

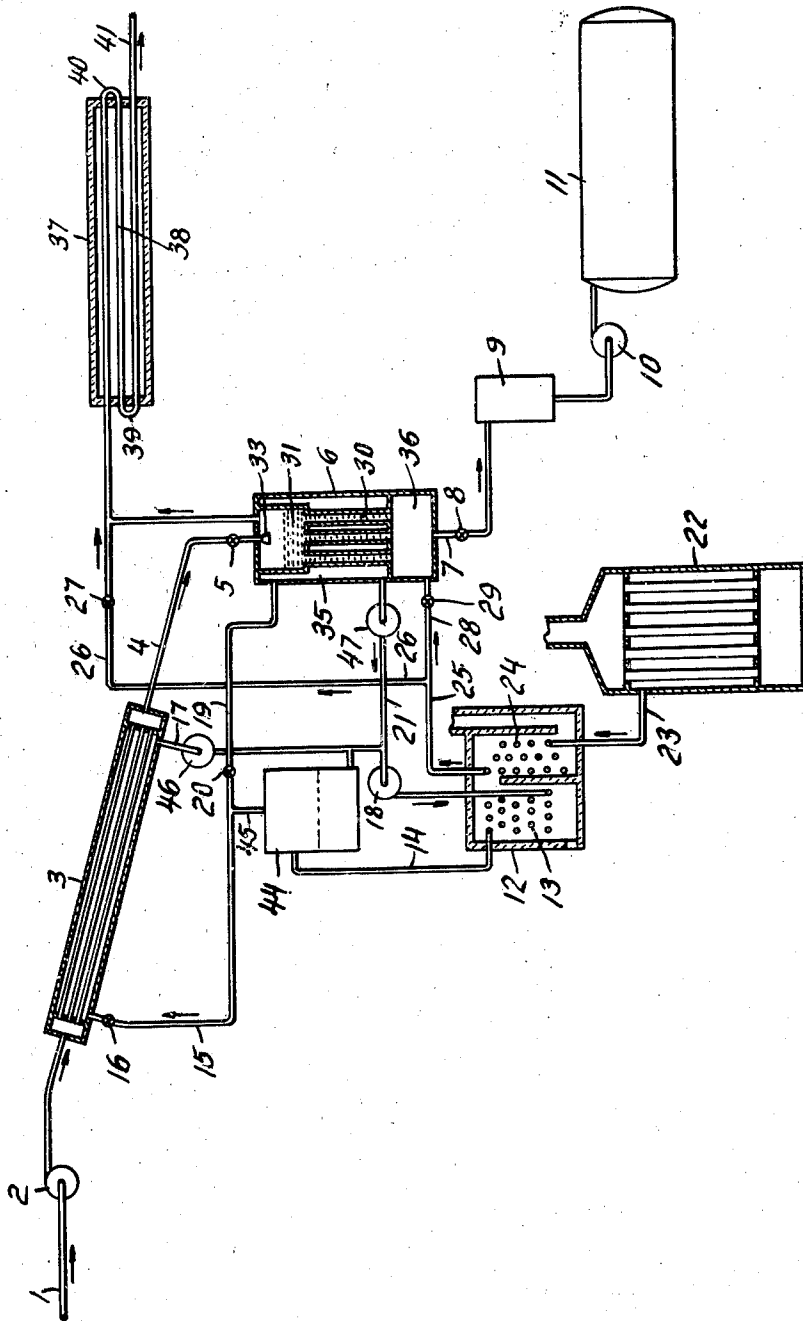
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PRODUCTION OF ETHYLENE, ETC

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PRODUCTION OF ETHYLENE, ETC.

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This invention relates to improvements in the vaporizing and cracking of sulfur-containing hydrocarbon oils, and more particularly the cracking of such oils at high temperatures for the production of olefins, such as ethylene, as primary products of the cracking operation.

The process of the invention has as one of its primary objects the conversion of the liquid hydrocarbon material treated into gas as the primary product of the cracking, to obtain a high yield, approximating the maximum yield, of olefin gases or vapors, such as ethylene and propylene, with carrying out of the cracking as a gaseous or vapor phase cracking operation with the supply of hydrocarbon vapors, admixed with steam, and free from entrained liquid, to the cracking treatment.

