

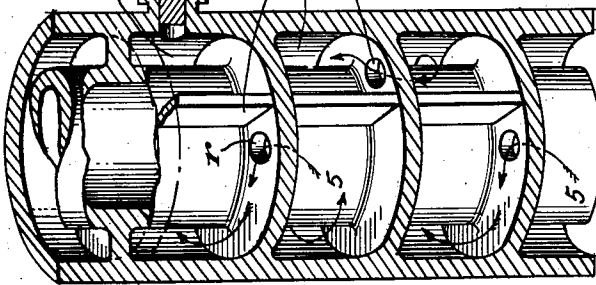
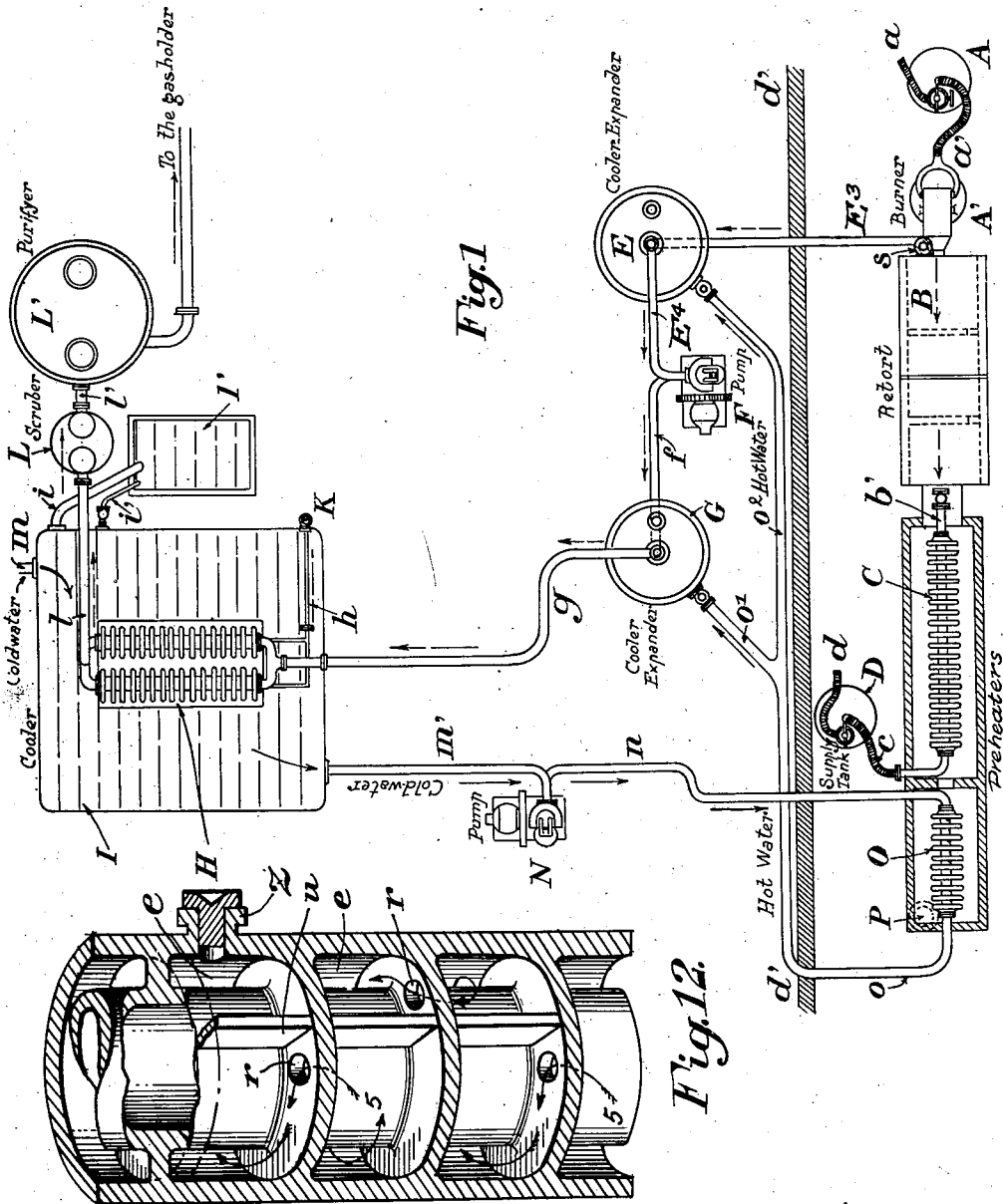
Oct. 28, 1930.

A. A. F. M. SEIGLE

1,779,828

METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original Filed July 16, 1920 5 Sheets-Sheet 1



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Oct. 28, 1930.

