951,272.

H. FRASCH.

OBTAINING PETROLEUM PRODUCTS.

APPLICATION FILED OCT. 30, 1903.

Patented Mar. 8, 1910.

5 SHEETS—SHEET 1.

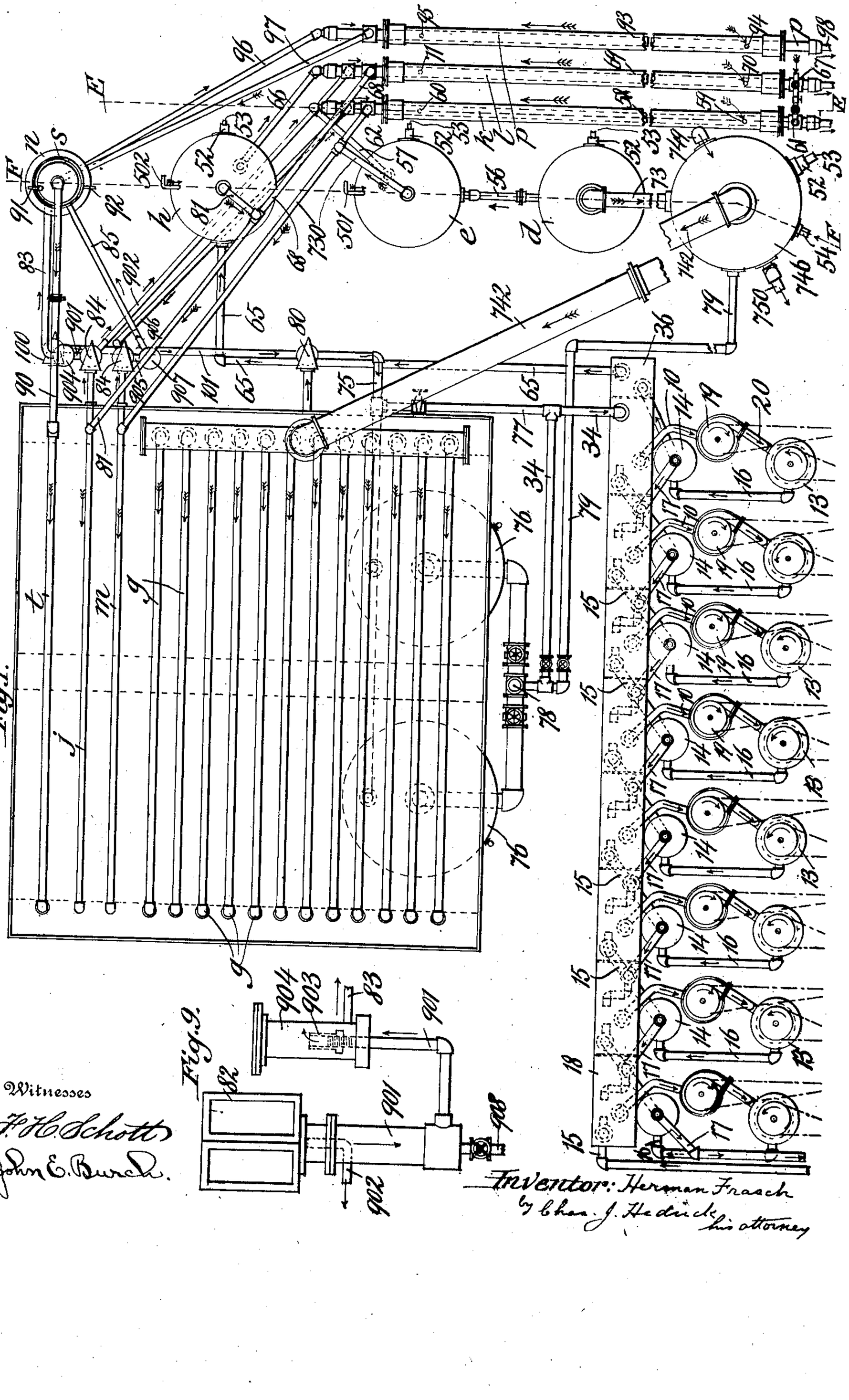

Fig. 1.

Fig. 9.

Witnesses

F. H. Schott

John E. Burch.

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5 SHEETS—SHEET 2.

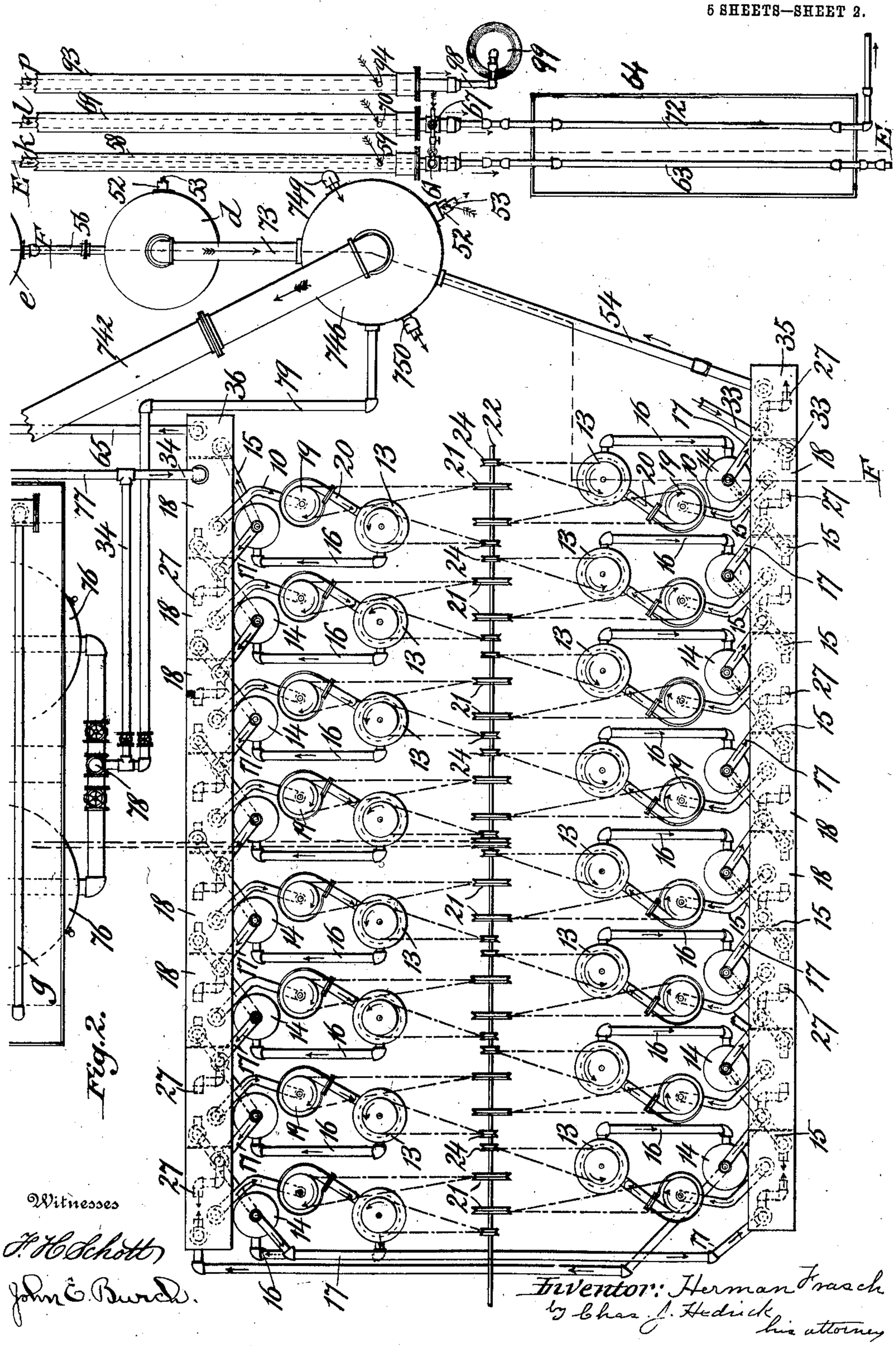

Fig. 2.

