unit II. A vacuum gas oil fraction comprising 2.67 percent of the crude feed and having a 5 to 95 percent boiling range of 579°-715° F. was recovered through line 2. The boiling range of this fraction was determined in a 15-plate distillation column operated at a 5 to 1 reflux ratio. The pour point of the vacuum gas oil fraction was +50° F. The vacuum gas oil fraction was then fed to an extraction unit 12, wherein the excess aromatic and polar components were removed by phenol extraction. The volume ratio of phenol, entering through line 3, and the vacuum gas oil fraction entering through line 2 was 1.2 to 1. Twenty-seven percent of the vacuum gas oil fraction was removed in the extract phase, through line 4. The remaining 73 percent was recovered from the raffinate phase and fed to the dewaxing unit 13 through line 5. The raffinate product was diluted with a 50/50 mixture of methyl ethyl ketone and methyl isobutyl ketone. The solvent to oil ratio was 1.5 to 1. The oil solvent mixture was cooled to -10° F. The precipitated wax was removed by filtration 14 and the ketone solvent removed by distillation 15. The dewaxed oil was recovered through line 8. The yield was 72 percent based on feed to the dewaxing unit of a dewaxed oil having a pour point of -15° F., a cloud point of -12° F., a gravity of 34.0° API, and a viscosity of 55.1 SUS at 100° F. It contained about 14 percent of aromatic components and 0.07 percent sulfur. The dewaxed oil was then hydrofined by contacting with a typical sulfided cobalt-molybdate catalyst to improve its color and oxidation stability and to reduce its sulfur content. The dewaxed oil was fed through line 8 to the hydrofiner 16. A space velocity of 1.0 v./v./hr., a temperature of 450° F., and a hydrogen pressure of 400 p.s.i.g. were used. The product obtained through line 9 had a sulfur content of 0.035 percent. The hydrofined product was then fed through line 9 to the fractional distillation tower 17. The fractionation column was designed such that the degree of separation was equivalent to that which would be obtained by the use of a 15-plate batch column operated at a 2 to 1 reflux ratio. In the preferred embodiment, four fractions were taken from this column; (a) the fraction distilling below 621° F.

TABLE I.—INSPECTIONS OF TRANSFORMER OIL FROM PARAFFINIC CRUDE

Test	ASTM method	Inspection result	CSA specification C50-1965
Flash point (open cup) ° F.....	D 92-57	355	290 min.
Fire point (open cup) ° F.....	D 92-57	>355	320 min.
Viscosity at 100° F., SUS.....	D 88-56	56.7	62.0 max.
Viscosity at 40° F., extrap., SUS.....	D 341-43	170	240 max.
Pour point, ° F.....	D 97-57	-50	-50 max.
ASTM color.....	D 1500-64	<1	1 max.
Dielectric strength, kv.....	D 877-64	>30	26 min.
Interfacial tension, dynes/cm.....	D 971-50	40.8	35 min.
Specific gravity, 60° F.....	D 1298-55	0.852	0.906 max.
Neutralization No. mg. KOH/g.....	D 974-64	<0.01	0.03 max.
Corrosive sulphur.....	D 1275-64	(1)	Non-corrosive.
Stability, visible sludge/interfacial tension/ neut. No.	D 1314-54 <sup>2</sup>	NIL/40.7/0.01	NIL/≥20/≥0.05.

<sup>1</sup> Non-corrosive.

<sup>2</sup> 24 hours.

The fraction (c) meets all specifications for a low pour point light lube oil.

(29.6 LV percent), (b) the fraction distilling between 621° and 685° F. (49.8 LV percent), and (c) the fraction distilling between 685° and 730° F. (15.0 LV percent), and (d) the residual fraction (5.6 LV percent). These fractions had the following 5 to 95 percent boiling ranges as determined with a 15-plate distillation column operated at a reflux ratio of 5 to 1: (a) 553° to 635° F., (b) 616° to 685° F., (c) 675° to 730° F., and (d) 715° to 780° F. Their pour points and viscosities at 100° F. were: (a) -5° F., 44.6 SUS; (b) <-30° F., 56.8 SUS; (c) -35° F., 78.5 SUS and (d) 15° F., 105 SUS.

When 0.06 percent of a 2,6-di-tertiary butyl paracresol oxidation inhibitor and 0.07 percent of a wax-alkylated naphthalene pour depressant were added to fraction (b), which was obtained in a yield of 0.7 percent based on crude, it met all Canadian Standards Association specifications (C50-1965 for transformer oils. The inspections and specifications are given in table I.

