

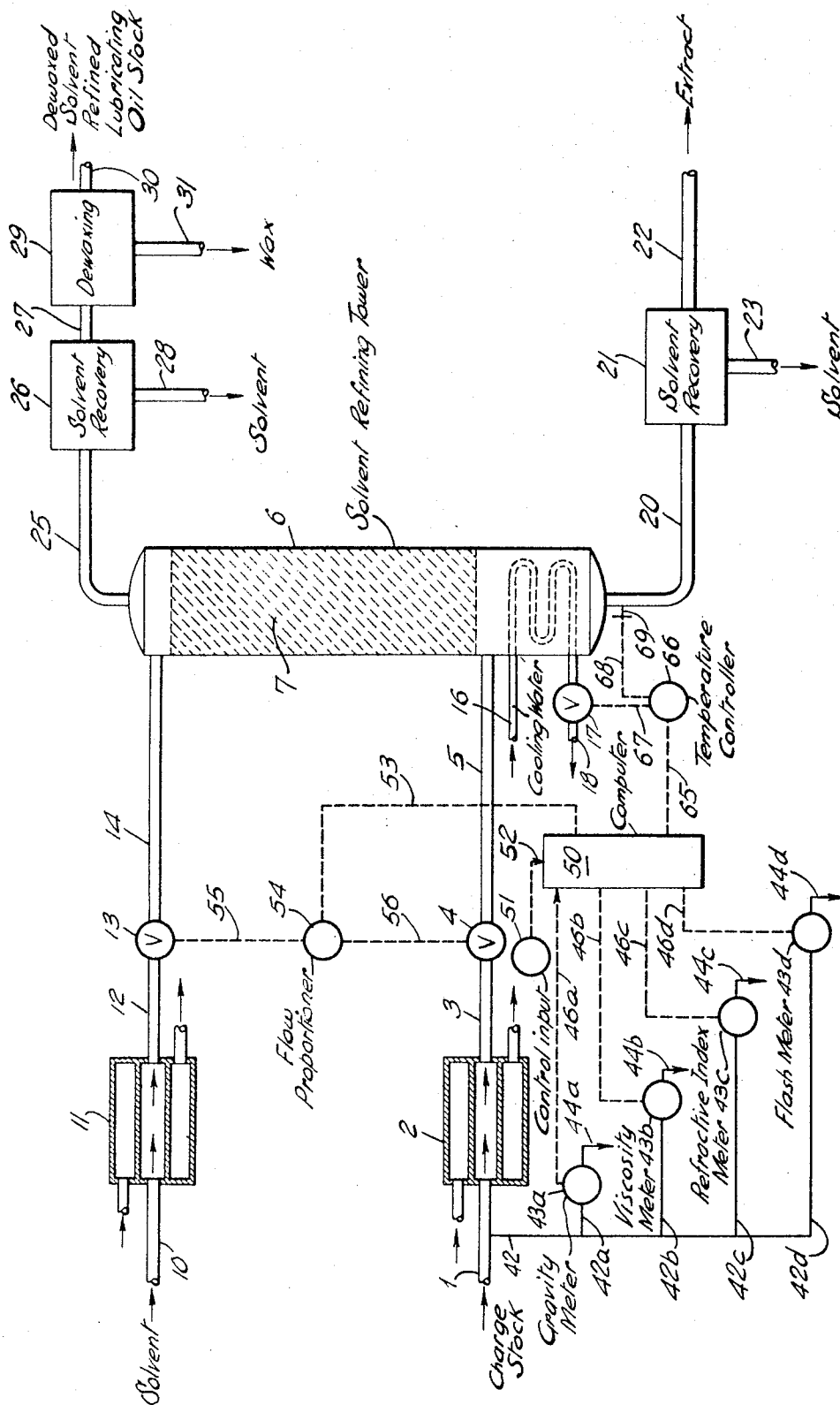
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LUBE OIL REFINING PROCESS

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LUBE OIL REFINING PROCESS

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ABSTRACT OF THE DISCLOSURE

Waxy feed stock oil is first dosed, or contacted, with a solvent to effect a separation of the waxy oil and solvent into a raffinate stream and an extract-mix stream. Second, the solvent is recovered from the raffinate to yield a refined solvent-free waxy oil. Third, the refined solvent-free waxy oil is dewaxed to yield lubricating oil. Signals representative of the measure of the quality, or measure of the various characteristics, of the undosed waxy feed stock oil (such as, for example, API gravity, viscosity, refractive index and flash point) being charged to a solvent dosing tower are fed to a computer. Also, a signal representative of a target, or preselected, viscosity index is fed to the computer; the target viscosity index being that viscosity index which the lubricating oil is to have ultimately. The computer processes the signals in accordance with a mathematical model developed for the process and output signals emanating from the computer are employed to control solvent dosage and the outlet temperature of the extract-mix stream in the solvent dosing tower. As a result the lubricating oil yielded from the third, or dewaxing, operation has the target viscosity index.

This is a continuation-in-part application of the continuation application S.N. 575,191, filed Aug. 25, 1966; the continuation application having derived from parent application S.N. 363,149, filed Apr. 28, 1964 now abandoned.

This invention relates to a hydrocarbon treating process. More particularly, it relates to a solvent refining process wherein the conditions for refining a lubricating oil charge stock, or feed stock, are controlled in response to the charge stock quality. In accordance with this process, the solvent dosage and the extract-mix outlet temperature are controlled to produce a product having a specific viscosity index upon subsequent dewaxing in response to signals generated in accordance with characteristics of the charge stock treated.

Two important characteristics of lubricating oils are fluidity at the temperatures of use and the ability to maintain adequate lubrication with variations of temperatures within the temperature ranges encountered in use. Criteria of these characteristics are the pour point (ASTM designation: D-97) and the viscosity index, VI (ASTM designation: D-567). Petroleum crude oils which are used in the manufacture of lubricating oils contain wax or waxy materials which adversely affect the pour point and substances of low hydrogen-to-carbon ratio which adversely affect the viscosity index. To produce lubricating oils of high quality, the petroleum refiner employs a solvent refining process to remove constituents of low viscosity index and a process such as solvent dewaxing to remove wax or waxy materials. Since dewaxing is a relatively expensive process and solvent refining removes a substantial amount of material from the lubricating oil stock, it is usually more economical to employ solvent refining first and then to subject the reduced quantity of solvent refined oil to dewaxing. However, since the waxy constituents of lubri-

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cating oil stocks have a high hydrogen-to-carbon ratio, dewaxing substantially changes the viscosity index of the lubricating oil stock. The refiner, then, usually solvent refines a lubricating oil stock to a considerably higher viscosity index than the finished lubricating oil product so that the oil after dewaxing will have the desired viscosity index.