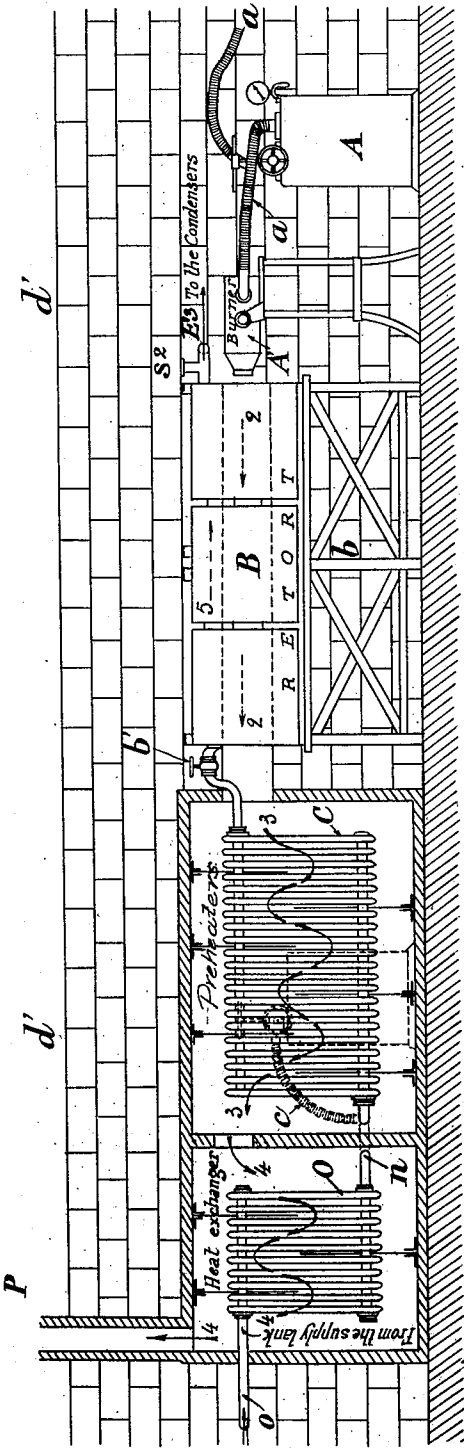
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METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original Filed July 16, 1920 5 Sheets-Sheet 2

Fig. 2



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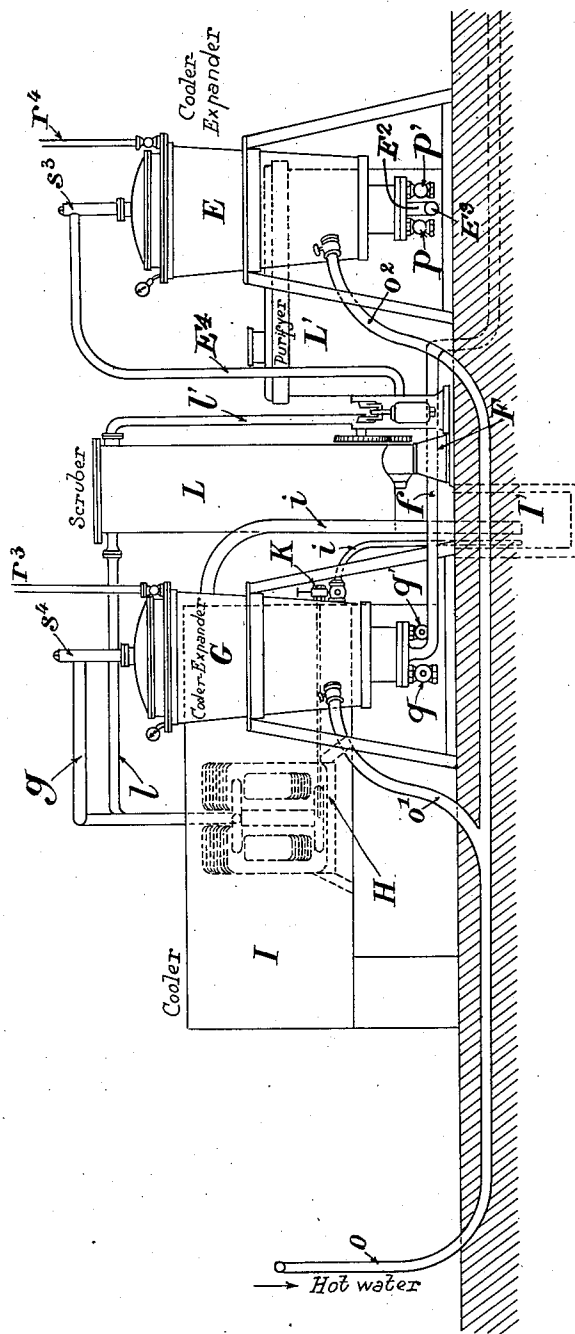
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METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original Filed July 16, 1920 5 Sheets-Sheet 3