Witnesses

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5 SHEETS—SHEET 3.

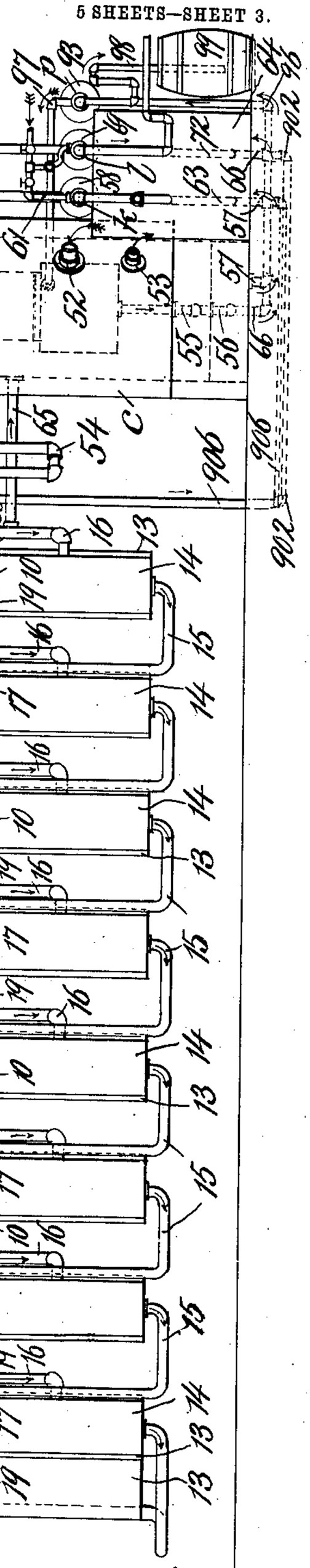

Fig. 6.

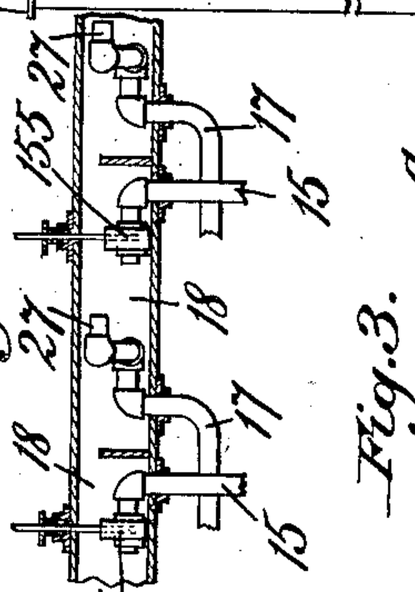

Fig. 3.

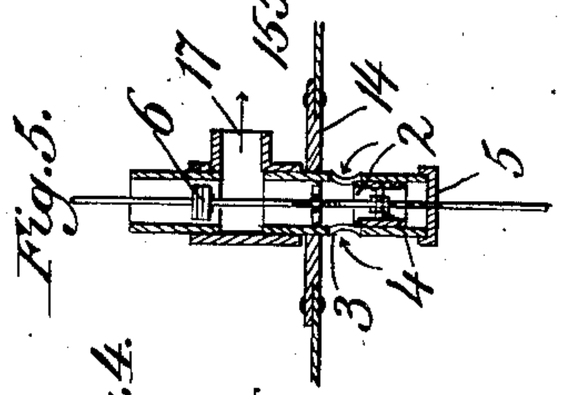

Fig. 5.

Fig. 4.

Witnesses

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5 SHEETS—SHEET 4.

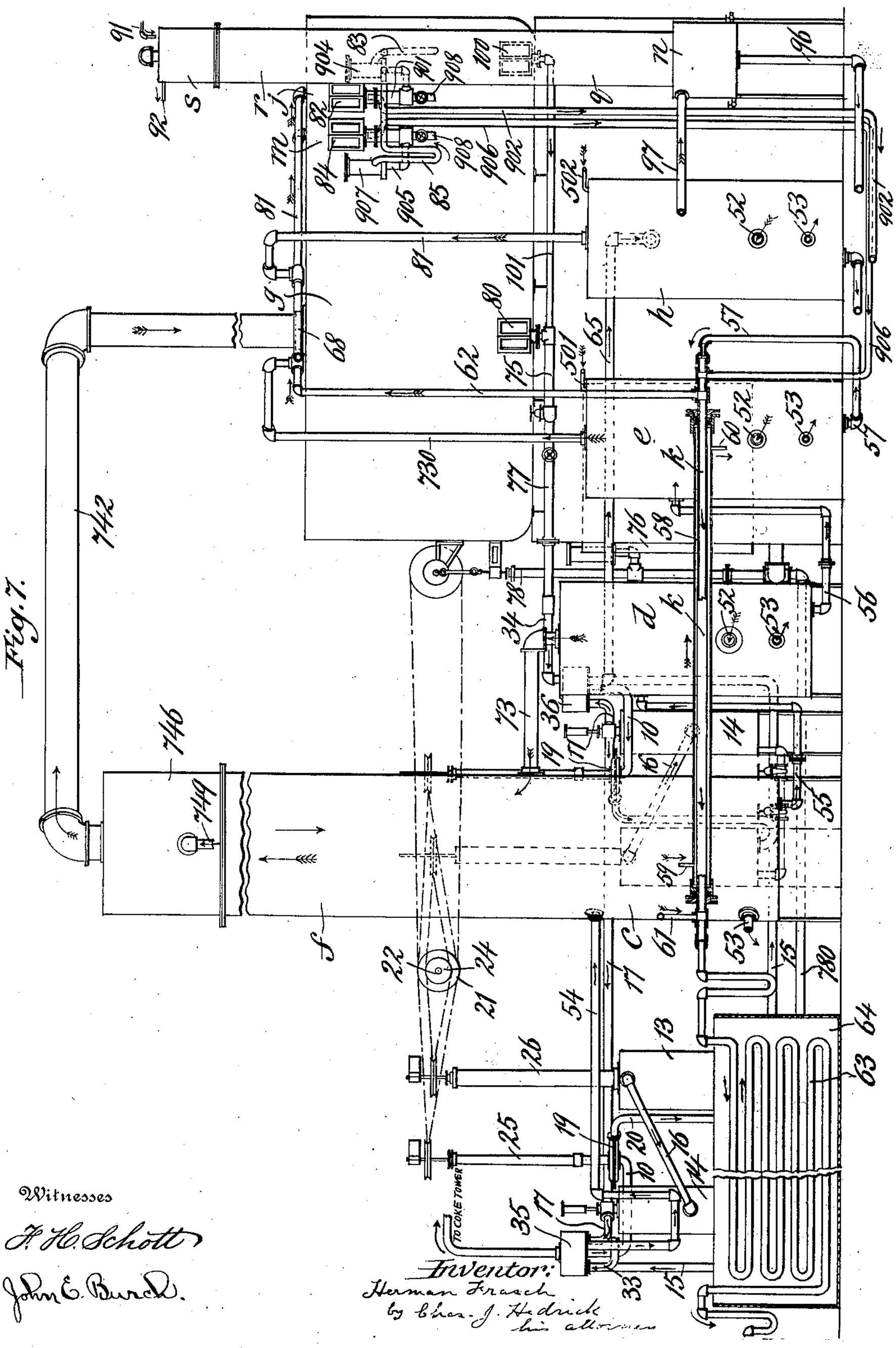

Fig. 7.

Witnesses

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5 SHEETS—SHEET 5.