For example, in furfural refining lubricating oil stocks, furfural dosages within the range of about 75 to 500 volume percent of the oil charge and extract-mix outlet temperatures within the range of about 100 to 300° F. are used. We have found that the furfural refining solvent dosage and extract-mix outlet temperature required may be correlated with the quality of the feed stock and with the concentration of oil in the extract-mix. One index of feed stock quality is the characterization factor, K_v , described by Watson et al., Ind. Eng. Chem., 25, 880 (1933), 27, 1460 (1935). Another index of feed quality is the refractive index, RI (ASTM designation: D-1218). Although refractive index alone is of very limited value in defining feed stock quality, we have developed useful and accurate relationships using RI together with the flash point (ASTM designation: D-92) of the waxy charge stock for predicting the extract oil yield corresponding to any desired dewaxed refined oil VI, hereinafter referred to as the target VI and designated VI_{targ} . The four tests required for the control of a furfural refining unit are the density, the viscosity, the refractive index and the flash point, all on the waxy feed stock. The use of these values in the control of a furfural refining unit is described in greater detail hereinafter.

Also, N-methyl-2-pyrrolidone (hereinafter referred to as NMP) instead of furfural may be used as the solvent for dosing the feed stock. The NMP refining solvent dosage and extract-mix outlet temperature may also be correlated with the quality of the feed stock and concentration of oil in the extract-mix; said correlation being related to the relationships, hereinbefore, mentioned for the use of furfural as a solvent. We have also developed useful and accurate relationships for NMP, correlated in relation to the use of furfural, using RI together with flash point (ASTM designation: D-92) of the waxy feed, or charge, stock for predicting the extract oil yield corresponding to any desired, or specified, dewaxed refined oil VI (VI_{targ}). The tests required for the control of an NMP refining unit are the density, viscosity, refractive index and flash point—all on the waxy feed stock. The control of the NMP refining unit is hereinafter described in greater detail.

An objective of this invention is to provide a means for controlling a solvent refining unit in response to charge stock quality.

A further objective is to change operating conditions on a given feed stock when the desired viscosity index, VI_{targ} , or the dewaxed and refined oil is changed such that product of the desired quality is immediately produced. Our invention provides for producing a refined oil product which has a desired VI on a dewaxed basis by control of the solvent dosage and extract-mix outlet temperature in accordance with charge stock quality. An advantage of this method of control is that it effectively anticipates changes in required refining conditions and eliminates the time lag inherent in control in response to product quality. If the VI_{targ} is changed, operating conditions are changed quickly to those required for the new product quality. This quick response maintains uniformity of product quality and maintains optimum refining conditions.

Another objective of this invention is to provide a mathematical model of a solvent refining process whereby completely automatic computer control may be effected.

Another objective of this invention is to provide a

method for controlling a furfural refining unit in response to charge, or feed, stock quality.

Another objective of this invention is to provide a method for controlling an NMP refining unit in response to charge, or feed, stock quality.

Another objective of this invention is to provide predictive, or feedforward—as distinguished from a feedback, control of a solvent refining tower to ultimately obtain, after dewaxing following the refining tower processing, lubricating oil of a specified, target, viscosity index.

We have found that the furfural refining of lubricating oil stocks at optimum processing conditions may be expressed by the following equation:

$$A = C_e T_R F^{0.5} \quad \text{Eq. 1}$$

where:

A is a constant of characteristic value for each charge stock;

C_e = concentration of extract oil in the extract-mix;

T_R is the difference between the extract-mix outlet temperature and the boiling point of furfural, 323° F.; and

F = furfural dosage, volumes of furfural charged to the refining tower per 100 volumes of charge stock.

In this and the following equations a term is defined when first used and has the same meaning in every succeeding use.

The value of A is Eq. 1 was found to correlate with The Watson Characterization Factor, K_v , according to the following equation:

$$A = 6099.74 - 487.58 K_v \quad \text{Eq. 2}$$

where:

K_v = Watson Characterization Factor

The following equation of K_v has been developed in terms of the API gravity (ASTM designation: D-287) and the kinematic viscosity (ASTM designation: D-445);

$$K_v = 9.2767 + 0.0765 \text{ } ^\circ\text{API} + 0.3733 \ln(\text{cs.}) - 0.0254 (\ln \text{cs.})^2 + (0.0021 \text{ } ^\circ\text{API}) (\ln \text{cs.}) \quad \text{Eq. 3}$$

where:

$^\circ\text{API}$ = Gravity in $^\circ\text{API}$ (ASTM designation: D-287); and
 cs. = Kinematic viscosity at 210° F. in centistokes (ASTM designation: D-445).

As brought out above, C_e is the concentration of the extract oil in the extract-mix leaving the bottom of the refining tower. It is determined from the relationship:

$$C_e = \frac{Y_{EO}}{Y_{EO} + F} \quad \text{Eq. 4}$$

where:

Y_{EO} = Percentage yield of extract oil; and
 F = Furfural dosage.

The concentration of extract oil in the extract-furfural mixture experienced in conventional commercial operation is related to the value of A in Eq. 1 by the following simple relationship:

$$C_e = 0.0004 A \quad \text{Eq. 5}$$

The yield of extract oil is a function of the VI of the dewaxed refined oil as expressed by the following relationship:

$$Y_{EO} = f VI_{\text{targ}} \quad \text{Eq. 6}$$

We have found that the yield of extract oil may be calculated from four quantities which are related to feed stock quality and VI_{targ} . These have been designated by the letters G, L, M and N in the equations which follow. The value of G is related to the refractive index of the

waxy charge, RI_{WC} , and the Cleveland Open Cup Flash Point, fl, in $^\circ\text{F.}$ by the following equation:

$$G = RI_{WC} - 0.0001(\text{fl}) \quad \text{Eq. 7}$$

where:

RI_{WC} is the refractive index at 70° C. of the waxy charge (ASTM designation: D-1218); and
 fl is the Cleveland Open Cup flash, $^\circ\text{F.}$ (ASTM designation: D-92).