Fig. 3



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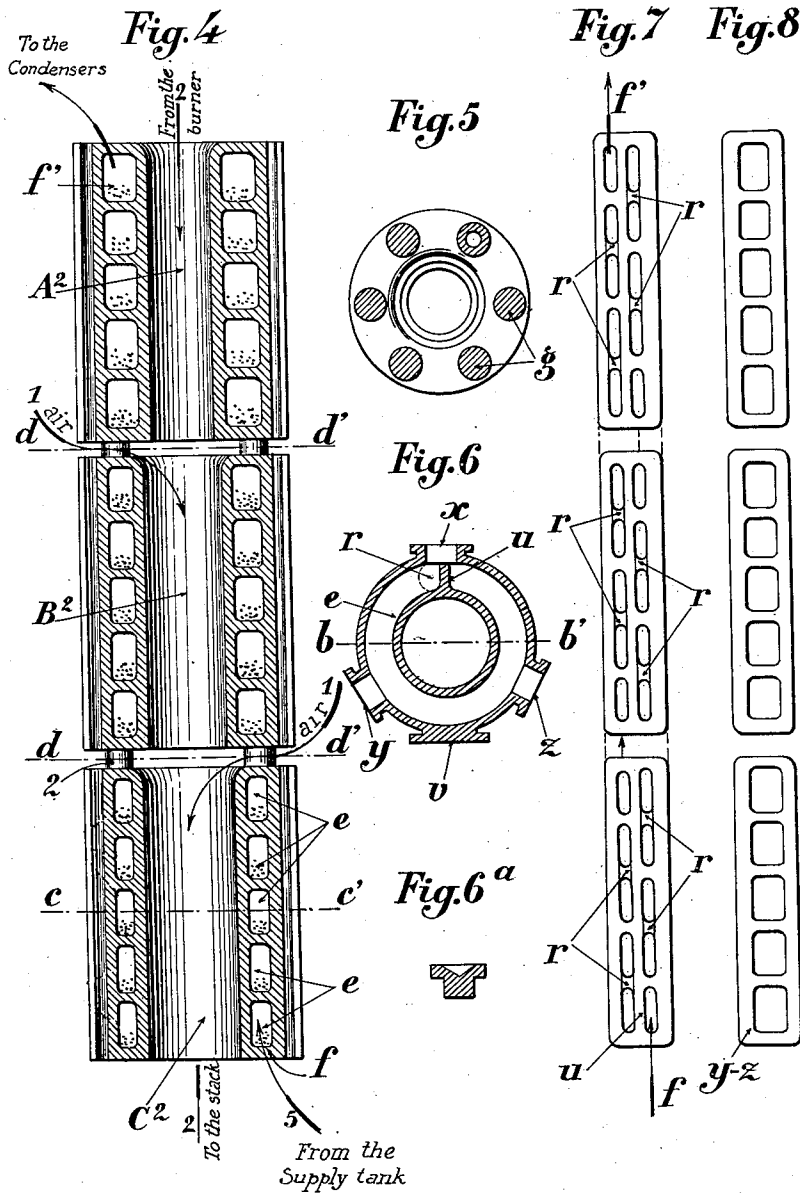
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METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original Filed July 16, 1920 5 Sheets-Sheet 4



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1,779,828

METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original Filed July 16, 1920 5 Sheets-Sheet 5