The cracking of various hydrocarbons has been practiced under various conditions and in different forms of apparatus. The usual cracking process has been developed with the view of producing the maximum possible amount of hydrocarbons which are liquid, rather than gaseous, at ordinary temperatures, and which are intended primarily for use as motor fuel or gasoline. Such cracking processes are commonly carried out by cracking in the liquid phase or condition, in such manner as to produce the greatest possible amount of lighter hydrocarbons, liquid under ordinary temperatures and pressures; and such processes are also commonly carried out in the vapor phase under conditions that will produce a minimum of gaseous products, that is, under conditions such that a considerable proportion of the gases unavoidably formed are reformed into lighter liquid products, as, for example, in the low temperature distillation or hydrogenation of oils and coals. In the production of gasoline by cracking heavier hydrocarbon oils by the vapor phase cracking process, the common procedure is to crack the oil vapors to an extent such that the quantity of liquid which condenses when the temperature is brought down to atmospheric is high, and approaches a maximum, while the gaseous products are low and are kept at a minimum.

In contrast with such cracking processes, where the production of gasoline, etc., is the primary product, the present invention has a different objective, namely, to vaporize and crack the oil to an extent such that the quantity of gaseous olefin hydrocarbons is high, and approaches a maximum, and such that the quantity of liquid which condenses when the temperature

is brought down to atmospheric is low and approaches a minimum.

The cracking process of the present invention is distinguished from cracking processes, whether in the gaseous or liquid phase, for making gasoline as the primary product, by the use of substantially higher temperatures, such that the hydrocarbon is cracked to produce a high yield of the desired gaseous olefins. In the ordinary cracking processes, for making gasoline, high pressures are commonly used; whereas in the present process high pressures are unnecessary, and in general disadvantageous, and the present process is advantageously carried out at relatively low pressures, around atmospheric, or somewhat higher or lower.

The sulfur-containing hydrocarbon oils used in the present process may be those such as are commonly used in cracking processes, such as light oils, intermediate or heavy hydrocarbon fractions or cuts, etc.; but the present process is particularly advantageous for cracking oils containing constituents vaporizable with difficulty, such as residues or residue-containing products from the distillation or cracking of petroleum oils. The present process enables such residues and residue-containing oils to be employed, with vaporization and utilization of such constituents, vaporizable with difficulty, without introducing unvaporized portions of such residues into the cracking zone. Crude oil, or crude oil which has been topped to remove various percentages of the lighter fractions, as well as residues from the distillation of crude oil or from cracking operations, etc., can advantageously be employed in the present process. Comparatively heavy fuel oil can thus be employed. Coal tars can also be employed.

Crude oil, or topped crude, containing both light and heavy fractions, can advantageously be treated as a whole, according to the present process, for the production of a high yield, approaching the maximum, of unsaturated hydrocarbon gases containing a large proportion of ethylene and propylene, as well as butylene and other olefins.

The temperatures employed in the present cracking process are materially higher than those commonly used for cracking oils to produce gasoline as a primary product. In general, the temperatures employed are in excess of about 1300° F., although temperatures as low as 1100° F. may be used. Temperatures around 1400 to 1600° F. have been found advantageous for the cracking of various crude oils and residues.

The present process can advantageously be carried out at atmospheric pressure, or at pressures somewhat above or below atmospheric. It is one advantage of the process that high pressures, such as are commonly used in cracking to make gasoline, are unnecessary, so that high pressure cracking stills are not required.

The process of the present invention is one in which steam is admixed with the hydrocarbons which are subjected to cracking, and in which the hydrocarbons are supplied to the cracking operation in the form of vapors, and in which the cracking is carried out in tubes made of a chromium-containing or nickel-containing alloy. The admixture of steam with the hydrocarbon vapors supplied to the cracking apparatus enables sulfur-containing oils to be employed, at the high temperatures used, and with avoidance of the disintegration of high-temperature alloy tubes which, in the absence of steam, would be disintegrated or corroded by the cracking of such sulfur-containing oils at such temperatures.