#### EXAMPLE 2

This example was carried out on the same feed and same conditions as set forth in example 1, except that only three fractions were obtained in the final distillation step. These fractions were: (a) the fraction distilling below 621° F. (29.6 LV percent), (b) the fraction distilling between 621° and 685° F. (49.8 LV percent), and (c) the residual fraction (20.6 LV percent). These fractions had the following 5 to 95 percent boiling ranges as determined by a 15/5 distillation: (a) 553° to 635° F., (b) 616° to 685° F., and (c) 678° to 748° F. Their pour points and viscosities at 100° F. were: (a) -5° F., 44.6 SUS; (b) less than -30° F., 56.8 SUS; (c) -20° F., 83.9 SUS. It will be noted that the fraction (b) has properties identical with those of the insulating oil fraction obtained in example 1. The fraction (c) has a slightly higher pour point and viscosity but was obtained in higher yields and still meets specifications as a light-lubricating oil.

#### EXAMPLE 3

In this example, the feedstock and conditions were the same as those in example 1, except that the hydrofining step was omitted. In this example, the insulating oil fraction, fraction (b), was obtained with a boiling range of 610° to 682° F. in a yield of 49.9 LV percent. This fraction had a viscosity of 57.0 SUS at 100° F. and a pour point of -55° F. without pour depressants. Its oxidation stability was, however, inferior to that of the oil which was hydrofined before distillation. It will be apparent that either of the product streams obtained by the process of this invention may be used directly as either an insulating oil or a light lube oil. In most cases, however, the superior products will be obtained by the addition of both an oxidation inhibitor and a pour point depressant. For this purpose, any of the well-known additives may be used. For example, suitable oxidation inhibitors include the butylated hydroxytoluenes, and suitable pour point depressants include the polyacrylates, wax-alkylated naphthalenes, etc.

Having thus described and illustrated this invention, what is claimed is:

1. The process for the preparation of a low-pour point insulating oil which comprises:

1. solvent extracting a broad waxy paraffinic vacuum gas oil fraction having a 5 LV percent to 95 LV percent boiling range between 550°-750° F. at atmospheric pressure;

2. dewaxing said extracted vacuum gas oil fraction to a pour point of +20° to -25° F.;

3. hydrofining said dewaxed, extracted vacuum gas oil fraction with hydrogen and a hydrogenation catalyst, said catalyst comprising hydrogenation components selected from the group consisting of the oxides and sulfides of metals of the sixth and eighth groups of the Periodic Table and mixtures thereof, and

4. distilling said hydrofined dewaxed oil and recovering as a heart cut an insulating oil having a 5 LV percent to 95 LV percent boiling range between 580°-720° F. at atmospheric pressure, thus obtaining further pour point improvement relative to step 2.

2. The process of claim 1 wherein the vacuum gas oil fraction has a 5LV percent to 95 LV percent boiling range of 579°-715° F. at atmospheric pressure.

3. The process of claim 1 wherein the vacuum gas oil is dewaxed at a temperature between about +20° to -40° F.

4. The process of claim 1 wherein said hydrofining is conducted at a temperature between about 350° and 650° F. and at a hydrogen pressure between about 300 to 900 p.s.i.g.

5. The process of claim 1 wherein the insulating oil has a 5 LV percent to 95 LV percent boiling range between 616°-685° F. at atmospheric pressure.

6. The process of claim 1 wherein said heart cut is recovered in more than one fraction.

7. The process of claim 1 wherein the extraction solvent is selected from the group consisting of phenol and furfural.

8. A process for preparing a low-pour point insulating oil which comprises the steps of (1) solvent-extracting a broad waxy paraffinic vacuum gas oil fraction having a 5 LV percent to 95 LV percent boiling range of 550°-750° F. at atmospheric pressure, (2) dewaxing said extracted vacuum gas oil fraction to a pour point between the range of +20° to -25° F., and then (3) distilling the dewaxed fractions and recovering as a heart cut an insulating oil fraction having a 5 LV percent to 95 LV percent boiling range of 580°-720° F. at atmospheric pressure, thus obtaining further pour point improvement relative to step 2.

9. The process of claim 8 wherein the heart cut is recovered in more than one fraction.

10. The process of claim 8 wherein the vacuum gas oil fraction has a 5 LV percent to 95 LV percent boiling range of 579°-715° F. at atmospheric pressure, and the insulating oil is obtained with a 5 LV percent to 95 LV percent boiling range of 616°-685° F. at atmospheric pressure.

11. The process of claim 8 wherein the extraction solvent is selected from the group consisting of phenol and furfural.

\* \* \* \* \*

60

65

70

75