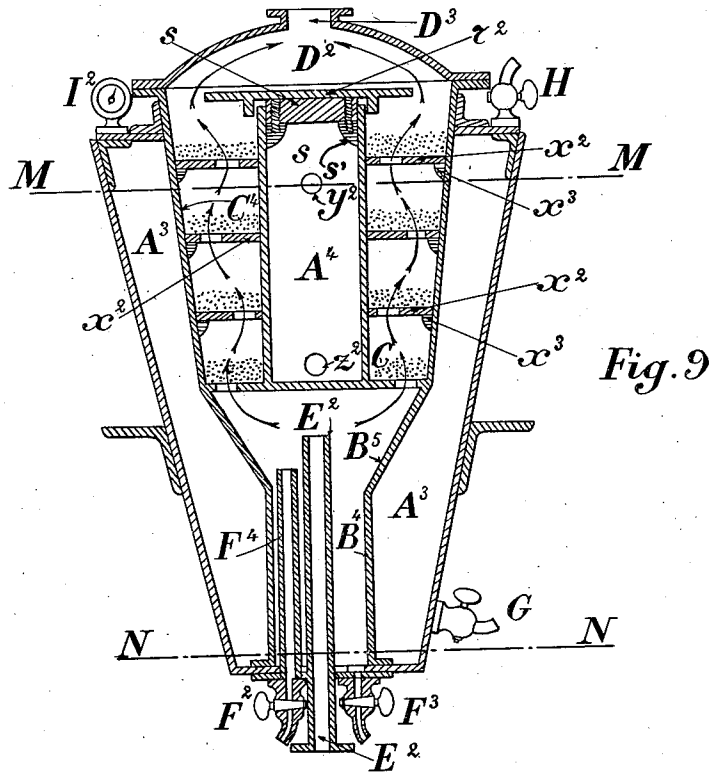


Fig. 9

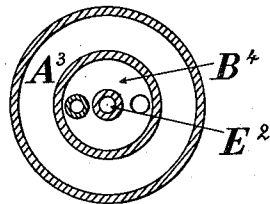


Fig. 10

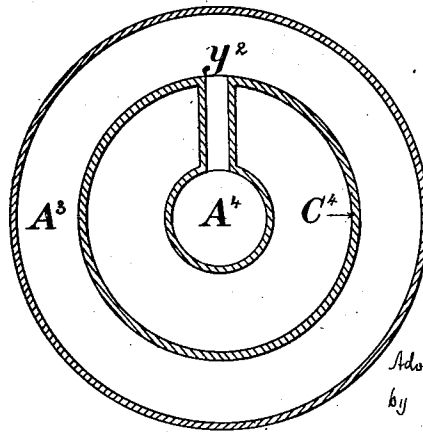


Fig. 11

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UNITED STATES PATENT OFFICE

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METHOD FOR TRANSFORMING AND DISTILLING HYDROCARBONS

Original application filed July 16, 1920, Serial No. 396,767, and in France July 17, 1918. Divided and this application filed January 14, 1926. Serial No. 81,144.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313)

I have filed application for patent in France July 17, 1918; July 25, 1918, and March 6, 1919, respectively, granted May 23, 1921; April 8, 1921, and January 23, 1922, under Nos. 524,901, 522,897 and 535,287; Germany filed July 15, 1920, granted February 22, 1927, under No. 445,657; Great Britain filed February 24, 1921, granted August 24, 1922, under No. 185,140; Argentine Republic filed February 2, 1923, granted July 14, 1923, under No. 20,271; Belgium filed December 24, 1921, granted December 31, 1921, under No. 300,575; Italy filed July 6, 1920, granted November 5, 1921, under No. 188,267; Mexico filed May 6, 1921, granted May 13, 1921, under No. 20,306; Poland filed January 14, 1922, granted October 30, 1926, under No. 5,899; Rumania filed January 4, 1922, granted same date under No. 7,249; Czechoslovakia filed December 21, 1921, granted May 5, 1924, under No. 14,064; Turkey filed February 27, 1923, granted September 26, 1923, under No. 20.

This invention described in this application which is a division of application No. 396,767 filed July 16, 1920, (Letters Patent No. 1,572,691) relates to an improved method for effecting in a practical and economical manner the regular and intensive vaporization and the subsequent transformation of certain hydrocarbons in the crude or the residual state such as mazout, heavy oils derived from peat, mineral oil, schist, lignite and other by-products of like chemical composition for instance heavy rosin oils.

The invention has for its purpose to obtain the following products in adjustable proportions: (1) hydrocarbons of a more or less light and inflammable nature which can have a considerable market value; especially as a fuel for automobile or aeroplane engines; (2) very heavy hydrocarbons of a more or less fatty character which can be employed to advantage for the lubrication of numerous machines; (3) a high grade gas of the same kind as oil gas already employed either for improving the quality of producer gas or for the lighting of railroad cars and of factories and small towns.

This high grade gas, in a sufficiently com-

pressed state, constitutes also a fuel which can be conveniently and advantageously employed in engines for tramways, motor coaches and the like.

The method according to the invention can be carried out by means of an improved installation comprising various apparatus as more fully described hereinafter.

In the accompanying drawings given by way of example:

Fig. 1 is a general diagrammatical plan view of an installation in accordance with the present invention.

Fig. 2 is an elevation view of a first group of apparatus forming a part of said installation.

Fig. 3 is an elevation view of a second group of apparatus.

Fig. 4 is a longitudinal section of a retort.

Fig. 5 is a cross section thereof along the line $d-d'$, Fig. 4.

Fig. 6 is a cross section thereof along the line $c-c'$, Fig. 4.

Fig. 6^a is a detail in cross section of a plug for said retort.

Figs. 7 and 8 are elevational views of the openings provided in the wall of the retort.

Fig. 9 is a longitudinal section of a cooling and expanding apparatus.

Figs. 10 and 11 are cross sections of the same along the lines N—N and M—M, Fig. 9, respectively.

Fig. 12 is a perspective detail view of a portion of the interior of a retort.

As shown in Fig. 1, the plant is composed of a first set of devices A, A', B, C, D, O and their accessory parts, which is separated by way of precaution by the wall $d' d'$ from a second set comprising the apparatus E, F, G, H, I, L, N and their accessories.

The first series of apparatus comprises one or more retorts B (Figs. 1 and 2); each retort is composed of several elements having the shape of a prism or a hollow or solid cylinder or like form and inside which is provided a coil shaped conduit having circulating therein the heavy hydrocarbons under treatment. The latter are previously raised to a temperature of about 150 to 180° C., this being readily effected by the use of

the heat of the burnt gases leaving the retort.