The high temperatures at which the cracking operation is carried out requires the use of tubes capable of withstanding such high temperatures. While iron or steel tubes can be used, the higher temperatures are such as would ordinarily indicate the use of tubes made of high temperature metals or alloys, such as nickel or alloys containing chromium or nickel or both chromium and nickel, etc. Such high temperature metals and alloys can be used with oils which are free from sulfur, and in the absence of steam; but when oil vapors containing sulfur are passed through such tubes, in the absence of steam, the tubes are attacked by the sulfur or sulfur compounds, and there is danger of rapid disintegration or corrosion, which makes impossible the use of such tubes with such oils under such circumstances. However, when steam is admixed with the oil vapors, as in the present process, it is possible to use such high temperature tubes, such as nickel or nichrome tubes, or tubes made of other chromium or nickel-chromium alloys, etc., without the objectionable disintegration or corrosion which is caused by sulfur-containing oils when steam is not used.

It appears to be important, in producing a high yield of olefins, to raise the admixed steam and hydrocarbon vapors to the cracking temperature rapidly and pass the steam and vapors through the cracking zone at such a rate that only a short time of cracking is permitted, so that prolonged heating is avoided. In general, the time of contact of the steam and vapors with the cracking temperature, or with the maximum cracking temperature, may be only a few seconds, or even as little as a fraction of a second. It is difficult to estimate definitely the time of contact of the steam and vapors at the cracking temperature, and the process can better be described with reference to the gallons of oil supplied per hour per cubic foot of tube space in the cracking zone, and the gallons of water supplied per hour (in the form of steam) per gallon of oil, as illustrated by the examples hereinafter given.

The ratio of steam to hydrocarbon vapors supplied to the cracking operation can be varied. Where steam is used in the preliminary counter-current vaporizer for aiding in the vaporization of high boiling constituents of the oil, the amount which is necessary or desirable for this vaporizing operation has to be taken into consideration, and may determine the proportions of

steam and oil vapors supplied to the cracking treatment.

The invention will be further illustrated by the accompanying drawing, and the process of the invention will be further described in connection therewith. The drawing shows in a somewhat conventional and diagrammatic manner, and without attempting to show the different parts to scale, an arrangement of apparatus illustrating the invention and adapted for the practice of the process of the invention; but it will be understood that the invention is not limited thereto. The drawing is diagrammatic or schematic in character.

In the apparatus illustrated, the oil supplied, such as crude oil, enters through the oil supply pipe 1 and is pumped by the liquid pump 2 to the preheater 3 from which the preheated oil flows through the pipe 4 to the top of the vaporizer 6, a valve 5 being provided in the line 4 to permit the use of a higher pressure in the preheater 3 than in the vaporizer 6, although the valve 5 may be left open so that there is no reduction in pressure at this point.

The vaporizer 6 has an outlet line 7 at the bottom for the unvaporized oil leading to the receiver 9, and with control valve 8 therein. From the receiver 9 the residue is pumped by the pump 10 to the fuel oil storage tank 11.

The furnace 12 is shown as a two-compartment furnace in the first of which is a heating coil 13 of a heater or boiler for vaporizing the liquid, for example, diphenyloxide, which is used in the preheater 3 and also in heating the vaporizer 6. The heating coil 13 is connected through the line 14 to the separator 44, in which a body of liquid is maintained, and in which the unvaporized liquid from the heating coil is separated from the vapors. The vapors from the separator pass through the line 45 and the branch line 15 to the space around the tubes of the preheater 3. The line 15 has regulating valve 16 therein. From the other end of the preheater a return line 17 having pump 46 therein leads the condensed liquid back to the suction of the pump 18 which forces the liquid through the heating coil 13.

A branch line 19, with valve 20 therein, leads from the line 45 to the heating jacket 35 of the vaporizer 6, and a return line 21 having pump 47 therein leads the condensed liquid back to the suction of the pump 18.

The heating coil 13 and vaporizer 44 form, in effect, a tubular boiler, with rapid circulation of the liquid by the pump 18, while the valves 16 and 20, and return pumps 46 and 47 permit the preheater and vaporizer to be operated at the same or different pressures.