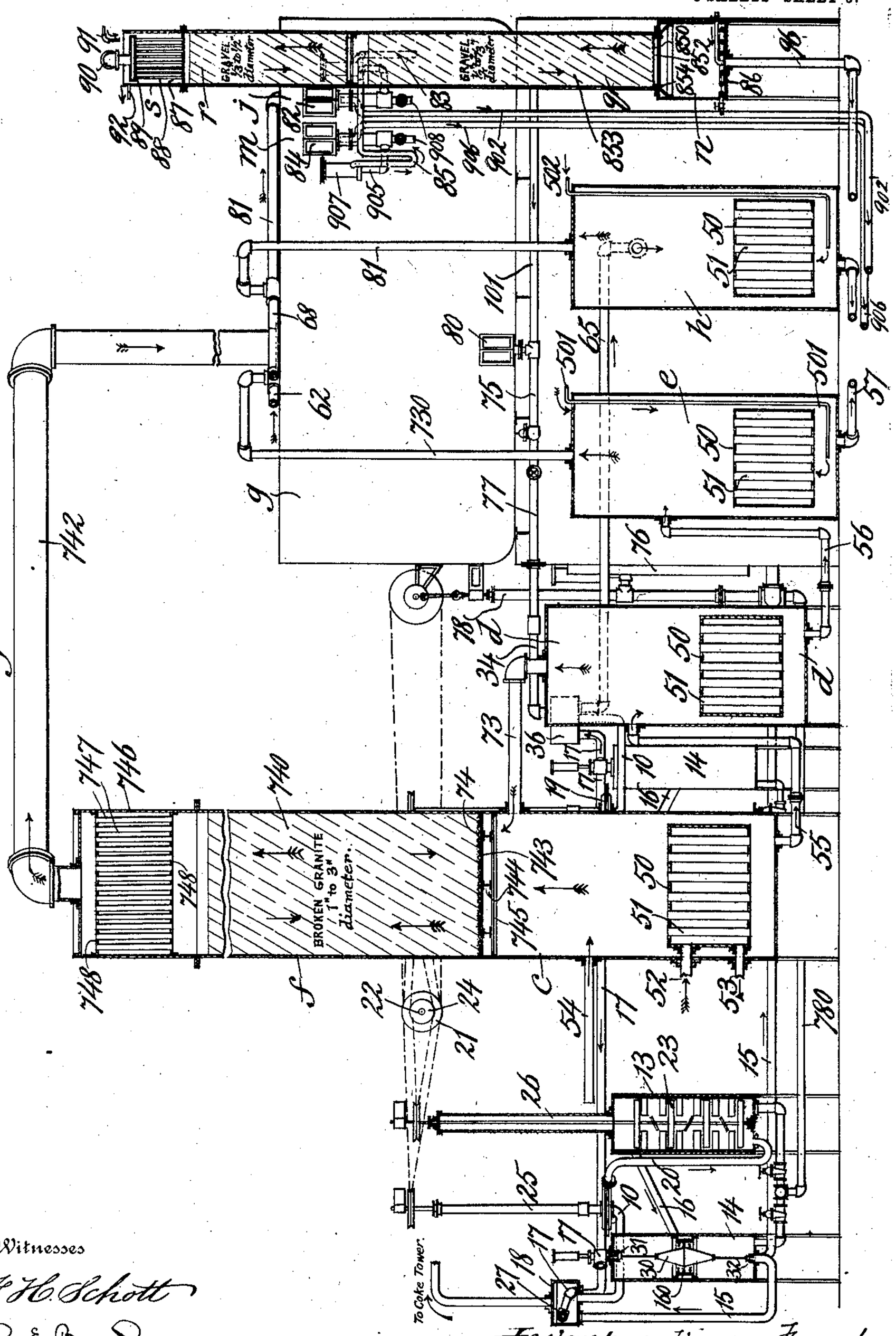

Fig. 8.

Witnesses

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UNITED STATES PATENT OFFICE.

HERMAN FRASCH, OF NEW YORK, N. Y., ASSIGNOR TO STANDARD OIL COMPANY, OF BAYONNE, NEW JERSEY, A CORPORATION OF NEW JERSEY.

OBTAINING PETROLEUM PRODUCTS.

951,272. Specification of Letters Patent. Patented Mar. 8, 1910.

Original application filed October 4, 1902, Serial No. 125,967. Divided and this application filed October 30, 1903. Serial No. 179,235.

To all whom it may concern:

Be it known that I, HERMAN FRASCH, a citizen of the United States, residing at New York city, Manhattan borough, New York county, in the State of New York, have invented new and useful Improvements in Obtaining Petroleum Products, of which the following is a specification.

This invention relates more particularly to obtaining burning oil (kerosene) from petroleum of the nature of that obtained from the wells near Beaumont, in Jefferson county, Texas, hereinafter referred to as Beaumont oil or Beaumont petroleum; but it also relates to obtaining burning oil (kerosene) from other kinds of petroleum as hereinafter set forth; and it is not wholly confined to the obtainment of burning oil (kerosene).

On subjecting Beaumont petroleum to fractional distillation (with or without cracking) and subjecting the entire distillate which comes over between the ordinary temperature limits of burning oil (say, from 250° F. to 600° F.) to treatment with sulfuric acid of 66° Baumé (followed by the customary washing with water, and with caustic soda solution, and again with water), the resulting product will be colorless and of a mobility to climb a lamp wick (although much denser than Pennsylvania burning oil); but it will smoke when burned in ordinary petroleum (kerosene) lamps, especially if the flame should be turned low. The raw distillate shows the same smoky flame. I have discovered that such distillate is composed in part only of smoky hydrocarbons and that these and the non-smoking hydrocarbons which accompany them in distillation can be separated from each other by the differentially solvent action of an appropriate menstruum, such as methyl alcohol or ethyl alcohol, not only to a useful extent, but sufficiently to obtain from such smoky distillate a non-smoking burning oil (kerosene) whose hydrocarbons have the same range of boiling points as those of said smoky distillate. And I believe the same to be true of any petroleum analogous to Beaumont oil in possessing a smoky burning oil fraction, to the extent at least that such a non-smoking burning oil (kerosene) can be obtained from smoky distillate representing a part (if not the whole) of the burning oil fraction of such petroleum. But said Beaumont or analogous petroleum is not the only petroleum the burning oil (kerosene) from which burns with a less clear flame than does burning oil (kerosene) from Pennsylvania petroleum or (when desulfurized) from Lima oil. Russian petroleum, for example, is of that character; and so I believe is any petroleum whose burning oil fraction is denser (heavier in gravity—less in degrees Baumé) than that of Pennsylvania or Lima oil. I have discovered in respect to Russian petroleum (and I believe it will hold true of petroleum of the last mentioned description in general) that the burning oil fraction thereof is composed of hydrocarbons which differ in their tendency to smokiness and that said hydrocarbons can be separated from each other by a differentially solvent menstruum of the aforesaid description, with the result of improving the burning oil (kerosene) from said petroleum in respect to the character of its flame. Moreover, an oil made from that fraction of Pennsylvania and certain other petroleum which is less volatile than burning oil (kerosene) has been used to a certain extent for illuminating purposes by burning in special lamps. Such heavier illuminating oil fraction of Beaumont or other petroleum mentioned can also, I believe, be improved in respect to smokiness or tendency thereto by subjecting the same to the aforesaid menstruum. Further, the illuminating oil fractions (namely, the burning oil fraction and said heavier fraction) of petroleum whose burning oil fraction is of the density of that of the Pennsylvania and Lima petroleum undergo, each of them, a separation of hydrocarbons, when subjected to the aforesaid menstruum, with the result of obtaining oil in which the ratio of carbon to hydrogen (which can be ascertained by elementary chemical analysis) is less than in the oil so treated.

Based on these discoveries, the invention consists in the processes of subjecting part at least of either or both of said illuminating oil fractions of Beaumont or other petroleum, as above mentioned, to the action of a menstruum, such as methyl alcohol or ethyl alcohol, which has a different solvent

action upon the non-smoking from that which it has upon the smoky hydrocarbons of said burning oil fraction of Beaumont petroleum, and thereby obtaining the results stated. The menstruum may be applied to all or to any desired part of either or both of said illuminating oil fractions before or after the separation of the same from other fractions or parts of the petroleum; but the application would best be made after such separation, the oil treated being in the form of an appropriate distillate. The distillation of the crude oil may, in any case, be performed with or without cracking.

In addition to giving an oil of less ratio of carbon to hydrogen than the oil treated, the separation which is effected by the differentially solvent menstruum also results in an oil of greater ratio of carbon to hydrogen; and this more highly carbonaceous product can be utilized for burning as fuel or for other appropriate purpose.