The value of L is related to G by the following equation:

$$L = 0.041 - 1.11 (G - 1.4200) \quad \text{Eq. 8}$$

The value of M is related to the VI of the dewaxed refined oil, i.e., VI_{targ} by the following equation:

$$M = C_1 + C_2 (100 - VI_{\text{targ}}) \quad \text{Eq. 9}$$

where:

C_1 = .805 for distillate charge stocks having a flash point less than 450° F., .794 for distillate charge stocks with a flash point equal to or greater than 450° F., .801 for residual charge stocks; and

C_2 = 0.001155 for distillate charge stocks having a flash point less than 450° F., .001445 for distillates having flash points equal to or greater than 450° F., and .001190 for residual charge stocks.

The value of N is simply the sum of L and M as indicated by the following equation:

$$N = L + M \quad \text{Eq. 10}$$

The yield of extract oil is determined from the value of N which, in turn, is a function of G, L and M as shown by Equations 7, 8 and 9. The relationship between Y_{EO} and N is expressed by the following equation:

$$Y_{EO} = 1154 (0.857 - N) \quad \text{Eq. 11}$$

The yield of extract oil determined by Eq. 11 and the concentration of the extract oil in the extract-mix determined from Eq. 5 determine the furfural dosage in accordance with Eq. 4 which may be rearranged in the following form:

$$F = Y_{EO} \left(\frac{1}{C_e} - 1 \right) \quad \text{Eq. 12}$$

Since T_R is the difference between the boiling point of furfural (323° F.) and the temperature, T_{EO} , of the extract-mix leaving the refining tower, T_{EO} is determined by Eq. 1 rearranged as follows:

$$T_{EO} = 323 - \frac{A}{C_e F^{0.5}} \quad \text{Eq. 13}$$

In summary, then, it may be stated that the furfural dosage F, and the extract oil outlet temperature T_{EO} , required to produce a lubricating oil which will have a specific viscosity index when dewaxed may be determined from the density ($^\circ\text{API}$), viscosity (cs.), refractive index (RI) and flash point (fl) data obtained on the waxy charge to a furfural refining process by the following successive steps:

Step 1: The Watson characterization value K_v is determined from density ($^\circ\text{API}$) and viscosity (cs.) determinations by Eq. 3,

Step 2: Value of the constant A in Eq. 1 is determined from the above determined value of K_v and Eq. 2.

Step 3: The value of C_e in Eq. 4 is determined from Eq. 5 by the value of A determined in Step 2,

Step 4: The extract oil yield, Y_{EO} , is determined from values of G, L, M and N which in turn are determined as follows:

Step 4a: G is determined from the measured flash point of the waxy charge by Eq. 7,

Step 4b: This value of G is used to determine the value of L by Eq. 8,

Step 4c: The value of M is determined from Eq. 9 using the VI_{targ} , i.e., the desired VI of the dewaxed

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refined oil, and the fixed values of the constants C_1 and C_2 .

Step 4d: The values of L and M are added to obtain a value for N according to Eq. 10,

Step 4e: This value of N is used to determine the yield of extract oil, Y_{EO} , from Eq. 11,

Step 5: Values of Y_{EO} determined by Steps 4a-e and C_e determined in Step 3 are used to determine the furfural dosage,

Step 6. The temperature of the extract oil, T_{EO} , leaving the refining tower is derived from Eq. 13 using the value of A determined in Step 2, value of C_e determined in Step 3 and the value of F determined in Step 5.

Obviously the foregoing six steps may be performed manually and since each operation is expressed as an equation, the entire series of determinations may be made by a computer for automatic control.

The accompanying drawing diagrammatically illustrates one form of the process of this invention. Although the drawing illustrates one arrangement of apparatus in which the process of this invention may be practiced, it is not intended to limit the invention to the particular materials or apparatus described.

Charge stock in line 1 is heated to the desired operating temperature in exchanger 2 and passed through line 3, valve 4 and line 5 into furfural refining tower 6. Solvent in line 10 is heated to the desired temperature in exchanger 11 and passed through line 12, valve 13 and line 14 to refining tower 6. Refining tower 6 is illustrated as a simple countercurrent contacting tower containing packing 7. However, other contacting devices may be employed, for example, rotating disc contactors, centrifugal countercurrent contactors, or box countercurrent contactors. In tower 6 the oil and solvent are contacted in countercurrent flow effecting extraction of low viscosity index constituents of the charge oil. Raffinate comprising refined oil and containing a small amount of dissolved solvent is withdrawn through line 25. A temperature gradient is maintained in furfural refining tower 6 by means of a cooling coil 16 in the bottom of the tower. Other means of achieving a temperature gradient may be employed in place of or in addition to a cooling coil, for example, by introducing feed at lower temperature than the solvent, by introducing a cold reflux steam or by withdrawing a side stream of liquid from the tower, cooling the withdrawn stream and reintroducing it into the tower. Cooling water introduced into coil 16 at a rate controlled by valve 17 is discharged through line 18. Extract-mix comprising solvent and dissolved low viscosity index constituents of the oil feed is withdrawn through line 20 at a temperature controlled by cooling coil 16.

Raffinate in line 25 is passed to solvent recovery facility 26 wherein the solvent is stripped from the refined oil. Solvent refined oil is discharged through line 27 and solvent is withdrawn through line 28 for return to solvent line 10 and re-use. Solvent refined oil in line 27 is passed to dewaxing facility 29 wherein wax is separated, for example, by solvent dewaxing employing chilling with a methylethyl ketonebenzene solvent mixture and filtration of precipitated wax. Dewaxed, solvent refined lubricating oil stock is discharged through line 30 for storage and blending of product lubricating oils, and wax is discharged through line 31 for further processing or use.