The arrangement for preheating the oil in the preheater 3 and for heating the oil in the vaporizer 6 is such that the vapors of the liquid, such as diphenyloxide, are circulated through the preheater and the vaporizer, with regulation of the flow by means of the valves 16 and 20, and with return of condensed liquid by means of the pumps 46 and 47.

The other section of the furnace, which might be considered a separate furnace, although shown as arranged in the same furnace structure that contains the heating coil 13, has a steam preheater 24 to which steam is supplied by the steam boiler 22 through the steam line 23. From the superheater coil 24 the superheated steam is led by the line 25 and branch pipe 28 having regulating valve 29 therein to the bottom of the vapor-

izer 6. Another branch line 26, with regulating valve or cut-off valve 27 therein, permits additional steam to be supplied through this line, although this line may be omitted.

5 The vaporizer 6 is shown conventionally as having tubes 30 which may be filled with suitable filling material, and with distributing material 31 in the upper portion of the preheater. The preheated oil enters from the pipe 4 through the nozzle or distributor 33 which is so arranged as to distribute the incoming oil and prevent en-
10 trainment by the outgoing steam and oil vapors. A heating jacket 35 surrounds the tube 30 and the upper portion of the vaporizer, and a chamber 36 is arranged at the bottom.

The cracking furnace 37 is shown schematically with a series of parallel heating tubes or coils 38 of chromium-containing or nickel-containing alloy therein, connected to inlet and outlet head-
20 ers or manifolds 39 and 40 and with the line 41 leading from the cracking chamber to the condensing apparatus (not shown).

In the operation of the apparatus illustrated, the incoming oil, which may be incoming crude oil, or topped crude, etc., is supplied continuously and in regulated amounts by the pump 2 and may be preheated in the preheater 3 to a temperature which should be below that which will cause any appreciable cracking or coke formation. The
30 preheating of the oil is advantageously accomplished by the use of a condensing vapor, such as diphenyloxide, circulated from a heating furnace to the preheater. The preheater may be operated under pressure, with regulation and release of the pressure by the valve 5, but the use of pressure is not essential. Some of the lower boiling constituents of the oil may be vaporized in the preheater or on reduction of pressure as the admixed preheated oil and vapors enter the
40 top of the vaporizer 6 through the nozzle or distributing device 33.

The pressure in the vaporizer 6 need be only sufficient to force the fluid through the various parts of the system, although the pressure may be somewhat higher or lower than atmospheric.

45 In the vaporizer 6 the preheated oil flows downwardly countercurrent to and in intimate contact with the steam and vapors which pass upwardly. The preheater is of such a size, and is provided with suitable means for bringing about sufficiently intimate contact, to insure effective countercurrent action of the steam and liquid oil.

The steam as it ascends through the tubes of the vaporizer comes into direct contact with the liquid descending therein. In order to prevent or minimize condensation, in the upper part of the vaporizer, of heavier oil constituents vaporized in the lower part, the vaporizer is itself heated, as by means of a diphenyloxide heating jacket, as previously described, to prevent or minimize cooling of the admixed steam and vapors in the upper part of the vaporizer, such as would be caused by the absorption of heat during the vaporization of the liquid oils which are being vaporized in the
60 vaporizer. By the combined action of the steam and the heating jacket, the content of oil vapors admixed with the steam is progressively greater from the bottom to the top. Furthermore, the liquid oil entering the vaporizer at the top will, as it descends, come into direct contact with a mixed steam and oil vapor mixture containing a greater and greater proportion of steam and a progressively smaller amount of oil vapor as it progresses downwardly through the vaporizer.
70 At the extreme lowest end of the vaporizer the

residual oil, now freed of practically all of the more volatile constituents, will come into contact with hot steam free from oil vapor.

The extent to which the preheater 3 and the vaporizer 6 are heated by the diphenyloxide vapors can be regulated by the regulating valve 16 and 20 in the branch lines leading to these respective parts of the apparatus. The arrangement is such that the hot diphenyloxide vapors enter the vaporizer 6 near the top, and the condensed diphenyloxide returns to the diphenyloxide cycle through the return pipe 21. The result is that the temperature of the vaporizer is kept practically uniform throughout its length, and the tendency for any of the heavier liquid
15 constituents, which are vaporized, to condense out in the ascending vapors as these approach the top, is prevented or minimized.