In the example shown, the retort B is composed of three elements A², B², C², which are placed end to end, and one upon the other if the retort is arranged vertically or in an inclined position. Each member has a central conduit or furnace formed therein, said conduits having a gradually increasing diameter. Between the inner wall and the external wall of each drum is left an annular space which is divided by cross partitions connecting the two walls into five annular passages *e* stopped by a longitudinal partition *u* and placed in communication by means of orifices *r* situated alternately on one side of the partition *u*, so as to form a coil shaped conduit. This construction is clearly illustrated in Fig. 12 in which the direction of flow of the hydrocarbon gases is shown by the arrows 5.

In the outer wall of each of the three elements are disposed three elongated rectangular openings *x*, *y*, *z* (Fig. 6) one of which, shown at the top of Fig. 6 and in front view in Fig. 7, shows the partition *u* at its middle part. These openings give access to the interior of the annular passages *e* for inspection and cleaning of the latter when desired or for disposing therein at certain points metal straw or turnings (iron, aluminium, etc.) granulated metal or the like, adapted to aid in the treatment of the hydrocarbons.

The said orifices are normally closed by stoppers having a corresponding shape (Fig. 6^a) and held in place by bolts or by arched closing members whereby they can be easily removed and replaced.

The elements A², B², C² which are situated in line and coaxially are connected together at the ends and spaced apart from one another by six cylindrical tenons *g* (Figs. 4 and 5), one of which is hollow and affords communication between the coil portion of the adjacent elements A², B², C².

The whole apparatus lies on its supporting frame by means of the base portions *v* (Fig. 6) forming part of the outer walls and secured to the frame by bolts or screw spikes.

The retort which has just been described is operated in the following manner.

At the inlet end, is disposed a projection device A' (Figs. 1 and 2) which projects axially into the central conduit or furnace of the retort and in the direction of the straight arrow 2, a stream of flame formed by gasified or atomized fuel which burns while mixing with the outer air which is automatically introduced (as shown by the curved arrows 1) through the spaces between the successive elements of the retort (Fig. 4) and produces a greater or less degree of heat in the retort according to the force of projection and the density of the said stream of flame.

The hydrocarbon which is to be treated is delivered into the coil conduit *e*, through the orifice *f* (Fig. 4) and passes successively through the whole series of convolutions of the three retort elements, being discharged at the other end *f'* of the coil passage after having followed the general direction shown by the arrows 5 and being submitted to a gradually increasing heating action.

The metal turnings placed in the coil conduit will divide and so to speak laminate into very fine streams the hydrocarbons and already more or less gasified vapor is produced in the first portion of this circulation, and the well known action of superheating upon the hydrocarbon vapors now becomes much stronger for a given temperature and also sufficiently easy to regulate. On the other hand since the iron and aluminum turnings have an active though small capacity for absorption of atoms of carbon and hydrogen at the respective temperatures of 650° and 450° C., this will afford another cause for the rupture of chemical equilibrium of the hydrocarburated molecules, which cause of rupture will be added in an efficient manner to that resulting from the methodical superheating to whose effects these same molecules are subjected at the said respective temperatures by reason of the successive passage of the same through the layers of aluminum turnings at about 450° and then through the layers of iron turnings at about 650° C.

However, as is well known to specialists, "all systems in chemical equilibrium will be submitted to a transformation in a determined sense by reason of the variation of a single one of the factors of this equilibrium such as pressure, temperature, electromotive force or concentration of the bodies under reaction." Said transformation, in the present case, causes an energetic increase of the decarburetted and the depolymerizing effect on the molecules in question. The result is that there is no sudden decomposition of the original hydrocarbons, as in an ordinary cracking operation, but a mere displacement of the atoms of hydrogen and carbon, no deposit of carbon taking place in the retort. In other words the percentage composition in carbon and hydrogen of the gaseous hydrocarbon mixture issuing from the retort is substantially the same as that of the raw hydrocarbons.

The installation shown comprises also two primary gas cooling and expanding apparatus E and G (Figs. 1 and 3).

One of the characteristic features of the apparatus resides in the fact that a sudden but purposely incomplete cooling of the hydrocarbon vapors or gases is obtained therein by the use as a cooling medium, not of a current of cold air or water as in the standard types of refrigerators for gases or vapors at high tem-

peratures, but of water in ebullition at a variable temperature.

Each cooler-expander (Figs. 9, 10 and 11) comprises an outer receptacle of iron or steel plate, A^3 , and a second receptacle more or less permanently secured in the middle of this latter. This inner receptacle comprises three cylindrical or truncated cone shaped portions preferably of cast iron, steel or the like, or of metal sheet (iron or copper) and welded together in order to provide a sufficient heat conductivity for the entire assemblage of these three parts.