Extract-mix in line 20 is passed to solvent recovery facility 21 wherein the solvent is stripped from the extract oil which is discharged through line 22 and recovered solvent is withdrawn through line 23 for return to line 10 and re-use.

A small stream of the charge stock is continuously withdrawn through sample line 42 and passed by branch lines 42a, 42b, 42c and 42d to analytical meters 43a, 43b, 43c and 43d, respectively. Effluent from the analytical meters is discharged through lines 44a, 44b, 44c, and 44d, respectively, for return to the charge sys-

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tem or for disposal as slop if this is more convenient. Meter 43a is a gravity meter which continuously determines the density of the charge stock and generates a control signal which is a measure of the gravity in terms of °API. Gravity meters which may be used for this purpose include, for example, the Arcco model R Gravitometer, Fisher Gravitometer and Automation Products Dynatrol model CL10RQ. The control signal is transmitted by conduit 46a to computer 50. The signal conduit may be, for example, a tube, a wire or a lever depending upon the nature of the signal, that is, whether it be pneumatic, hydraulic, electrical or mechanical. The use of electric signals is preferred since they may be introduced directly into the computer system.

Meter 43b determines the viscosity of the oil at some convenient temperature, for example, at 130° F. or 210° F., and transmits the corresponding signal to computer 50 through conduit 46b. Viscosity meters which may be used include, for example, that described by J. M. Jones, Jr., in U.S. Patents 2,791,902 and 3,025,232. Meter 43c determines the refractive index of the charge and transmits a signal in response thereto to computer 50 through conduit 46c. A suitable refractive index meter is described by G. C. Eltenton in U.S. Patent 2,569,127. Meter 43d determines the Cleveland Open Cup Flash Point of the wax charge and transmits a signal in response thereto to computer 50 through conduit 46d. A suitable meter for this purpose is the Precision Scientific Automatic Flash Tester recalibrated for Cleveland Open Cup Flash Point. Additional control input data such as the target VI of the dewaxed refined oil is introduced into computer 50 through conduit 52 from control input 51.

The output of computer 50 comprises a signal controlling the furfural dosage through conduit 53 and a signal controlling the extract outlet mix temperature through conduit 65. The signal in conduit 53 sets the control point in the flow proportioner 54 to maintain the desired ratio between solvent flowing in line 14 and waxy charge flowing in line 5. The flow rate for the solvent is set at some near limiting value and is maintained at that value by a signal through conduit 55 to valve 13. The ratio determining signal through conduit 56 to valve 4 maintains the desired charge rate.

The signal controlling the temperature of the extract-mix is transmitted through conduit 65 to set the control point of temperature controller 66. The temperature of the extract-mix leaving tower 6 is determined by temperature sensing means 69 and a signal responsive thereto is transmitted through conduit 68 to temperature controller 66. Temperature controller 66 then transmits a control signal through conduit 67 adjusting valve 17 to maintain the temperature called for by the control signal transmitted through conduit 65.

EXAMPLE 1

A heavy wax distillate is solvent refined with furfural and dewaxed to produce a solvent refined dewaxed lubricating oil stock having a viscosity index of 95. The following data are obtained on the waxy feed by means of meters determining the gravity, viscosity, refractive index and flash point.

Density	-----°API--	22.2
Viscosity SUS at 210° F.	-----sec--	87.8
RI at 70° C.	-----	1.4940
Flash, COC	-----°F--	520

The yield of extract oil that is produced from this feed stock at a target VI of 95.0 is calculated as follows:

By the six step procedure described above, The Watson Characterization Factor K_w is determined to be 12.0 by Eq. 3. The value of A by Eq. 2 is 244. Substituting this value of A in Eq. 4, C_e is found to be 0.0976.

G is determined to be 1.4420 by Eq. 7. A value of $L=0.017$ is determined from Eq. 8. The value of M is found to be 0.8012 by Eq. 9. The value of N therefore is

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0.8012+0.017 or 0.8182. The extract oil yield, Y_{EO} , is found to be 54.0 percent by substitution of the value of N in Eq. 11. The values for Y_{EO} and C_e , having been determined, the furfural dosage is found to be 409 percent by Eq. 12. The temperature of extract oil is found to be 200° F. by Eq. 13. Computer signals then set the temperature control of the extract-mix outlet at 200° F. and the flow proportioner to give a furfural dosage 409 percent of the charge rate. The raffinate oil after dewaxing is found to have the target VI of 95.0.

Upon switching charge stock tanks, the feed stock quality meters detect a slight change in quality, the new stock having the following characteristics:

Density	-----°API--	21.7
Viscosity SUS at 210° F.	-----	89.3
RI at 70° C.	-----	1.4957
Flash, COC	-----F°--	500

The processing of this feed to a target VI of 95.0 on the dewaxed refined oil is continued. The tests indicated by the charge meters results in new computed values for G , L and N . The original values and new values compare as follows:

	Original value	New value
G -----	1.4420	1.4457
L -----	0.0170	0.0125
N -----	0.8182	0.8137
M -----	0.8012	0.8012

The value of M remains unchanged since it is a function of the target VI. From these values, an extract oil yield, Y_{EO} , of 49.5 percent is calculated. The K_v is 11.95. The value of A is 268; the value of C_e is 0.1172. From these results the desired furfural dosage is redetermined to be 381 percent of the charge rate and the temperature of the extract-mix outlet to be 206° F. The flow proportioner is reset by the computer to reduce the ratio of furfural to fresh feed from 4.09:1 to 3.81:1 and the temperature controller is reset by the computer to maintain the temperature of the extract-mix leaving the furfural treating tower at 206° F. rather than 200° F.

After processing a portion of the second tank of heavy wax distillate the target VI of the dewaxed refined oil is charged from 95.0 to 90.0 for manufacture of a different product. Values for G , L , M and N are computed to be 1.4457, 0.0125, 0.8084 and 0.8209, respectively, from which the yield of extract oil is computed to be 41.5 percent. The values K_v , A and C_e remain unchanged. The furfural dosage is redetermined to be 268 percent and the temperature of the extract-mix leaving the furfural treating tower is redetermined to be 195° F. for this operation. Accordingly, the computer resets the flow controller to change the ratio of furfural to fresh feed from 3.81:1 to 2.68:1 and resets the temperature controller on the extract-mix outlet to maintain a temperature of 195° F. rather than 206° F. These changes in furfural dosage and extract oil outlet temperature result in a refined oil which has a VI of 90.0 upon dewaxing.