As illustrative of the temperature to which the oil may be preheated in the preheater and vaporizer, or in the vaporizer, the upper limit will be that attainable by the heating means and the temperature at which excessive decomposition or cracking will take place. In general, the oil should be preheated to a temperature below that at which any appreciable amount of cracking takes place. The oil may thus be preheated, for example, to temperatures of from 400 or 500° F. up to temperatures around 700 or 800° F., or higher, depending somewhat on the oil and the method of preheating. For East Texas crude oil, the temperature to which the preheating is carried may be about 700° F.

Crude oils, topped crudes, coal tars, and residues containing heavy ends, which are advantageously treated by the present process, and with utilization of an unusually large proportion of such oils, are oils which usually contain considerable amounts of sulfur or sulfur-containing compounds, and may contain other corrosive constituents. When the oil is subjected to vaporization, in the manner above described, more or less of these sulfur compounds are vaporized and enter the cracking apparatus with the steam and hydrocarbon vapors. The presence of steam, which has already served the purpose of promoting the vaporization of the oil, serves the added purpose of protecting high temperature metal and alloy tubes, through which the sulfur-containing steam and oil vapors pass, in the crack-
50 ing zone.

The present process is an advantageous process for the utilization of sulfur-containing crude oil, residues, etc., which are cheap and readily available, which are advantageous materials for use in producing a high yield of olefins by the present process, and the objectionable sulfur content of which is taken care of by the presence of the steam, making possible the use of high temperature alloy tubes in the cracking furnace which could not otherwise be used if the steam was not present in admixture with the oil vapors.

From the preheater, the admixed steam and oil vapors, free from entrained particles of liquid oil, pass to the cracking tubes or cracking coils which may be heated in any suitable manner to the necessary high temperature for accomplishing the desired cracking operation, and the production of a high yield of gases rich in olefins. The cracking tubes can thus be heated by furnace gases or by electric heating or otherwise to the necessary high temperature. The cracking conditions maintained are those which will give a high production of gaseous unsaturated hydrocarbons. The velocities of gas and vapor flow
75

through the cracking tubes, and the tube temperatures in the furnace, are regulated so as to obtain a high conversion of the heavier hydrocarbon oil into olefin hydrocarbons, that is, into ethylene and propylene, etc.

For obtaining the maximum production of unsaturated hydrocarbons the temperature in the cracking tubes should, as previously pointed out, be above 1100° F., and preferably between about 1300° F. and 1900° F. Good results may be obtained at still higher temperatures, provided the mixed steam and oil vapors are passed through the tube at a sufficiently high velocity. When carrying out the process with metal cracking tubes, the temperature of the tube should be lower than that at which too rapid a deterioration of the tube material takes place.

With an East Texas crude oil a temperature of about 1400° F. to 1600° F. was advantageously used with a velocity of the oil vapors through the tubes corresponding to a supply of around 7 to 10 gallons of oil, as liquid, per hour, per cubic foot of tube space, plus 3 gallons of water per hour supplied in the form of steam admixed with the oil vapors. There may be a considerable variation in the ratio of steam to oil vapors. With East Texas crude oil the ratio of water to oil of 1 to 4 or of 1 to 2 by volume was found satisfactory.

In general, the ratio of steam to oil vapors may be varied from 0.1 of steam to 1 of oil vapors to as much as 2 of steam to 1 of oil vapors by weight relative to the weight of the oil.

The apparatus illustrated provides for admixing additional steam, if desired, with the steam and oil vapors after they leave the vaporizer 6 and before they enter the cracking tubes, although such addition is not ordinarily necessary or desirable where the amount of steam from the vaporizer is sufficient. This added provision is the branch pipe 26 having control valve 27 therein.