The solvent action of methyl or ethyl alcohol is greater on the smoky hydrocarbons (or those having a higher ratio of carbon to hydrogen) than on the non-smoking hydrocarbons (or those having a lower ratio); but it is evident that a menstruum having the reverse differential action might serve to separate the two kinds of hydrocarbons; and it is believed that a differential solvent in general has now for the first time been applied as indicated.

Nearly anhydrous methyl alcohol is regarded as the best menstruum; but another appropriate menstruum (differential solvent) can be used without exceeding the limits of the invention. As already indicated, ethyl alcohol can be used. I believe other appropriate menstrua to exist.

After the desired and feasible separation of the hydrocarbons from each other has been obtained, the menstruum is separated from the hydrocarbons in solution therein in order to obtain purer oil and also to recover the valuable menstruum; and where it and they have sufficiently different boiling points fractional distillation would best be resorted to; but the menstruum separation might be effected in different ways which might vary with the menstruum. Addition of water followed by settling and decantation will separate methyl alcohol or ethyl alcohol from hydrocarbons (of whatever volatility) in solution therein. Methyl alcohol and ethyl alcohol can each of them be separated by distillation from the hydrocarbons which compose the heavier portion of the burning oil fraction of any kind of petroleum. The separated menstruum can be disposed of in any way; but it would best be used over again on a new lot of oil, with or without being subjected to an intermediate treatment, as may be found necessary or desirable. The invention includes the processes or improvements in which such separation, or such separation and repeated use, of the menstruum is a feature.

In obtaining burning oil from petroleum I have found advantage in the case of Beaumont oil, and I believe advantage would also be found in other cases, in subjecting the heavier portion of the burning oil fraction of the oil to the menstruum after preliminarily separating the light end of said fraction therefrom. In my experience with Beaumont oil I have found such light end sufficiently free from smoky hydrocarbons to be used in burning oil (kerosene) without subjection to the menstruum; but it can, if necessary or desired, be subjected thereto. By the preliminary separation of the light end and omission to subject it to the menstruum a saving of oil is effected; since if said light end were allowed to remain it would be deprived of utilizable constituents by the menstruum; and such a saving would result whenever or however the heavier portion of the burning oil fraction of petroleum (after removal of the light end therefrom) should be treated by agents which would remove utilizable constituents from said light end, if applied thereto. The invention covers this feature generally.

Other or further improvements in obtaining petroleum products are also included in the invention, as herein set forth.

Another part of my present invention consists in certain new petroleum or hydrocarbon oils. Oils within this part of the invention can be obtained from Beaumont petroleum by means of the improvements or processes herein set forth; and such products are more especially, although not exclusively, intended; since the invention includes oils in general having the necessary characteristics (hereinafter pointed out).

In the accompanying drawings, Figures 1 and 2, taken together, but omitting those parts from Fig. 1 which also appear on Fig. 2, make a plan view of an oil washing apparatus or plant for carrying the invention into effect, certain intermediate portions of said apparatus being broken away in Fig. 1 in order to get the figure on the sheet; Fig. 3 is a front elevation of the same apparatus; Fig. 4 is a view in vertical section of one of the settling and decanting vessels forming part of said apparatus; Fig. 5 is a detail view on a larger scale of a form of valve considered more advantageous than that shown in Fig. 4; Fig. 6 is a detail view of another valve arrangement; Fig. 7 is a side elevation, partly in section, of the apparatus or plant of Figs. 1, 2 and 3, looking to the left from the line E—E of Figs. 1 and 2; Fig. 8 is a sectional elevation on line F—F of Figs. 1 and 2 looking to the left, so much of the vapor pipe 730 as lies to the right of the section line being shown in elevation;

and Fig. 9 (Sheet 1) is a view of a device for skimming oil from the aqueous methyl alcohol of condensation which runs from one of the condensers employed.

In Figs. 3, 7 and 8 one of the rectifying columns and its vapor pipe are shown partly broken away, in order to get the figures on their respective sheets.

The apparatus shown contains a series of mixing vessels 13 for churning together the oil and the menstruum. Each such vessel is provided with a rotary stirrer 23 (see left hand end of Fig. 8) operated by a belt from a pulley 24 on shaft 22; and the stuffing box for each stirrer shaft is located on top of a sleeve 26 so as to be raised above the level of liquids in vessel 13. Thus arranged, it is less likely to allow material to escape from the vessel 13 than if it were in position to be wet by said liquids. The liquids enter each vessel 13 at the bottom through a pipe 20 and leave it at the top (see left hand end of Fig. 7) through a pipe 16. For each mixing vessel 13 there is a settling and decanting vessel 14 (Figs. 4, 7 and 8) for separating the mixture of oil and menstruum into a lighter and a heavier component. Each such vessel 14 is provided with an inlet at the middle to which the pipe 16 leads from vessel 13 and with outlets at top and bottom respectively from which pipes 17 and 15 carry off the separated liquids. The pipe 16 opens into an annular space between a perforated distributing plate 160 and the wall of the vessel 14. In the vessel is a submerged float 30 (Figs. 4 and 8) carrying a valve 31 for the upper outlet and a valve 32 for the lower outlet. In normal working both valves are open; and their adjustments depend upon the gravity of the mixture of oil and menstruum in vessel 14; for as such mixture becomes lighter the float rises, closing the valve 31 and opening the valve 32 correspondingly; and as it becomes heavier the float falls, opening the valve 31 and closing the valve 32. An automatic control is thus exercised on the proportions of the entering mixture which leave by pipes 17 and 15 respectively. In Fig. 5 the valve 2 is placed between the outlet ports 3 and the middle of the vessel 14; so that the current does not tend to close the same. The body of the valve has holes 4 therein so that the liquid may pass into and out of the space between the valve 2 and the cap 5. At 6 are weights for loading float 30. In Fig. 6 the end of the pipe 15 is provided with a simple gate valve 155 for controlling the outflow from vessel 14 by pipe 15. It can be used instead of or in addition to either or both of the float operated valves 31, 32. The pipe 17 has a vertically adjustable end 27, the elbows which connect it with the body of the pipe allowing it to be set at different elevations for regulating the flow of liquid through the pipe 17. For each mixing vessel 13 there is also a centrifugal pump 19 whose shaft passes through a stuffing box at top of sleeve 25 (used for the same purpose as sleeve 26) and is driven by a belt from a pulley 21 on shaft 22. The mixture of oil and menstruum enters the pump by a pipe 10 and is fed by it to the corresponding vessel 13 through pipe 20. Further, for each vessel 13 there is a supply chamber 18 for supplying by pipe 10 (Fig. 8) to the corresponding pump 19 the mixture of liquids which it receives (Fig. 6) from pipes 15 and 17; only the supply chamber at the lower right hand end of Fig. 2 receives liquids from pipes 33 and 17; while that at the upper right hand end of the same figure receives liquids from pipes 34 and 15. The series of supply chambers 18 is formed by partitions (Fig. 6) in the troughs shown at top and bottom respectively of Fig. 2. These troughs as shown are closed (Fig. 6) on top to prevent evaporation of the menstruum therein; and the stems of valves 155 pass through stuffing boxes in the cover. The chamber 35 receives liquid by a pipe 17 and discharges it by a pipe 54. The chamber 36 receives liquid by a pipe 15 and discharges it by a pipe 65. A pane of glass 9 in the wall of each chamber allows observation of the interior.