EXAMPLE 2

A light waxy distillate is furfural refined to produce a target VI of 90 on the dewaxed refined oil. The following tests are obtained on the waxy charge stock:

Density	-----°API--	24.4
Viscosity SUS at 210° F.	-----sec--	48.8
RI at 70° C.	-----	1.4838
Flash, COC	-----°F--	415

The yield of extract oil which is obtained from this feed stock at a target VI of 90 is calculated from Equations 7, 8, 9, 10 and 11 to be 28.2 percent. The values of G , L , M and N are respectively, 1.4423, 0.016, 0.8165 and 0.8325. K_v is determined to be 11.90 by Eq. 3. A is found to have a value of 293 by Eq. 2 and C_e is found to be 0.1172 by Eq. 5. From these results the furfural dosage is com-

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puted to be 224 percent and the extract-mix outlet temperature is 156° F. The computer sets the flow proportioner to maintain a furfural to waxy feed ratio of 2.24:1 and sets the temperature controller to maintain an extract-mix outlet temperature of 156° F. Under these conditions a product is made which upon dewaxing has a VI of 90.0.

We have also found that the NMP refining of lubricating oil stocks at optimum processing conditions may be expressed as follows:

$$A_{NMP} = C_e T_R S^{0.4} \quad \text{Eq. 14}$$

where:

A_{NMP} is a constant of characteristic value for each charge stock;

C_e = concentration of extract oil in the extract-mix;

T_R is the difference between the extract-mix outlet temperature and the boiling point of NMP, 300° F.; and

S = NMP dosage, volumes of NMP charged to the refining tower per 100 volumes of charge stock.

Furthermore we have found that if the A value using furfural dosing is known or can be predicted from physical properties such as, for example, gravity and viscosity, of a particular charge stock then a value can be predicted for A_{NMP} . For example, we have found that there exists the relationship:

$$A_{NMP} = 0.9A \quad \text{Eq. 15}$$

Hence, using Eq. 15, supra, to transform Eq. 2, A_{NMP} as a function of K_v is:

$$A_{NMP} = 0.9(6099.74 - 487.58 K_v) \quad \text{Eq. 16}$$

where K_v is defined by Eq. 3.

Similar to Eq. 4, C_e is determined from the relationship

$$C_e = \frac{Y_{EO}}{Y_{EO} + S} \quad \text{Eq. 17}$$

where Y_{EO} and S are as hereinbefore defined.

The concentration of oil in the extract-NMP mixture experienced in conventional commercial operation is related to the value of A_{NMP} , as defined in Equations 5 and 15 by the following equations:

$$C_e = \frac{0.0004}{0.9} A_{NMP} \quad \text{Eq. 18}$$

Also the yield of extract oil is a function of the VI of the dewaxed refined oil as expressed by the following relationship:

$$Y_{EO} = fVI_{\text{targ}} \quad \text{Eq. 6A}$$

We have found that the yield of extract oil may be calculated from four quantities which are related to feed stock quality and VI_{targ} . These have been designated by the letters G , L , M and N in the equations which follow. The value of G is related to the refractive index of the waxy charge, RI_{WC} , and the Cleveland Open Cup Flash Point, fl , in ° F. by the following equation:

$$G = RI_{WC} - 0.0001 (fl) \quad \text{Eq. 7A}$$

where:

RI_{WC} is the refractive index of the waxy charge (ASTM designation: D-1218); and

fl is the Cleveland Open Cup flash, ° F. (ASTM designation: D-92).

The value of L is related to G by the following equation:

$$L = 0.041 - 1.11 (G - 1.4200) \quad \text{Eq. 8A}$$

The value of M is related to the VI of the dewaxed refined oil, i.e., VI_{targ} by the following equation:

$$M=C_1+C_2(100-VI_{targ}) \quad \text{Eq. 9A}$$

where:

$C_1=.805$ for distillate charge stocks having a flash point less than 450°F ., $.794$ for distillate charge stocks with a flash point equal to or greater than 450°F ., $.801$ for residual charge stocks; and
 $C_2=0.001155$ for distillate charge stocks having a flash point less than 450°F ., $.001445$ for distillates having flash points equal to or greater than 450°F ., and $.001190$ for residual charge stocks.

The value of N is simply the sum of L and M as indicated by the following equation:

$$N=L+M \quad \text{Eq. 10A}$$

The yield of extract oil is determined from the value of N which, in turn, is a function of G, L and M as shown

Step 4e: This value of N is used to determine the yield of extract oil, Y_{EO} , from Eq. 11A.

Step 5: Values of Y_{EO} determined by Steps 4a-e and C_e determined in Step 3 are used to determine the NMP dosage, S, by Eq. 19.

Step 6: The temperature of the extract oil, T_{EO} , leaving the refining tower is derived from Equation 20 using the value of A_{NMP} determined in Step 2, value of C_e determined in Step 3 and the value of S determined in Step 5.

Obviously the foregoing six steps may be performed manually and since each operation is expressed as an equation, the entire series of determinations may be made by a computer for automatic control.

The accompanying drawing diagrammatically illustrates one form of the process of this invention. Although the drawing illustrates one arrangement of apparatus in which the process of this invention may be practiced, it is not intended to limit the invention to the particular materials or apparatus described. The process using NMP is the same as that hereinbefore described for furfural. Examples of NMP refining follow.

