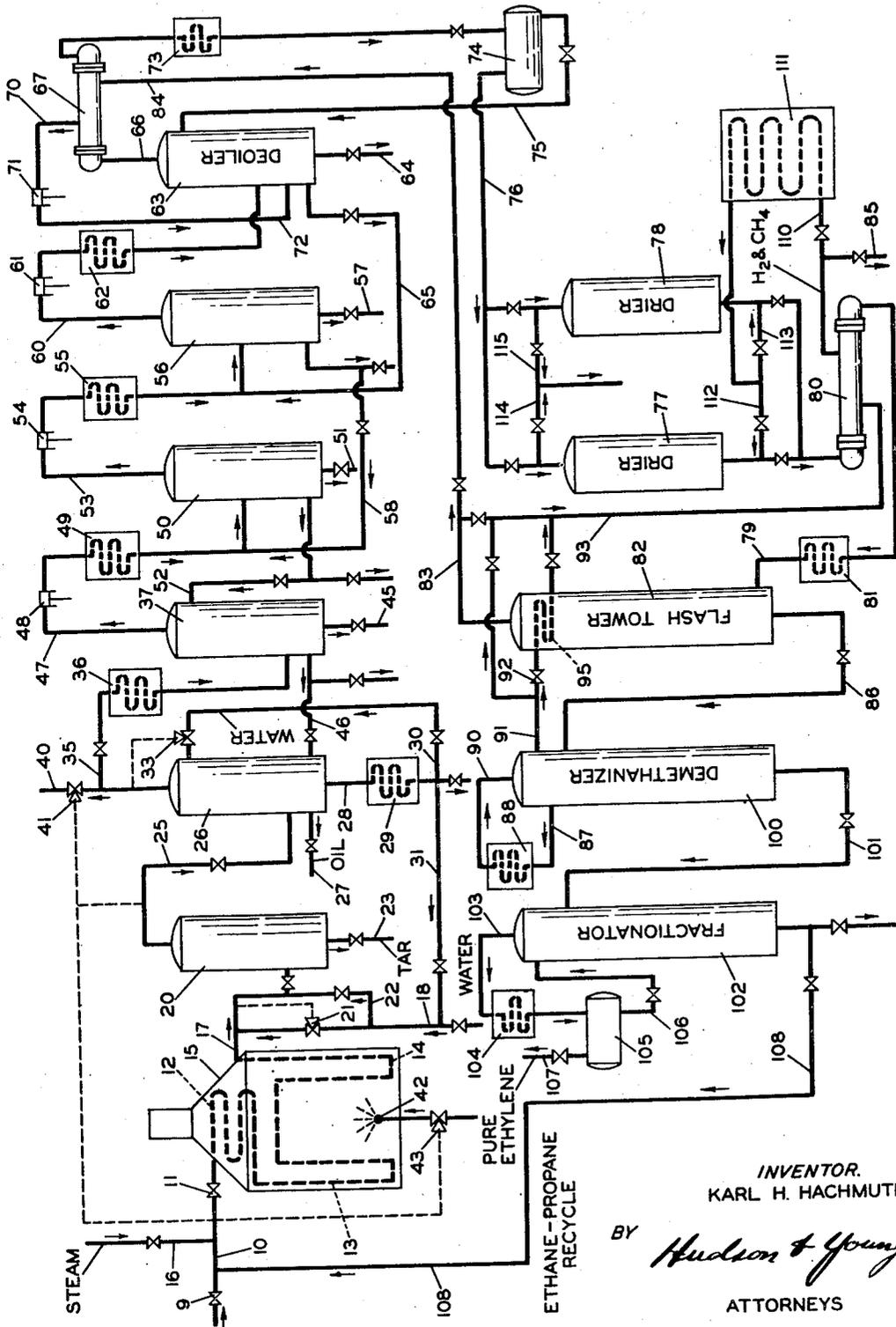


Feb. 28, 1950

K. H. HACHMUTH
PRODUCTION OF ETHYLENE

2,498,806

Filed July 16, 1946



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2,498,806

PRODUCTION OF ETHYLENE

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Application July 16, 1946, Serial No. 683,904

4 Claims. (Cl. 62—175.5)

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This invention relates to the production of ethylene. In one embodiment it relates to the conversion of low-boiling paraffin hydrocarbons or of low-boiling olefin hydrocarbons, other than ethylene, to ethylene and the recovery of the resulting ethylene in a highly concentrated form. Another embodiment of the invention relates to the production of ethylene in a concentration of about 95% or more from a mixture of ethylene with methane, hydrogen, ethane, and higher-boiling hydrocarbons.

Ethylene is a valuable hydrocarbon material which can be readily produced from other hydrocarbons by catalytic or by noncatalytic conversion. However, because it is difficult to liquefy ethylene and separate it by distillation, and because ethylene is not very reactive chemically under readily obtained conditions, the recovery of ethylene in a relatively pure form has been one of the major obstacles preventing its use on a large scale. By the combination of steps hereinafter more fully disclosed and discussed, I have found that ethylene can be readily produced in a relatively pure state.

It is an object of this invention to produce ethylene in a relatively pure state.

Another object of this invention is to convert other hydrocarbons to ethylene.

Still another object of this invention is to separate ethylene from other normally gaseous materials which have boiling points both above and below the boiling point of ethylene.

Further objects and advantages of my invention will become apparent, to one skilled in the art, from the accompanying disclosure and discussion.