Assuming that a mixture of oil and differentially solvent menstruum in the proportions in which they are to be used should yield (on settling) two liquids; of which the bulk of the menstruum should be contained in the lighter liquid, the oil would flow through pipe 33 into what may, therefore, be regarded as the first supply chamber of the series; while the menstruum would flow through pipe 34 into the last supply chamber of the series. The oil in the first supply chamber meets the liquid flowing from the top of the second settling and decanting vessel of the series by pipe 17 and consisting of menstruum charged with hydrocarbons. The two liquids are then supplied by aid of the first pump 19 to the first mixing vessel 13 of the series; and from it they pass by pipe 16 to the first settling and decanting vessel 14; where a separation of liquids takes place, the once washed oil passing by pipe 15 to the second supply chamber 18. Here it meets the charged menstruum from the third decanting and settling vessel; and the two liquids are supplied therefrom by the second pump 19 to the second mixing vessel 13; from which they pass by pipe 16 to the second settling and decanting vessel 14. The twice washed oil then flows by pipe 15 to the third supply chamber 18. In turn the oil passes through each set of washing appliances (composed of a supply chamber 18, a pump 19, a mixing vessel 13 and a settling and decanting vessel 14); and it is washed

in each with menstruum. In the last set of said appliances it is washed with the fresh menstruum from pipe 34, and passes off by way of chamber 36 and pipe 65. The menstruum which flows through pipe 34 into the last of the supply chambers 18 meets therein the already often washed oil from the last but one of the settling and decanting vessels 14 and is supplied with it by the last pump 19 to the last of the mixing vessels 13; from which the mixture passes into the last of the settling and decanting vessels. From this the menstruum with its dissolved hydrocarbons passes by pipe 17 to the next to last supply chamber 18, where it meets oil from the last but two of the settling and decanting vessels 14. The oil and menstruum are then fed by the next to last pump 19 to the next to last mixing vessel 13; from which they pass into the next to last settling and decanting vessel 14. From this the menstruum with its dissolved hydrocarbon passes by pipe 17 to last but three of the supply chambers 18. Thus the menstruum passes through each set of washing appliances in inverse order to that in which the oil passes through them. From chamber 35 it passes away by the pipe 54.

Assuming that methyl alcohol is used as the differentially solvent menstruum, the means shown for recovering it from the hydrocarbons carried away by it through pipe 54 consist of a series of four stills *c d e k* (Figs. 1, 7 and 8), two of them (*c d*) heated by dry heat only and provided with rectifying column *f*, dephlegmator 746 and condenser *g* common to the two stills, and the other two of them (*e k*) having means for subjecting the liquid therein to free steam and provided with a condenser *m* common to both. In each still *c d e* there is a tight steam drum 50 (Fig. 8) with tubes 51 open at both ends and extending through the drum for circulation of liquid. The steam inlet for each of these drums is marked 52; and the outlet for each for the water of condensation is marked 53 (Figs. 7 and 8). The pipe 54 leads to still *c;* and the residual liquid passes from it by pipe 55 into still *d*. The vapors from the latter enter still *c* by pipe 73 and pass with the vapors evolved in still *c* through the column *f* and dephlegmator 746 and pipe 742 (Fig. 1) to the condenser *g*. The column *f* has a perforated floor 74, it may be of any desired form, and is provided with a filling 740 of (most advantageously) broken granite of from an inch to three inches in diameter. As shown (Fig. 8) a disk 74 of wire cloth rests upon a perforated plate 743 which in turn rests upon I beams 744 supported at the ends by the ring 745 fastened to the walls of the column. The dephlegmator consists of upright vapor tubes 747 set between heads 748 and surrounded by water introduced by the pipe 749 (Fig. 3) and escaping by pipe 750. The water supply may be so regulated as to have a temperature of about 120° F., more or less, at the outlet 750. The condensate formed in the dephlegmator and in the column *f* returns to the still *c* and in its return is brought thoroughly into contact with the rising vapors. The condensate from condenser *g* passes by pipe 75 to menstruum storage tanks 76 or (proper cocks being closed) by the pipe 75, by-pass 77 and pipe 34 to the last supply chamber 18 of the series, as before mentioned. From the storage tanks 76 the menstruum can be delivered by the pump 78 through pipe 34 to said supply chamber. The stuffing box for the piston rod of the pump 78 is elevated, as described for those around the stirrer shafts of the mixing vessels 13. The same pump 78 will serve (on occasion) for exhausting the contents of vessels 13 and 14 through pipe 780 (Fig. 8) and for delivering the same through pipe 79 (Figs. 2 and 3) into still *e* (the valve in pipe 34 being closed and that in pipe 79 being opened for the purpose). Between the condenser *g* and the pipe 75 is a box 80 with glass sides and movable slide so that the flow of condensate can be observed and a test sample taken, if desired. The residual liquid from still *d* passes by pipe 56 into still *e* and into it free steam is introduced in small quantity by pipe 501. The vapors (including the steam) pass by pipe 730 to the condenser *m* along with the vapors from still *k*. This still receives the residual liquid by pipe 57 (Figs. 1 and 7) from still *e* and as shown consists of a long tube *k* inclosed for the greater part of its length by a jacketing tube 58 which is closed at the ends and provided with a steam inlet 59 at one end on top and an exhaust 60 at the opposite end underneath. The tube *k* is provided at its oil outlet with a free steam inlet pipe 61 and at its oil inlet end with a vapor escape pipe 62. The vapors (including the steam) pass by said pipe 62 to the pipe 730 and so to the condenser *m*. The residual liquid (oil) from which the menstruum (methyl alcohol) has been recovered passes to and through a cooling pipe 63 immersed in water in the box 64 and thence to a storage tank, not shown. It consists of the hydrocarbons which were washed out of the oil in the vessels 13, 14 by the methyl alcohol.

For recovering the menstruum (methyl alcohol) which accompanies the washed oil, a series of two stills *h l* with a condenser *j* common to both stills *h l* is shown. The pipe 65 delivers the washed oil into the still *h* (Fig. 8), which is of the same construction as still *e;* and the vapors generated therein, with free steam from pipe 502, pass over by pipe 81 to the condenser *j*. The

residual oil flows by pipe 66 to still *l*, which has a steam jacket 69 corresponding with the jacket 59 of still *k* (as shown for latter in Fig. 8) and having on top a steam inlet 70 (Fig. 1) and outlet 71 underneath for the water of condensation. The still *l* also has a pipe 67 (Figs. 1 and 3) corresponding with the pipe 61 (Figs. 1, 3 and 8) for the still *k* for introducing free steam into the still *l*. The vapors generated in still *l*, with the so introduced steam, pass over by pipes 68 and 81 to the condenser *j*. The washed oil, freed from methyl alcohol, passes through a cooling coil 72 (Fig. 2) in water tank 64 to a storage tank, not shown. The condensate from condenser *j* consists of methyl alcohol diluted with water and accompanied by some oil. It flows into box 82 (Figs. 1, 7 and 9) (of known form with glass sides and a slide to allow a sample of condensate to be taken) and then into the inverted siphon 901. The oil rises and overflows by pipe 902, which conducts it to still *l;* while the dilute methyl alcohol flows over the top of the adjustable pipe section 903 in chamber 904 and is introduced by pipe 83 into the column *q r s*. The condensate from condenser *m* also consists of methyl alcohol diluted with water and accompanied by some oil. It flows into box 84 (Figs. 1 and 7) and then into the inverted siphon 905 (Fig. 7) (similar to that marked 901 in Fig. 9); the oil rises and is conveyed by pipe 906 (Figs. 1, 3, 7 and 8) to tubular still *k;* while in chamber 907 the dilute methyl alcohol flows over the top of an adjustable pipe section (similar to that marked 903 in Fig. 9) and is thence conveyed by pipe 85 to the column *q r s*. Each of the inverted siphons is provided with a valve pipe 908 for emptying it and the corresponding condenser when desired. The lower section *q* of column *q r s* (Fig. 8) has at its bottom a perforated floor in the form of a disk of wire cloth resting on a grating 852, upheld by annular ledge 854 and is provided with a filling of gravel 853 which will pass through a sieve of wire cloth of three meshes to two linear inches and not through one of two meshes to the linear inch. The middle section *r* has at its bottom a perforated floor, on which rests a filling of gravel which is best a little finer than that in the column *q;* say, gravel which will pass through a sieve of two meshes to the linear inch and not through one of three inches to the linear inch. The upper section *s* consists of a number of vertical pipes 88 open at both ends and inclosed in a water box 87. The bottom ends of the pipes are set in the sheet which closes the top of section *r;* and their upper ends are joined to a manifold 89; from which the vapor pipe 90 leads to condenser *t* (Fig. 1). The water box 87 has a water inlet 91 and overflow 92 which may be so regulated that the water in the box has a temperature of about 120° F., more or less, at the overflow. The column *q r s* surmounts the still *n*, which is a plain box provided with a close steam coil 86. The dilute methyl alcohol is fed into the top of section *q;* which acts as a distilling column as well as a rectifier of the vapors formed in it and in the still *n*. These vapors are further rectified in sections *r* and *s*, being deprived of nearly all the then remaining water in the section *s*. They pass over into condenser *t;* and the resulting condensate passes by way of box 100 and pipes 101 and 75 (Figs. 7 and 1) into the menstruum storage tanks 76, or by way of box 100 and pipes 101, 75, 77 and 34 into the oil washing apparatus. The poor liquid flows from still *n* by pipe 96 to the tubular still *p* (having a close steam jacket 92 with steam inlet at 94 on top and an outlet 95 for water of condensation underneath); and in it the last traces of methyl alcohol are expelled. The residual liquid flows by pipe 98 to a barrel 99 which may have a sewer connection. The vapors pass by pipe 97 to the still *n* and ascend the column *q r s* with whatever vapors may be generated in still *n*.