These three portions comprise the cylinder B^4 having at its base a circular flange which is bolted to the bottom of the outer receptacle or digester A^3 ; the truncated cone shaped portion B^5 which connects the said cylinder B^4 to the upper truncated cone shaped part C^4 , this being closed at the lower part by a disk having therein a series of orifices near the periphery (Fig. 9) and finally the cylindrical portion A^4 which is in communication with the outer receptacle A^3 by means of two horizontal connecting tubes y^2 and z^2 . This cylindrical portion A^4 is closed at the top by a metal stopper s which is permanently held in place by means of four screws cooperating with the four lugs or shoulders s' .

Upon the bottom of the outer receptacle A^3 are bolted the flange of the cylinder B^4 and a sufficiently thick disk of cast iron, steel or bronze having integral therewith the central tube E^2 . On this same disk are screwed two cocks F^2 and F^3 one of which carries a pipe F^4 . This pipe extends up to a certain height within the part B^5 and serves to make sure that the level of the liquid therein is above its upper end.

In the annular space between the central receptacle A^4 and the inner wall of the cone C^4 are disposed three metal plates x^2 which rest upon suitable supports x^3 (Fig. 9). Two of these plates have a transverse cut out portion whereby they can be inserted below the connecting tube y^2 . The three plates x^2 are provided with apertures which are so disposed that the apertures of one plate are staggered relatively to those of the adjacent plates, so that the path of the gases through said holes is that shown by the arrow (Fig. 9).

The operation of placing the plates or disks x^2 and the disk y^2 is carried out at the same time as the filling of the spaces between the said three disks and the upper disk by iron turnings, granulated cast iron or like material adapted for the thermo-chemical treatment of the heavy hydrocarbons to be treated. When these operations are completed, the cap D^2 is hermetically secured to the truncated cone shaped portion C^4 .

The operation of the said cooler-expander is as follows:

For the sake of clearness the complete digester formed by the receptacle A^3 , the two

tubes y^2 and z^2 and the central cylinder A^4 are assumed as entirely filled with water already heated to the desired boiling temperature, the latter being obtained by varying the pressure and observing the same by means of the pressure gauge I^2 (Fig. 9).

A valve or cock (not shown here) is now opened to connect the tube E^2 for instance with the discharge piping of a retort such as that described above in which a certain quantity of mazout or astatki is treated.

The hydrocarbon vapors thus admitted by the tube E^2 will now be caused to expand in the portions B^4 and B^5 of the cooling and expanding apparatus and will circulate through the sets of staggered orifices as shown by the arrows (Fig. 9) and through the entire mass of iron turnings, granulated cast iron or like material filling the annular space between the inner cylinder A^4 and the truncated cone shaped portion C^4 as explained above. They will finally escape through the discharge orifice D^3 (Fig. 9).

The outer surfaces of the cylinder B^4 and the truncated cone shaped portions B^5 and C^4 as well as the inner surface of the cylinder A^4 are in direct contact with the water which has been preliminarily heated to its boiling temperature. Now, as is well known, the vaporization of water absorbs, in a practically instantaneous manner, at least ten times the amount of heat that this same quantity of water would absorb more or less slowly when passing for instance from 10 to 60° C. as it is the case for many of the customary cooling devices. It is now obvious that, when passing through the cooling and expanding apparatus, the gases or vapors discharged by the retort will be incompletely, but very rapidly cooled, thus giving up a considerable proportion of their heat which will be instantaneously absorbed by the instantaneous vaporization of a corresponding quantity of water already at its boiling temperature. This results in a partial depolymerization of the said gases or vapors.

The installation comprises additional apparatus arranged as follows:

The liquid fuel of cheap quality which is used for heating the retort is delivered into a pressure tank A (Figs. 1 and 2) by the pipe a . Said fuel is led by the pipe a' into a gasifying apparatus A' of a standard type, which may be replaced either by a steam or compressed air atomizer or by a plurality of suitable burners supplied with producer gas or the like.

The powerful flame or flames thus produced are directed into the central conduit or furnace of the retort B. As previously stated, said retort may be arranged either horizontally, as shown, or vertically or in an inclined position.

The gases of combustion which leave the heating conduit of the retort B will travel in zigzag path (Fig. 2) through the double

rectangular chamber of a recuperator of heat, wherein they pass according to the arrows 3 and 4 and they will be successively cooled upon the outer walls of the two heat exchanging members C and O and they are finally discharged into the atmosphere through the stack P.

The hydrocarbon to be treated is constantly maintained in the cold and liquid condition and at a low pressure of about 1 atmosphere, in the tank D (Fig. 1). It is delivered into said tank by the pipe *d*. This cold hydrocarbon is conveyed by the pipe *c* provided with a cock into the heat-exchanger C (Figs. 1 and 2) where it is highly heated and then introduced by the pipe *b'* into the retort B. The more or less superheated hydrocarbon vapors escaping from the retort B are conveyed by a pipe *E*³ through the partition wall *d'* *d'* to the central intake pipe *E*² of the first cooler-expander E (Figs. 1 and 3) the construction of which has been explained above.