EXAMPLE I.—NMP REFINING OF A WAXY DISTILLATE

Run No.	I	II	III	IV	V	VI	VII
(S) NMP dosage, vol. percent	107	200	298	407	108	299	403
(T_{EO}) ext. out temp., $^\circ\text{F}$	200	200	200	200	180	180	180
(Y_{EO}) ref'd oil yield, vol. percent	60	45	37	19.5	70	45	37
C_e	0.290	0.222	0.178	0.165	0.234	0.159	0.136
$C_e S^{0.4}$	1.88	1.85	1.74	1.85	1.52	1.56	1.50
$T_R, ^\circ\text{F}$	100	100	100	100	120	120	120
$A_{NMP}=C_e T_R S^{0.4}$	188	185	174	182	182	187	180

by Equations 7, 8 and 9. The relationship between Y_{EO} and N is expressed by the following equation:

$$Y_{EO}=1154(0.857-N) \quad \text{Eq. 11A}$$

The yield of extract oil determined by Eq. 11A and the concentration of the extract oil in the extract-mix determined from Eq. 18 determine the NMP dosage in accordance with Eq. 17 which may be rearranged in the following form:

$$S=Y_{EO} \frac{1}{C_e} - 1 \quad \text{Eq. 19}$$

Since T_R is the difference between the boiling point of NMP (300°F .) and the temperature, T_{EO} , of the extract-mix leaving the refining tower, T_{EO} is determined by Eq. 14 rearranged as follows:

$$T_{EO}=300-\frac{A_{NMP}}{C_e S^{0.4}} \quad \text{Eq. 20}$$

In summary, then, it may be stated that the NMP dosage, S, and the extract oil outlet temperature T_{EO} , required to produce a lubricating oil which will have a specific viscosity index when dewaxed may be determined from the density ($^\circ\text{API}$), viscosity (cs.), refractive index (RI) and flash point (fl) data obtained on the waxy charge to an NMP refining process by the following successive steps:

Step 1: The Watson Characterization value K_v is determined from density ($^\circ\text{API}$) and viscosity (cs.) determinations by Eq. 3.

Step 2: Value of the constant A_{NMP} in Eq. 14 is determined from the above determined value of K_v and Eq. 16.

Step 3: The value of C_e in Eq. 17 is determined from Eq. 18 by the value of A_{NMP} determined in Step 2.

Step 4: The extract oil yield, Y_{EO} , is determined from values of G, L, M and N which in turn are determined as follows:

Step 4a: G is determined from the measured flash point of the waxy charge by Eq. 7A;

Step 4b: This value of G is used to determine the value of L by Eq. 8A;

Step 4c: The value of M is determined from Eq. 9A using the VI_{targ} , i.e. the desired VI of the dewaxed refined oil, and the fixed values of the constants C_1 and C_2 ;

Step 4d: The values of L and M are added to obtain a value for N according to Eq. 10A;

EXAMPLE II.—NMP REFINING OF A PARAFFIN DISTILLATE

Run No.	I	II	III
(S) NMP dosage, vol. percent	300	202	407
(T_{EO}) ext. out temp., $^\circ\text{F}$	160	140	140
(Y_{EO}) ref'd oil yield, vol. percent	42.0	61.7	43.5
C_e	0.166	0.165	0.125
$C_e S^{0.4}$	1.63	1.39	1.41
$T_R, ^\circ\text{F}$	140	160	160
$A_{NMP}=C_e T_R S^{0.4}$	228	222	226

EXAMPLE III.—NMP REFINING ON A WAXY DISTILLATE

Solvent	NMP	NMP	NMP
(S, F) percent dosage	216	296	295
(T_{EO}) extract out temp., $^\circ\text{F}$	181	180	160
(Y_{EO}) ref'd oil yield, vol. percent	53.1	44.9	54.4
C_e	0.185	1.160	0.137
$T_R, ^\circ\text{F}$	119	120	140
$S^{0.4}$	8.58	9.77	9.75
A_{NMP}	189	188	187

While specific practices of the invention have been shown and described in detail to illustrate the application of the inventive principles, it will be understood that the invention may be practiced otherwise without departing from such principles.

We claim:

1. A method of controlling a furfural refining process wherein a lubricating oil stock is contacted with furfural which comprises generating a first signal which is a measure of the viscosity of said charge oil at a first temperature, generating a second signal which is a measure of the flash point of said charge oil at a second temperature, generating a third signal which is a measure of the refractive index of said charge oil, generating a fourth signal which is a measure of the gravity of said charge oil, combining said first, second, third and fourth signals producing a fifth signal and a sixth signal, and controlling the furfural dosage in response to said fifth signal and the extract-mix outlet temperature in response to said sixth signal.

2. In a process wherein a lubricating oil stock is solvent refined by contact with furfural at a furfural dosage and at an extract-mix outlet temperature effecting extraction of substances of low hydrogen to carbon ratio which adversely affect viscosity index and the solvent refined oil is thereafter cooled in the presence of a dewaxing solvent effecting separation of wax producing a furfural

refined, dewaxed oil, the method of controlling the furfural dosage and extract-mix outlet temperature in said solvent refining such that the viscosity index of said solvent refined, dewaxed lubricating oil stock is maintained constant with variation in said lubricating oil charge stock which comprises generating a first signal which is a measure of the API gravity of said lubricating oil charge stock, generating a second signal which is a measure of the refractive index of said lubricating oil charge stock, generating a third signal which is a measure of the viscosity of said lubricating oil charge stock, generating a fourth signal which is a measure of the flash point of said lubricating oil charge stock, combining said first, second, third and fourth signals thereby generating a fifth signal determinative of the furfural dosage and a sixth signal determinative of the extract-mix outlet temperature related to maintain the viscosity index of said solvent refined dewaxed oil at said pre-determined value, and controlling said furfural dosage and said extract-mix outlet temperature in response to said fifth and sixth signals respectively.