To prevent loss of methyl alcohol by escape of the vapors thereof from the different portions of the apparatus, ventilating pipes can be provided, as shown at left of Figs. 7 and 8, which pipes would lead to absorbing apparatus, such as a coke tower wherein water would flow over coke in the well known manner.

Assuming that a non-smoking burning oil (kerosene) is to be obtained from the smoky burning oil fraction of Beaumont petroleum by the aid of methyl alcohol as the differentially solvent menstruum, the procedure would best be as follows. The crude Beaumont oil is distilled with cracking (in apparatus not shown), cracked oil equal to about 82 per cent. by volume of the charge of crude oil being collected as distillate, namely, about 30 per cent. before cracking commences and 52 per cent. afterward. This cracked oil is then distilled in a fire heated still in the ordinary way, except that the still should have a rectifying column (say, one similar to column *f* without the water cooled dephlegmator 746). When a temperature of about 350° F., more or less, is indicated at the top of the column, the previously collected distillate (amounting, say, to about 7 per cent. more or less, by volume of the charge of crude oil) represents the light end of the burning oil fraction, with whatever naphtha may be yielded by the crude oil. The delivery of distillate from the condenser is then changed to another receiver; and the distillation is continued preferably until all the burning oil fraction is off. The distillate collected after the change of receiver

(amounting, say, to about 48 per cent., more or less, by volume of the charge of crude oil) represents the heavier portion of the burning oil fraction and constitutes the oil to be subjected to the hydrocarbon separating menstruum.

The methyl alcohol to be used as the menstruum should be nearly anhydrous and of a density equal to or less than 44° B., and may have a boiling point between 146° F. and 150° F. Practically crude wood alcohol, or the 97 per cent. methyl alcohol of commerce, can be used effectively when dehydrated by distilling and rectifying the same in column *q r s* (Figs. 7 and 8) herein above described. What is considered the best ratio of oil to methyl alcohol in washing is one volume of the former to six of the latter. The general principle recommended to be followed is to use as little methyl alcohol as will yield a satisfactory non-smoking oil. The oil and the methyl alcohol are introduced in these proportions by the pipes 33 and 34 respectively; and they travel in opposite directions through the oil washing apparatus, the oil and the methyl alcohol being mixed in each vessel 13 and allowed to separate (with the constituents extracted by each from the other) in each settling and decanting vessel 14. The washed oil which runs from the chamber 36 by pipe 65 is freed from the portion of the menstruum (methyl alcohol) which accompanies it by heating and subjection to free steam in the stills *k l*. On leaving still *l* it is cooled in pipe 72 (Fig. 2). The oil obtained (amounting to about 24⅔ per cent., more or less, by volume of the crude oil) and the preliminarily separated light end (amounting to 7 per cent. of the crude oil) are mixed together, giving 31⅔ per cent. of non-smoking oil of the burning oil fraction. The so obtained oil is finished for sale or use as burning oil (kerosene) by treatment with sulfuric acid of 66° B. and washing with water and with solution of caustic soda and again with water; or, in other words, it is finished by the well known sulfuric acid treatment. If not of proper fire test it is brought thereto in the known way. This burning oil (as also the oil before finishing by the sulfuric acid treatment) has had a refractive index below 1.4600 (say, 1.4545); it has had the proper mobility for combustion in ordinary petroleum (kerosene) lamps; it has distilled almost wholly between 250° F. and 600° F. (98$\frac{8}{10}$ per cent. by volume coming over between 275° F. and 600° F.); the portions distilling over in 25° F. between 300° F. and 550° F. have ranged from about 52° B. for the lightest to about 31° B. for the heaviest of these ten portions; the portions distilling over in 25° F. intervals between 300° F. and 400° F. have contained on an average a larger percentage of hydrocarbons which are readily attacked by fuming sulfuric acid than have the portions distilling over in such intervals between 400° F. and 500° F. or 550° F. (say, between five and seven per cent. more of them); the portions distilling over in 25° F. intervals between 400° F. and 500° F. or 550° have exhibited an average bromin absorbing capacity of more than six per cent. (say, between twelve and thirteen per cent.) and the oil has necessarily possessed the characteristics of an oil obtained out of burning oil distillate from petroleum by subjecting such distillate to a differentially solvent menstruum; in contradistinction to oil of like refractive index, mobility, boiling point and gravity obtained out of burning oil distillate from petroleum by subjecting such distillate merely to sulfuric acid of 66° B. or to fuming sulfuric acid (ten per cent. anhydrid). The average gravity of the four portions distilling over in 25° F. between 400° F. and 500° F. has been about 36½° B. and their average refractive index below 1.4590 (say, 1.4571).

In addition to the washed oil which runs from the oil washing apparatus by the pipe 65, another oil accompanies the hydrocarbon separating menstruum which runs from chamber 35 by pipe 54, the same having been washed by the menstruum out of the oil supplied by pipe 33, as before mentioned. The menstruum (methyl alcohol) is distilled off from this oil mainly in stills *c d* by dry heat and as to the remaining part in stills *e k* by heat and subjection to free steam. The oil leaving still *k* is cooled in pipe 63 (Figs. 2 and 7). It has amounted to about 23⅓ per cent., more or less, of the crude oil and has had a ratio of carbon to hydrogen of more than seven parts by weight of the former to one of the latter (say, 87½ parts of the former to 12½ parts of the latter); its viscosity has been less than 25 per cent. greater than that of water (say, 38½ seconds for the oil as compared with 34 seconds for water); it has distilled almost wholly within the temperature limits of burning oil (to wit, 250° F. to 600° F.) (97$\frac{8}{10}$% by volume coming over between 400° and 575° F.); the portions distilling in 25° F. intervals between 400° F. (the commencement of distillation) and 550° F. have ranged from about 34° B. for the lightest to about 23° B. for the heaviest of these six portions; and the oil has necessarily possessed the characteristics of the more highly carbonaceous of the two oils obtained out of burning oil distillate from petroleum by subjecting such distillate to a differentially solvent menstruum in contradistinction to oil obtained by distillation from gas tar and the like or by addition of water or by steaming from acid sludge from treatment of petroleum, and having the like percentage of car-

bon to hydrogen, viscosity, boiling point and gravity. The main part of the menstruum (methyl alcohol) is recovered in a nearly anhydrous state by the evaporation of the same from the oil by dry heat alone (no free steam) in stills *c d* with rectification of the vapors in column *f* and dephlegmator 746 and condensation of the so rectified vapors in condenser *g*. The rest of it is recovered first in a state of dilution with water by the distillations with introduction of free steam into the liquid in stills *e h* and above the liquid in stills *k l*, with condensation of the vapors in condensers *m* and *j*, and then in a nearly anhydrous state by distilling the aqueous condensate from condensers *m j* (after separation of oil in the inverted siphons 901 and 905, Figs. 7, 8 and 9), the distillation being performed in section *q* of column *q r s* and in stills *n* and *p*, with rectification of the vapors in column *q r s* and condensation of the nearly anhydrous vapors in condenser *t*. The so recovered menstruum is returned to the oil washing apparatus by pipe 34, with or without intermediate storage tanks 76 as may be necessary or be thought proper; and it is used over and over again, with such replenishment (if any) as may be necessary.