The uncondensed portion of the said partially gasified vapors is then taken off through the pipe *E*⁴ from the upper outlet *D*³ of said apparatus E by means of the motor pump F which delivers the gases through a pipe *f* to a second cooler expander G. The uncondensed vapors are discharged from the latter apparatus through the pipe *g* directly into the final cooling apparatus H, I, I' (Figs. 1 and 3) whence the noncondensable gases pass through the pipe *l* to the scrubber and the purifier L, L' and are finally collected in a gas holder.

The remaining portion of the gases and vapors which are led from the top of the apparatus G into the apparatus H, I, I' (Figs. 1 and 3) is cooled to a rather low temperature by passing within a cooler having cold water circulated therethrough, this water being brought by means of the pipe *m* into the lower part of the tank I and leaving this latter through the pipe *m'* whose outlet is situated almost at the upper edge of the said tank.

It will be seen that the water which is supplied as cold as possible through the pipe *m*, reaches the pipe *m'* in a more or less warm state and is sucked through the latter by means of a motor pump N (Fig. 1) which delivers it through the pipe *n* into the heat exchanger O, and thence arrives, through the pipe *o* and its two branches *o*¹ and *o*², and substantially at the boiling temperature into the outer chambers of the two cooler-expanders G and E (Figs. 1 and 3) from which it is finally discharged by the pipes *r*³ and *r*⁴ (Fig. 3) in the state of saturated steam.

In connection with the operation of the plant above described and more particularly with respect to the cooler-expanders E and G, it will be noted that in the latter apparatus, the rupture of chemical equilibrium is

chiefly due in the first place to an intense and very sudden cooling and secondly to the rather strong expansion or reduction of pressure to which the hydrocarbon gases or vapors are submitted when passing successively through each of the said cooler-expanders.

On the other hand, this rupture of chemical equilibrium is further caused by the action of the successive layers of metal scrapings or turnings which are placed in any desired quantity within the inner receptacle C⁴ (Fig. 9) of each cooler-expander, upon the removable plates *x*².

In order to properly specify this action it will be necessary to explain how and for which reason the cooler-expanders E and G (Figs. 1 and 3) operate at different inner temperatures and pressures.

The question will be simplified by considering only the case in which, for instance, the pressure of the steam, and therefore that of the feed water to be vaporized, will be 5 kilogrammes per square centimeter in both the outer receptacles or chambers of these two cooler-expanders, a difference of 80° C. being however obtained between their respective inner temperatures due to the fact that the outer receptacle of the apparatus E is first filled with a saturated solution of chloride of calcium up to the normal level.

Under these conditions of operation, the more or less gasified vapors will escape from the retort at a temperature of, for instance, 650° C. and a pressure of 1.2 kilogrammes per square centimeter are then brought very suddenly to a theoretical temperature of 235° C., but a practical temperature of 260° C. and also to a pressure of about 4 hectogrammes per square centimeter, by their passage through the first cooler-expander E whence the motor pump F draws out the same and compresses them while at the same time heating them to a slight degree; in this manner they are submitted to a second and still considerable reduction of temperature and pressure by flowing through the second cooler-expander G, entering therein at about 300° C. and under a pressure of 9 hectogrammes per square centimeter and leaving the same at about 160° C. under a pressure of about 2 to 3 hectogrammes.

The different temperatures and pressures of the hydrocarbons under treatment are controlled by means of fixed or movable pyrometers and pressure gauges disposed at various suitable points for instance at *s*², *s*³, *s*⁴ (Figs. 2 and 3). The said temperatures and pressures are regulated by making the necessary variations of the discharge orifices of the valves or cocks disposed upon the piping connecting the different apparatus, especially at *b'*, *E*³ and *E*⁴ (Figs. 1 and 2).

In short, when passing successively through both cooler-expanders E and G, the

temperature of the hydrocarbon molecules is lowered successively from 650° C. and then to 160° C. However, as already explained, the iron and aluminium turnings aid in producing the rupture of chemical equilibrium of the said molecules at the respective temperatures of about 450° and 650° C., on the other hand, thin copper turnings have an active affinity for hydrogen between 300° C. and 500° C. and ferro-nickel turnings have a double affinity for carbon and hydrogen between 200° C. and 350° C.

In consequence, the successive layers of the above mentioned metal turnings or scrapings are to be distributed according to the thermo-chemical data above set forth, in the inner receptacles of the two cooler-expanders.

Each pair of cocks p, p', q, q' (Fig. 3), corresponding to those indicated by F^2 and F^3 in Fig. 9, serves to control the filling operation and to drain out the lower portion of the inner receptacle of one of the apparatus E and G (Figs. 1 and 3).

The very heavy condensed hydrocarbons drop into this portion, and are condensed by reason of the intense cooling action successively produced in the first and then in the second of the said cooler-expanders.

The cock K is employed for the discharge of the light and inflammable hydrocarbons which are condensed in the final cooling apparatus H, I (Figs. 1 and 3).