3. In the process of refining waxy crude oil to obtain lubricating oil wherein the waxy crude oil is treated with furfural to effect separation of the waxy crude oil and furfural into a raffinate stream and an extract-mix stream, wherein furfural is recovered from the raffinate stream to yield refined furfural-free waxy oil, and wherein the refined furfural-free waxy oil is dewaxed to yield the lubricating oil, a method for obtaining said lubricating oil at a pre-selected viscosity index, said method comprising: generating a first signal representing the API gravity of the waxy crude oil prior to its furfural treatment; generating a second signal representing the viscosity of the waxy crude oil prior to its furfural treatment; generating a third signal representing the refractive index of the waxy crude oil prior to its furfural treatment; generating a fourth signal representing the flash point of the waxy crude oil prior to its furfural treatment; generating a fifth signal representing the selected viscosity index which the dewaxed lubricating oil is to have; processing said first and second signals to obtain a sixth signal representing the Watson Characterization Factor in accordance with the equation

$$K_v = 9.2767 + 0.0765 \text{ } ^\circ \text{API} + 0.37733 \ln (\text{cs.}) - 0.0254 (\ln \text{cs.})^2 + (0.0021 \text{ } ^\circ \text{API}) (\ln \text{cs.})$$

wherein K_v is the Watson Characterization Factor, $^\circ \text{API}$ is the gravity in $^\circ \text{API}$ represented by the first signal and cs. is the kinematic viscosity in centistokes represented by the second signal; processing said sixth signal to obtain a seventh signal representing a constant of characteristic value for the waxy crude oil in accordance with the equation

$$A = 6099.74 - 487.58 K_v$$

wherein A is the constant of characteristic value of the waxy crude oil; processing said seventh signal to obtain an eighth signal representing the concentration of extract oil in the extract-mix stream in accordance with the equation

$$C_e = 0.0004 A$$

wherein C_e is the concentration of extract oil in the extract-mix stream; processing said third and fourth signals to obtain a ninth signal in accordance with the equation

$$G = RI_{wc} - 0.0001 (\text{fl})$$

wherein G represents the ninth signal, RI_{wc} is the refractive index of the waxy crude oil represented by the third signal and fl is the Cleveland Open cup flash in $^\circ \text{F.}$ represented by the fourth signal; processing said ninth signal to obtain a tenth signal in accordance with the equation

$$L = 0.041 - 1.11 (G - 1.4200)$$

wherein L represents the tenth signal; processing said fifth signal to obtain an eleventh signal in accordance with the equation

$$M = C_1 + C_2 (100 - VI_{\text{tag}})$$

wherein M represents the eleventh signal, VI_{tag} is the aforesaid selected viscosity index represented by said fifth signal, wherein $C_1 = .805$ for distillate waxy crude oil stocks having a flash point less than 450°F. , $.794$ for distillate charge stocks with a flash point equal to or greater than 450°F. , $.801$ for residual stocks, and $C_2 = 0.001155$ for distillate waxy crude oil stocks having a flash point less than 450°F. , $.001445$ for distillates having flash points equal to or greater than 450°F. , and $.001190$ for residual stocks; adding said tenth and eleventh signals together to obtain a twelfth signal; processing said twelfth signal to obtain a thirteenth signal representing the percentage yield of extract oil in the extract-mix stream in accordance with the equation

$$Y_{EO} = 1154 (0.857 - N)$$

wherein Y_{EO} is the percentage yield of extract oil in the extract mix stream represented by the thirteenth signal and N represents the twelfth signal; processing said eighth and thirteenth signals to obtain a fourteenth signal representing the furfural dosage in volumes of furfural used in treating each volume of waxy crude oil in accordance with the equation

$$F = Y_{EO} \left(\frac{1}{C_e} - 1 \right)$$

wherein F is the furfural dosage represented by the fourteenth signal; processing said seventh, eighth and fourteenth signals to obtain a fifteenth signal representing the temperature of the extract-mix stream in accordance with the equation

$$T_{EO} = 323 - \frac{A}{C_e F} 0.5$$

wherein T_{EO} is the temperature in $^\circ \text{F.}$ of the extract-mix stream represented by the fifteenth signal; utilizing said fourteenth signal to control the furfural dosage of the waxy crude oil; and utilizing said fifteenth signal to control the temperature of the extract-mix stream.

4. In the process of refining waxy crude oil to obtain lubricating oil wherein the waxy crude oil is treated with solvent to effect separation of the waxy crude oil and solvent into a raffinate stream and an extract-mix stream, wherein solvent is recovered from the raffinate stream to yield refined solvent-free waxy oil, and wherein the refined solvent-free waxy oil is dewaxed to yield the lubricating oil, a method for obtaining said lubricating oil at a pre-selected viscosity index, said method comprising: generating a first signal representing the API gravity of the waxy crude oil prior to its solvent treatment; generating a second signal representing the viscosity of the waxy crude oil prior to its solvent treatment; generating a third signal representing the refractive index of the waxy crude oil prior to its solvent treatment; generating a fourth signal representing the flash point of the waxy crude oil prior to its solvent treatment; generating a fifth signal representing the selected viscosity index which the dewaxed lubricating oil is to have; processing said first and second signals to obtain a sixth signal representing the Watson Characterization Factor of the waxy crude oil; processing said sixth signal to obtain a seventh signal representing the concentration of extract oil in the extract-mix stream; processing said third, fourth and fifth signals to obtain an eighth signal representing the percentage yield of extract oil in the extract-mix stream; processing said seventh and eighth

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signals to obtain a ninth signal representing the solvent dosage in volumes of solvent used in treating each volume of waxy crude oil; processing said seventh and ninth signals to obtain a tenth signal representing the temperature of the extract-mix stream; utilizing said ninth signal to control the solvent dosage of the waxy crude oil; and utilizing said tenth signal to control the temperature of the extract-mix stream.

5. A method of controlling a solvent refining process wherein a lubricating oil stock is contacted with the solvent which comprises generating a first signal which is a measure of the viscosity of charge oil at a first temperature, generating a second signal which is a measure of the flash point of said charge oil at a second temperature, generating a third signal which is a measure of the refractive index of said charge oil, generating a fourth signal which is a measure of the gravity of said charge oil, combining said first, second, third and fourth signals thereby producing a fifth signal and a sixth signal, and controlling the solvent dosage in response to said fifth signal and the extract-mix outlet temperature in response to said sixth signal.