A slightly modified mode of obtaining the same burning oil (kerosene) and the same highly carbonaceous oil is to distil the before mentioned cracked oil (representing 82 per cent. by volume of the crude Beaumont oil) until the entire burning oil fraction (55 per cent. of the crude oil) is off and to separate the light end (7% by volume of the crude oil) from the heavier portion of said fraction in a steam heated still with rectification of the vapors. Said heavier portion would then be subjected to the menstruum; and the washed oil would be mixed with the light end and finished by the sulfuric acid treatment, the menstruum being recovered and the oil of higher ratio of carbon to hydrogen being collected.

When considered necessary or desirable, the preliminarily separated light end can be subjected to the hydrocarbon separating menstruum; and the latter can be removed from it, or from other oil, in any appropriate way. The oil which is to be subjected in whole or in part to the differentially solvent menstruum can be subjected to any desired preliminary treatment without exceeding the limits of the invention.

The foregoing description will also enable those skilled in the art to subject to the differentially solvent menstruum the heavier illuminating oil fraction of Beaumont petroleum and either or both the illuminating oil fractions of other petroleum as herein above set forth.

The oil can be washed with the menstruum as often as may be necessary to accomplish the desired and feasible separation of hydrocarbons. The ratio of menstruum to oil can be varied. Good results with the burning oil fraction of Beaumont petroleum have been obtained by distilling the before mentioned cracked oil (equal to 82 per cent. by volume of the crude oil) with collection separately of a light end (equal as before to 7 per cent. of the crude oil) and a heavier portion of the burning oil fraction equal to 35 per cent. of the crude oil and by subjecting this latter to four times its volume of the nearly anhydrous methyl alcohol, with subsequent separation of the latter, admixture of the washed oil with the light end, and treating the mixture with sulfuric acid of 66° B.; but the yield of burning oil was less and its quality little (if any) better than that of the oil obtained by washing with six volumes of methyl alcohol, the cut representing 48 per cent. of the crude oil and composed of the heavier portion of the burning oil fraction after the light end (equal to 7 per cent. of the crude oil) had been separated therefrom.

The composition of the two oils obtained from the burning oil fraction of petroleum by the separating action of the differentially solvent menstruum will depend (1) upon the kind of crude petroleum whose burning oil fraction is subjected thereto, (2) upon the particular composition of the crude petroleum of the kind treated, (3) upon the particular points at which cuts are made in the fractional distillation, (4) upon the condition of the oil as cracked or uncracked, (5) upon whether all or part only of said fraction is subjected to the menstruum, (6) upon the particular character of the menstruum employed, (7) upon the ratio in volume of the menstruum to the oil subjected thereto, (8) upon the mode of washing, (9) upon the number of times the washing is performed, and possibly (10) upon other conditions. The smoky oil, for example, could be run again through said apparatus and be separated into two oils, one of which would have a higher ratio of carbon to hydrogen than the other. Moreover, oils of various compositions could be obtained by admixture with other oil; and in some cases, at least, such mixed oil might be recognizable as composed in part of one or other of the new or improved oils of the present invention.

To determine whether a given oil of the burning oil fraction of petroleum is smoky or non-smoking, the same may be burned in a common flat flame lamp with the wick projecting between an eighth and a quarter of an inch above the cone and with a porcelain dish supported between a half inch and an inch above the top of the lamp chimney, so as not to interfere with the draft. If substantially no soot should be deposited on the dish in half an hour, the oil may be re-

garded as non-smoking; while a substantial deposit of soot in half an hour's burning in the described manner would indicate a smoky oil. Another test would be to determine the reflective index of the oil with a refractometer. An index of 1.4545 would indicate a non-smoking oil; one of 1.4600 would indicate a smoky oil. A burning oil (kerosene) would probably be practically non-smoking with a refractive index of 1.4560 and may possibly be so with an index somewhere between 1.4560 and 1.4600.

To determine the proportion of an oil which is readily attacked by fuming sulfuric acid (ten per cent. anhydrid) the acid can be applied in small lots to a sample of the oil until after settling and decantation the acid continues to fume. The total amount withdrawn by the acid represents the percentage of hydrocarbons readily attacked by fuming sulfuric acid.

The statement of certain omissions, additions or other modifications herein is not intended to exclude other omissions, additions and substitutions within the spirit of the invention.

The present application is a division and continuation of my application of October 4, 1902, No. 125,967, which latter included claims to apparatus as well as to process and product and has been divided in consequence of official requirement.

The petroleum of the hereinafter written process claims is primarily Beaumont or analogous petroleum as hereinabove defined; but it is not exclusively so, except in claim 1. In the remaining process claims other petroleum having a burning oil fraction denser than that of Pennsylvania or Lima petroleum is by extension included along with Beaumont or analogous petroleum; while in process claims numbered 3 and following other petroleum in general is by further extension included also.

The burning oil fraction or the illuminating fraction of the petroleum is primarily to be treated in the form of burning oil distillate or an illuminating oil distillate; but it is intended by extension to include the treatment of the same in other forms also. The expression "illuminating oil fraction" applies to either the burning oil fraction or the next heavier fraction of petroleum or to both of these fractions taken together; and an "illuminating oil distillate" is one composed mainly (if not wholly) of hydrocarbons distilling over within the temperature limits of one or other or of both of said fractions. The distillate may be cracked or uncracked; except in claim 8, which recites cracked oil exclusively.

The menstruum primarily intended is one which has a greater solvent action on the smoky than it has on the non-smoking hydrocarbons which compose the burning oil fraction of Beaumont petroleum; but it is also intended to include a menstruum in general, such as methyl alcohol or ethyl alcohol, which has a differentially solvent action on the smoky and non-smoking hydrocarbons respectively of said burning oil fraction of Beaumont petroleum.

Oil from which hydrocarbons having a greater ratio of carbon to hydrogen than the oil itself have been separated has a lower refractive index and is also lighter in gravity than in its untreated state; and, at least as a general proposition, the burning oil fraction of petroleum which is naturally lighter in gravity than that of another is likewise lower in refractive index. It is possible, therefore, to compare the improvement in refractive index effected by treating oils of different gravities or by mixing treated and untreated oils of like boiling point and different gravities only by making an allowance in the refractive index for difference of gravity. In claims 15, 16 and 17 allowances, estimated as reasonable, are recited.

I claim as my invention or discovery;

1. The process of treating Beaumont or analogous petroleum, by subjecting part at least of the smoky burning oil fraction of such petroleum to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, and thereby effecting a sufficient separation of such hydrocarbons from each other for the obtainment of a non-smoking burning oil (kerosene), substantially as described.

2. The process of treating petroleum having a denser burning oil fraction than that of Pennsylvania or Lima petroleum, by subjecting part at least of an illuminating oil fraction of such petroleum to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, and thereby effecting a useful amelioration of said fraction in respect to smokiness or tendency thereto, substantially as described.

3. The process of treating petroleum, by subjecting part at least of an illuminating oil fraction of the petroleum to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, and thereby obtaining an oil of such fraction which has a less ratio of carbon to hydrogen than that possessed before treatment by the oil from which it has been so derived, substantially as described.

4. The process of treating petroleum, by mixing oil containing part at least of an illuminating oil fraction of the petroleum

with a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, separating the two resulting liquids from each other, and separating the menstruum from the hydrocarbons in solution therein, substantially as described.

5. The process of treating petroleum with repeated use of the same body of hydrocarbon separating menstruum, by mixing an oil containing part at least of an illuminating oil fraction of the petroleum with a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont petroleum, separating the two resulting liquids from each other, separating and recovering the menstruum from the hydrocarbons in solution therein, subjecting other oil of the same description to the so recovered menstruum, and so continuing, substantially as described.

6. In obtaining burning oil from petroleum, the improvement consisting in subjecting the less volatile portion of the burning oil fraction of the petroleum to a treatment whereby the removal therefrom is effected of hydrocarbons which are readily attacked by fuming sulfuric acid, and mixing the so treated portion with a more volatile portion of the same petroleum which has not received such a treatment, substantially as described.