In certain cases where for instance it will be found advantageous to increase the output of liquid and very inflammable fuel to the detriment of the high grade gas, the above described plant can be completed by one of the systems now in use for the industrial hydrogenation of olive oil, linseed oil, fish oil and the like as well as hydrocarbons, by means of usual metals employed as catalyzers.

For this purpose, there may be provided a simultaneous suction and a very intimate mixture of the vapors discharged from the apparatus E (Figs. 1 and 3) with a portion of the high grade gas stored in the gas holder and to which hydrogen can be added if required, the perfect mixture thus obtained is thus delivered into the inner receptacle of the second cooler-expander which is previously provided with suitable catalyzer elements.

In the normal application of the above described thermo-chemical cycle, it is required to use heavy hydrocarbons properly so called, that is, not giving off vapor below about 200° C. or if possible 250° C.

However, in industrial practice, it may happen that an economical supply can only be obtained of crude hydrocarbons such as naphtha or crude oil. In this case it is first required to effect a previous separation of the heavy hydrocarbons by a mere distillation which can be very easily carried out by means of the installation represented in Figs. 1, 2

and 3, but exclusive of the apparatus for purifying and collecting the gas which are not required here. It is of course understood that the operation of the installation thus transformed should be carried out at temperatures suitable for the new and comparatively easy result to be here obtained and not with the use of the temperatures previously mentioned for obtaining the depolymerization of the hydrocarbons.

The invention is not restricted to the details of execution shown or described which have been given only by way of example.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is:

1. Method for the transformation of heavy hydrocarbons comprising a preheating stage for vaporizing the raw hydrocarbons, a main heating stage which consists in heating, in a very regular and gradual manner, the vapors formed during said preheating stage and the gaseous hydrocarbons produced during said main heating stage being successively submitted to the action of catalyzers, the maximum temperature being substantially comprised between 500° C. and 700° C. and the pressure being maintained below substantially 1.2 kilogrammes per square centimeter, and a number of successive cooling stages to which the mixture of gases and vapors issuing from said main heating stage are subjected the cooling medium used in the first cooling stage being boiling water at a predetermined superatmospheric pressure so that the sudden drop in the temperature of said gases and vapors through said first cooling stage be substantially comprised between 250° C. and 450° C.

2. In the method as claimed in claim 1 the further feature residing in that said mixture of gases and vapors is subjected, during said cooling stages, to the action of other catalyzers having respectively their specific action at the temperature in said stages.

3. In the method as claimed in claim 1 the further feature residing in that said mixture of gases and vapors is subjected, during said cooling stages, to the action of other catalyzers having respectively their specific action at the temperature in said stages, the catalyzers used respectively in said main heating stage and in said cooling stages being adapted for the conversion of the raw hydrocarbon vapors into condensable lighter hydrocarbons, the percentage composition in carbon and hydrogen of said latter hydrocarbons being substantially the same as that of the raw hydrocarbon.

4. In the method as claimed in claim 1, the further feature residing in that the gases and vapors resulting from each cooling stage are further submitted to a reheating and compression stage before they are submitted to the following cooling stage.

5. Method for the transformation of heavy hydrocarbons which comprises a heating stage which consists in heating said hydrocarbons by causing the same to circulate around a central furnace according to a path formed of successive and adjacent convolutions in planes at right angles to the direction of circulation of the heating gases within said furnace and alternately in opposite direction in the successive convolutions, the longitudinal direction of the hydrocarbon flow being opposite that of said heating gases through the furnace, the gases and vapors produced along said path flowing through catalyzers respectively disposed at successive points thereof and having respectively their maximum action at the temperatures prevailing at said successive points, the maximum temperature being substantially comprised between 500° and 700° C., and the pressure being maintained below substantially 1.2 kilogrammes per square centimeter, and a number of successive cooling stages to which the mixture of gases and vapors issuing from said main heating stage are subjected, the cooling medium used in the first cooling stage being boiling water at a predetermined super-atmospheric pressure so that the sudden drop in the temperature of said gases and vapors through said first cooling stage be substantially comprised between 250° C. and 450° C.

6. Method for the transformation of heavy hydrocarbons which comprises a preheating stage for vaporizing the raw hydrocarbons, a main heating stage which consists in heating, in a very regular and gradual manner, the vapors formed during said preheating stage, substantially up to 450° C., submitting the gaseous hydrocarbons thus produced to the catalytic action of aluminum and further heating the resulting hydrocarbons up to a maximum temperature between 500° C. and 700° C. and submitting them to the catalytic action of iron, the maximum pressure of said gaseous hydrocarbons being substantially 1.2 kilogrammes per square centimeter, and two successive cooling stages, to which the mixture of gases and vapors issuing from said main heating stage are subjected, by means of boiling water successively at a temperature between 250° C. and 300° C., and between 150° C. and 200° C., respectively in the presence of copper and ferro-nickel.

In testimony whereof I have signed my name to this specification.

ADOLPHE ANTOINE FRANÇOIS MARIUS SEIGLE.