In the cracking tubes the pressures, temperatures and velocities can be somewhat varied but in general will be such as to provide for a high production of olefin hydrocarbon gases, using, in general, high temperatures such as those already described, pressures which need not be materially in excess of atmospheric, and high velocities such that there is a rapid heating of the admixed steam and oil vapors to the cracking temperature, and with a minimum of time of contact of the gases and vapors at the high temperature, the conditions being such as to favor the production of a high proportion of olefins in the gases which are then promptly withdrawn and cooled to prevent further cracking.

The admixed vapors and gases leaving the

the separation of olefins therefrom, as by selective extraction with solvents, or better by liquefaction and rectification operations.

The condensed oil recovered from the operation contains an appreciable fraction boiling within the gasoline range, or within the boiling point range of benzene, toluene and xylene. Such light fractions may be advantageously removed and the residue may be returned for further use as raw material for the process. A fraction has been obtained boiling in the benzene-toluene range, up to about 240° F., representing about 6 gallons of the fraction obtained from about 100 gallons of topped crude charged to the apparatus.

The gaseous product produced by the process, after purification and separation of the desired olefins, may be used as a fuel or may be in part returned as a diluent gas for admixture with steam in the further carrying out of the process. The recovered oil, condensed from the escaping gases and vapors, after the removal of any more valuable constituents such as the benzene-toluene fraction, or a gasoline fraction, may likewise be returned to the system for cracking.

It is a characteristic of the present process that it enables a gaseous product to be produced which is not only unusually high in olefins, such as ethylene, etc., but which is also relatively small in content of hydrogen produced.

The results obtained by the present cracking process may be contrasted with the results obtained by one of the well known vapor phase cracking processes for producing gasoline by comparing the volume of gas produced by the respective processes. One barrel of East Texas oil, by such vapor phase cracking process, would yield a maximum of 800 to 1000 cubic feet of gas; whereas the present process yields from 2500 to 3000 cubic feet or more of gas with an unusually high content of olefin hydrocarbons.

The beneficial results obtained by treating oils according to the present process are illustrated by the following example.

Example.—This example illustrates the application of the present process to the production of olefins in high yields from "topped" crudes and still residues, and illustrates the extent to which crude oil, or crude oil from which various percentages of lighter oils have been removed, may to advantage be utilized. In the carrying out of the process the crude oils, or the topped oils or residue, were subjected to vaporization with steam in the countercurrent vaporizer previously described and the resulting admixed vapors and steam were passed through a cracking apparatus made of heat resisting alloy known under the trade name KA2, electrically heated.

Oil used	Darst Creek crude 3a	Darst Creek crude topped oils				East Texas still residue	
		10% off 3b	20% off 3c	30% off 3d	30% off 3e	3f	3g
Tube temp., °F. (external).....	1500	1500	1525	1490	1490	1500	1500
Gal. per hr. oil in per cu. ft. of tube space.....	8.5	12.2	11.5	11.3	10.9	9.2	10.6
Vaporizer temp., °F.....	671	608	622	622	644	662	650
Gal. H ₂ O: gal. oil.....	1:1.5	1:2.0	1:1.9	1:1.8	1:1.8	1:3.0	2:3.2
Percent oil vaporized.....	93.3	84.7	82.0	78.5	84.7	59.8	76.7
Cu. ft. exit gas per gal. oil in.....	66.5	60.2	57.4	55.2	61.2	42.5	52.1
Percent C ₂ H ₄	32.5	31.4	32.4	31.9	31.7	32.5	34.2
Percent higher olefins.....	23.9	25.9	24.5	25.9	24.9	18.0	24.5

cracking tubes will pass to a condenser or cooler where condensable oils will be condensed, together with excess of water. The remaining gases, after removal of condensable oil and excess water, are rich in olefins, and may be treated for

From this tabulation it will be noted that high percentages of the crude oil, or of the topped crudes, and also of the residue, were vaporized in the countercurrent steam vaporizer hereinbefore described, leaving only a small percentage of

the crude oil or residue to be removed in liquid form from the vaporizer. With continuous and constant feed of the crude oil or residue to the vaporizer, and continuous operation of the process, vapors of substantially constant composition, free or relatively free from asphaltic or other non-volatile material, were supplied to the cracking apparatus, in admixture with steam, and the cracking operation was carried out so that the apparatus remained free from carbon, even though the gas produced amounted to around 2800 cubic feet per barrel of oil fed to the vaporizer, and the gas obtained was high in olefins.