7. For obtaining burning oil from petroleum, the process of three steps whereof one step consists in distilling the light end from the heavier portion of part at least of the burning oil fraction of said petroleum, another step in subjecting said heavier portion after removal of said light end therefrom to a menstruum which has a different solvent action upon the smoky from that which it has upon the non-smoking hydrocarbons of the burning oil fraction of Beaumont oil, and the third step in mixing said lighter end with the so treated heavier portion, substantially as described.

8. For obtaining burning oil from petroleum, the process of two steps whereof the first consists in distilling the crude oil with cracking, and the second in subjecting cracked oil so obtained and containing part at least of the burning oil fraction of the petroleum to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont petroleum, thereby obtaining oil of said fraction which has a less ratio of carbon to hydrogen than that possessed before treatment by the cracked oil from which it has been so derived, substantially as described.

9. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene) in part below and in part above 400° F., and containing on an average in the portions distilling over in 25° F. intervals between 300° F. and 400° F. a larger percentage of hydrocarbons which are readily attacked by fuming sulfuric acid than in the portions distilling over in such intervals between 400° F. and 500° F. or 550° F., substantially as described.

10. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene) and in part at least above 400° F. and exhibiting to a recognizable extent the characteristics of an oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils having a like refractive index, like mobility and like boiling point obtained out of burning oil distillate from petroleum by agitating such distillate with sulfuric acid of 66° Baumé or with fuming sulfuric acid (ten per cent. anhydrid), and removing the resultant acid sludge, substantially as described.

11. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene) in part below and in part above 400° F., exhibiting in the larger number at least of the portions distilling over in 25° F. intervals a greater density than is exhibited by those distilling over in the same temperature intervals from Pennsylvania petroleum, and containing on an average in the portions distilling over in 25° F. intervals between 300° F. and 400° F. a larger percentage of hydrocarbons which are readily attacked by fuming sulfuric acid than in the portions distilling over in such

intervals between 400° F. and 500° F. or 550° F., substantially as described.

12. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene), and in part at least above 400° F., exhibiting in the larger number at least of the portions distilling over in 25° F. intervals a greater density than is exhibited in those distilling over in the same temperature intervals from Pennsylvania petroleum, and also exhibiting to a recognizable extent the characteristics of an oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils having a like refractive index, like mobility and like boiling point obtained out of burning oil distillate from petroleum by agitating such distillate with sulfuric acid of 66° Baumé or with fuming sulfuric acid (ten per cent. anhydrid), and removing the resultant acid sludge, substantially as described.

13. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene) in part below and in part above 400° F., exhibiting in the portions distilling over between 400° F. and 500° F. or 550° F. an average bromin absorbing capacity of not less than six per cent., and containing on an average in the portions distilling over in 25° F. intervals between 300° F. and 400° F. a larger percentage of hydrocarbons which are readily attacked by fuming sulfuric acid than in the portions distilling over in such intervals between 400° F. and 500° F. or 550° F., substantially as described.

14. A new or improved petroleum or hydrocarbon oil having a refractive index less than that of oil of like boiling point obtained from Beaumont petroleum by fractional distillation and treatment with sulfuric acid of 66° Baumé, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the temperature limits of burning oil (kerosene) and in part at least above 400° F., exhibiting in the portions distilling over between 400° and 500° F. or 550° F. an average bromin absorbing capacity of not less than six per cent., and also exhibiting to a recognizable extent the characteristics of an oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils having a like refractive index, like mobility and like boiling point obtained out of burning oil distillate from petroleum by agitating such distillate with sulfuric acid of 66° Baumé or with fuming sulfuric acid (ten per cent. anhydrid), and removing the resultant acid sludge, substantially as described.

15. A new or improved petroleum or hydrocarbon oil having a mobility sufficient for burning oil (kerosene), distilling largely at least within the limits of burning oil (kerosene) in part below and in part above 400° F., exhibiting in the portions distilling over in 25° F. intervals between 400° F. and 500° F. an average refractive index not more than 1.4590 for an average gravity of 36½° B. with a deduction of fifteen ten thousandths for each degree Baumé, which said average gravity may be lighter than 36½° B. and with an addition of twenty ten thousandths for each degree Baumé, which said average gravity may be heavier than 36½° B., and containing on an average in the portions distilling over in 25° F. intervals between 300° F. and 400° F. a larger percentage of hydrocarbons which are readily attacked by fuming sulfuric acid than in the portions distilling over in such intervals between 400° F. and 500° F. or 550° F., substantially as described.

16. A new or improved petroleum or hydrocarbon oil having a mobility sufficient for burning oil (kerosene), distilling largely at least within the limits of burning oil (kerosene) in part below and in part above 400° F., exhibiting in the portions distilling over in 25° F. intervals between 400° F. and 500° F. an average refractive index not more than 1.4590 for an average gravity of 36½° B. with a deduction of fifteen ten thousandths for each degree Baumé, which said average gravity may be lighter than 36½° B. and with an addition of twenty ten thousandths for each degree Baumé, which said average gravity may be heavier than 36½° B., and also exhibiting to a recognizable extent the characteristics of an oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a mentruum which has a differ-

ent solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils having a like refractive index, like mobility and like boiling point obtained out of burning oil distillate from petroleum by agitating such distillate with sulfuric acid of 66° Baumé or with fuming sulfuric acid (ten per cent. anhydrid), and removing the resultant acid sludge, substantially as described.

17. A new or improved petroleum or hydrocarbon oil, having a mobility sufficient for burning oil (kerosene), distilling largely at least within the limits of burning oil (kerosene) and in part at least above 400° F., and exhibiting in the portions distilling over in 25° F. intervals between 400° F. and 500° F. an average density not less than about 35° B. nor more than about 38° B. and a refractive index not more than 1.4590 for an average gravity of 36½° B. with a deduction of twenty ten thousandths for each degree Baumé, which said average gravity may be lighter than 36½ B. and with an addition of twenty ten thousandths for each degree Baumé, which said average gravity may be heavier than 36½° B. and also an average bromin absorbing capacity of not less than six per cent., substantially as described.

18. A new or improved petroleum or hydrocarbon oil containing carbon and hydrogen in the ratio of not less than about seven parts by weight of the former to one of the latter, having a viscosity not more than about 25 per cent. greater than that of water, distilling largely at least within the temperature limits of burning oil (kerosene) and in part at least above 400° F., exhibiting in the larger number at least of the portions distilling over in 25° F. intervals between 400° F. and 500° F. or 550° F. a density heavier by not less than ten degrees Baumé than the portions distilling over in the same temperature intervals from Pennsylvania petroleum, and also exhibiting to a recognizable extent the characteristics of oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils of like ratio of carbon to hydrogen, like mobility, and like boiling point obtained respectively by fractionally distilling gas tar or the like and by agitating the acid sludge of petroleum refiners with water or by steaming it, substantially as described.

19. A new or improved petroleum or hydrocarbon oil containing carbon and hydrogen in the ratio of not less than about seven parts by weight of the former to one of the latter, having a viscosity not more than about 25 per cent. greater than that of water, distilling largely at least within the temperature limits of burning oil (kerosene) and in part at least above 400° F., exhibiting in the larger number at least of the portions distilling over in 25° F. intervals between 400° F. and 500° F. or 550° F. a density heavier by not less than ten nor more than twenty degrees Baumé than the portions distilling over in the same temperature intervals from Pennsylvania petroleum, and also exhibiting to a recognizable extent the characteristics of oil which has been obtained out of burning oil distillate from petroleum by subjecting such distillate to a menstruum which has a different solvent action upon the non-smoking from that which it has on the smoky hydrocarbons of the burning oil fraction of Beaumont oil, that is to say, exhibiting said characteristics to such extent that the oil examined can thereby be distinguished from oils of like ratio of carbon to hydrogen, like mobility, and like boiling point obtained respectively by fractionally distilling gas tar or the like and by agitating the acid sludge of petroleum refiners with water or by steaming it, substantially as described.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

HERMAN FRASCH.

Witnesses:
F. W. LOTHMAN,
J. C. UPDEGROVE.