6. The method according to claim 5 wherein said solvent is NMP.

7. In a process wherein a lubricating oil stock is solvent refined by contact with a solvent at a solvent dosage and at an extract-mix outlet temperature effecting extraction of substances of low hydrogen-to-carbon ratio which adversely affect viscosity index and the solvent refined oil is thereafter cooled in the presence of a dewaxing solvent effecting separation of wax producing a solvent refined, dewaxed oil, the method of controlling the solvent dosage and extract-mix outlet temperature in said solvent refining such that the viscosity index of said solvent refined, dewaxed lubricating oil stock is maintained constant with variation in said lubricating oil charge stock which comprises generating a first signal which is a measure of the API gravity of said lubricating oil charge stock, generating a second signal which is a measure of the refractive index of said lubricating oil charge stock, generating a third signal which is a measure of the viscosity of said lubricating oil charge stock, generating a fourth signal which is a measure of the flash point of said lubricating oil charge stock, combining said first, second, third and fourth signals thereby generating a fifth signal determinative of the solvent dosage and a sixth signal determinative of the extract-mix outlet temperature related to maintain the viscosity index of said solvent refined dewaxed oil at said pre-determined value, and controlling said solvent dosage and said extract-mix outlet temperature in response to said fifth and sixth signals respectively.

8. The process according to claim 7 wherein said solvent NMP.

9. In the process of refining waxy oil to obtain lubricating oil wherein the waxy oil is treated with NMP to effect separation of the waxy oil and NMP into a raffinate stream and an extract-mix stream, wherein NMP is recovered from the raffinate stream to yield refined NMP-free waxy oil, and wherein the refined NMP-free waxy oil is dewaxed to yield the lubricating oil, a method for obtaining said lubricating oil at a pre-selected viscosity index, said method comprising: generating a first signal representing the API gravity of the waxy oil prior to its NMP treatment; generating a second signal representing the viscosity of the waxy oil prior to its NMP treatment; generating a third signal representing the refractive index of the waxy oil prior to its NMP treatment; generating a fourth signal representing the flash point of the waxy oil prior to its NMP treatment; generating a fifth signal representing the selected viscosity index which the dewaxed lubricating oil is to have; processing said first and second signals to obtain a sixth signal representing the Watson Characterization Factor in accordance with the equation

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$$K_v = 9.2767 + 0.0765 \text{ } ^\circ\text{API} + 0.37733 \ln (\text{cs.})$$

$$-0.0254 (\ln \text{cs.})^2 + (0.0021 \text{ } ^\circ\text{API}) (\ln \text{cs.})$$

wherein K_v is the Watson Characterization Factor, $^\circ\text{API}$ is the gravity in $^\circ\text{API}$ represented by the first signal and cs. is the kinematic viscosity in centistokes represented by the second signal; processing said sixth signal to obtain a seventh signal representing a constant of characteristic value for the waxy crude oil in accordance with the equation

$$A_{NMP} = 0.9(6099.74 - 487.58 K_v)$$

wherein A_{NMP} is the constant of characteristic value of the waxy oil; processing said seventh signal to obtain an eighth signal representing the concentration of extract oil in the extract-mix stream in accordance with the equation

$$C_e = \frac{0.0004}{0.9} A_{NMP}$$

wherein C_e is the concentration of extract oil in the extract-mix stream; processing said third and fourth signals to obtain a ninth signal in accordance with the equation

$$G = RI_{wc} - 0.0001 (\text{fl})$$

wherein G represents the ninth signal, RI_{wc} is the refractive index of the waxy oil represented by the third signal and fl is the Cleveland Open Cup flash in $^\circ\text{F.}$ represented by the fourth signal; processing said ninth signal to obtain a tenth signal in accordance with the equation

$$L = 0.041 - 1.11 (G - 1.4200)$$

wherein L represents the tenth signal; processing said fifth signal to obtain an eleventh signal in accordance with the equation

$$M = C_1 + C_2(100 - VI_{\text{targ}})$$

wherein M represents the eleventh signal, VI_{targ} is the aforesaid selected viscosity index represented by said fifth signal, wherein $C_1 = .805$ for distillate waxy oil stocks having a flash point less than 450°F. , .794 for distillate charge stocks with a flash point equal to or greater than 450°F. , .801 for residual stocks, and $C_2 = 0.001155$ for distillate waxy oil stocks having a flash point less than 450°F. , .001445 for distillates having flash points equal to or greater than 450°F. , and .001190 for residual stocks; adding said tenth and eleventh signals together to obtain a twelfth signal; processing said twelfth signal to obtain a thirteenth signal representing the percentage yield of extract oil in the extract-mix stream in accordance with the equation

$$Y_{EO} = 1154(0.857 - N)$$

wherein Y_{EO} is the percentage yield of extract oil in the extract-mix stream represented by the thirteenth signal and N represents the twelfth signal; processing said eighth and thirteenth signals to obtain a fourteenth signal representing the NMP dosage in volumes of NMP used in treating each volume of waxy oil in accordance with the equation

$$S = Y_{EO} \left(\frac{1}{C_e} - 1 \right)$$

wherein S is the NMP dosage represented by the fourteenth signal; processing said seventh, eighth and fourteenth signals to obtain a fifteenth signal representing the temperature of the extract-mix stream in accordance with the equation

$$T_{EO} = 300 - \frac{A_{NMP}}{C_e S^{0.4}}$$

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wherein T_{EO} is the temperature in ° F. of the extract-mix stream represented by the fifteenth signal; utilizing said fourteenth signal to control the NMP dosage of the waxy oil; and utilizing said fifteenth signal to control the temperature of the extract-mix stream.

10. The process according to claim 4 wherein the solvent is furfural.

11. The process according to claim 4 wherein the solvent is NMP.

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HERBERT LEVINE, Primary Examiner

U.S. Cl. X.R.

196—14.5, 14.52; 208—311, 326, 327

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,458,432

July 29, 1969

Robert A. Woodle et al.

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 6, line 71, "244" should read -- 249 --; line 72, "Eq. 4, C_e is found to be 0.0976." should read -- Eq. 5, C_e is found to be 0.0995. --. Columns 9 and 10, the table identified as Example I, first column, line 6 thereof, "Tr, °F." should read -- T_R , °F. --. Column 10, the table identified as Example II, first column, line 7 thereof, " A_{NMR} " should read -- A_{NMP} --. Column 12, lines 40 to 42, the equation should appear as shown below:

$$T_{EO} = 323 - \frac{A}{C_e F^{0.5}}$$

Signed and sealed this 11th day of August 1970.

(SEAL)
Attest:

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