Insofar as I am aware, it has not heretofore been possible to utilize crude oil or residues to any such extent or with any such advantages as are obtained with the present process.

The data given under 3b, 3c, 3d and 3e illustrates the results obtained with the same Darst Creek crude from which increased amounts of the lighter constituents were removed prior to utilization according to the present process.

A comparison of the results indicated in 3g with those of 3f shows the effect of increasing the proportion of steam to oil while other conditions are held approximately constant. At a vaporizer temperature of 662° F. approximately 60% of the residue can be vaporized with one gallon of water to three gallons of oil; while by approximately doubling the amount of steam used, the percentage of oil vaporized was increased to about 76.7%, even though the vaporizer temperature was somewhat lower; and an increased yield of total gas produced, and also of olefins, was obtained.

The residue referred to in 3g was a heavy oil, highly viscous at ordinary temperatures, and having an initial boiling point of 550° F.

A comparison of the results indicated under 3g with those under 3f shows that, under 3g, where a much greater amount of the heavier constituents was vaporized than was the case under 3f, around 2200 cubic feet of gas was obtainable per barrel of oil supplied to the vaporizer, an increase of about 23% in volume, while the percentage of olefin gases increased from around 50.5% to around 58.7%, and this being accomplished without the formation of any appreciable amount of carbon either in the vaporizer or in the cracking zone. Such a still residue as that utilized in connection with 3f and 3g has little or no value commercially except as fuel; but it can be advantageously employed in the production of olefin hydrocarbons by the process of the present invention.

In the practice of the present process the cracking tubes can be made of various high temperature alloys, such as nichrome, the chromium-nickel alloy known as KA2, etc., and the presence of sulfur in the crude oil or residue utilized does

not have the injurious effect on such alloys, even at the high temperatures employed, as would be obtained in the absence of steam at such temperatures. The utilization of steam in the process therefore makes it possible to use alloys which are desirable for high temperature work, but which could not otherwise be used at the high temperatures of the present process were it not for the presence of steam admixed with the sulfur-containing oil vapors entering the cracking tubes.

It will thus be seen that the present invention provides an improved method for the cracking of sulfur-containing oil, particularly for the production of high yields of olefins.

I claim:

1. The method of cracking sulfur-containing hydrocarbons for the production of a high yield of gases rich in unsaturated hydrocarbons, which comprises passing admixed hydrocarbon vapors and steam through chromium-containing alloy tubes and heating the same therein by heat supplied to the tubes to a high cracking temperature, of 1400 to 1600° F., the tubes being of alloys readily attacked by sulfur compounds in the absence of steam at such high temperatures, and the steam being present in amount sufficient to protect the tubes from such attack, whereby the cracking operation can be continued in such tubes at such high temperatures for prolonged periods of time.

2. The method of cracking sulfur-containing hydrocarbons for the production of a high yield of gases rich in unsaturated hydrocarbons, which comprises passing admixed hydrocarbon vapors and steam through nickel-containing alloy tubes and heating the same therein by heat supplied to the tubes to a high cracking temperature of 1100 to 1900° F., the tubes being of alloys readily attacked by sulfur-compounds in the absence of steam at such high temperatures, and the steam being present in amount sufficient to protect the tubes from such attack, whereby the cracking operation can be continued in such tubes at such high temperatures for prolonged periods of time.

3. The method of cracking sulfur-containing hydrocarbons for the production of a high yield of gases rich in unsaturated hydrocarbons, which comprises passing admixed hydrocarbon vapors and steam through chromium-nickel-containing alloy tubes and heating the same therein by heat supplied to the tubes to a high temperature, of 1400 to 1600° F., the tubes being of alloys readily attacked by sulfur compounds in the absence of steam at such high temperatures, and the steam being present in amount sufficient to protect the tubes from such attack, whereby the cracking operation can be continued in such tubes at such high temperatures for prolonged periods of time.

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