A preferred embodiment of my invention, together with various preferred subcombinations thereof, will now be discussed in connection with the accompanying drawing, which is a diagrammatic flow sheet illustrating schematically various pieces of equipment which can be used in the practice of my invention. Referring now to the drawing, a suitable hydrocarbon charge stock, such as a material comprising a major portion of propane together with some ethane, is introduced through line 10. This is composed of a net charge, introduced through valve 9, and of a recycle

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stream passed through line 108. It is often preferred that this material be substantially free from sulfur compounds, such as hydrogen sulfide and mercaptans, and when so desired a sulfur-containing charge may be treated to reduce its sulfur content. This charge stock, at a pressure not greater than about 40 pounds per square inch absolute, is passed through an expansion valve or orifice 11 to preheating coil 12 and cracking or dehydrogenating coils 13 and 14, which are situated in furnace 15. In coils 13 and 14 the charge is heated to a temperature of about 1350 to about 1650° F. for a time sufficient to give an optimum yield of ethylene. This will generally be about 1.5 to about 0.15 seconds. In actual plant operation, a furnace 15 will have a plurality of preheating and cracking coils operating in parallel and by means of an expansion valve or expansion orifice 11 at the inlet to each set of coils the flow through the sets of coils will be more or less uniform. To the hydrocarbon stream, prior to its introduction to the reaction zone, steam is added through line 16. Enough steam is used to passivate catalytic metal and depress coke formation. In some instances about 0.1 to about 0.5% by weight of sulfur will have a similar effect. However, when about 0.2 to 0.5 mol of steam per mol of hydrocarbon is used, the effect of the decreased partial pressure of hydrocarbon reactants is beneficial, and leads to optimum ethylene production and serves to minimize polymerizing of olefin produced. It is preferred that, in the cracking coils 13 and 14, tubes of a small diameter be used, receiving direct radiant heat and with the diameter of the tubes and the flow of reactants through the tubes so correlated that a lower than usual rate of heat transfer through the tube walls takes place. This results in having a smaller temperature differential between the walls and the reacting gases, thereby minimizing production of high-boiling hydrocarbons and coke.

Effluents of cracking coil 14 are passed through line 17 to tar separator 20. Immediately upon the exit of the gases from cracking coil 14, water is injected through line 18 to cool the gases as rapidly as possible, to a temperature below about 1000° F. Unless this is done, beneficial results from careful design of the cracking coils and control of flow rates with heat transfer rates to produce

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optimum ethylene production will be lost, because ethylene is highly reactive and readily polymerizes, and also undergoes hydrogenation, at the reaction temperatures. The amount of water introduced through line 18 is controlled by valve 21, which is responsive to the temperature in line 17 at a point which is down stream from the point of water injection. Additional quantities of water are added through line 22 to bring the temperature of the gases to about 250 to about 350° F. As a result, some of the heaviest tars produced during the cracking reaction are condensed and separate from the uncondensed gaseous material. Such tars can be discharged from the process through line 23.

Uncondensed gases pass from tar separator 20 through line 25 to quench tower 26, wherein they are intimately contacted with a spray of water introduced through line 30. The temperature at the top of tower 26 is maintained at about 120 to about 160° F., resulting liquid oils are discharged through line 27. Water at a temperature of about 165 to about 265° F. is removed through line 28, cooled in cooler and condenser 29, and recirculated to the process either through line 30 to the spray in the top of tower 26, or through line 31 to line 17. Recirculation of water in a completely closed cycle, as illustrated, has the advantage that the water can be readily maintained free from oxygen.

Uncondensed gases are removed from quench tower 26 through line 35, and passed through cooler 36 to a first separator 37. The temperature of these gases is maintained at between about 120 and 160° F., by control of valve 33 in water line 30 in response to the temperature of the discharged gases. In the event that something goes wrong with the injection of quench water through lines 18 and 22, and the temperature of the gases passing through line 25 rises to the neighborhood of about 1000° F., gases passing from the top of quench tower 26 can be quickly discharged from the system through line 40 by the opening of valve 41. At the same time, the fire in burner 42 is extinguished by control valve 43 in the fuel line. In this way the operation of the subsequent separation equipment is protected from excessive variations in the composition of the gases passing through line 35. This protection feature is more fully disclosed and claimed in copending application Serial No. 683,889, filed July 16, 1946 of Sam P. Robinson, issued April 6, 1948, as Patent No. 2,439,023.

In separator 37 condensed water is removed from line 45 and light oils are passed back to the water quench tower 26 through line 46. The gases in this first separator are at a temperature of about 75 to 130° F. and under a pressure which is slightly above atmospheric pressure, but not greater than about 30 pounds per square inch absolute. Gases are removed through line 47, compressed by compressor 48 to a pressure of about 45 to 75 pounds per square inch absolute, and then passed through cooler and condenser 49 to a second separator 50. These compressed gases are cooled in cooler 49 with ordinary cooling water to a temperature between about 75 and 130° F. Resulting condensed water is discharged through line 51 and resulting light oils are passed through line 52 to the top of separator 37. Uncondensed gases are removed through line 53, compressed by compressor 54 to a pressure between about 165 and 200 pounds per square inch absolute, again cooled with cooling water in cooler

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55 and passed to a third separator 56. Condensed water is removed through line 57 and condensed light oils are passed through line 58 and admixed with the material entering separator 50. Uncondensed gases are removed through line 60, compressed in compressor 61 to a pressure of about 400 to about 625 pounds per square inch absolute, cooled with ordinary cooling water in cooler 62 to a temperature in the range of about 75 to about 130° F., and passed to a deoiler 63.

Separators 37, 50, and 56 are generally relatively small vertical cylinders, equipped with not more than a mist separator in the top to effect a separation between liquids and gases. On the other hand, deoiler 63 is a small fractional distillation column with about 15 to about 30 bubble trays. This effects a final removal of hydrocarbons having 4 or more carbon atoms per molecule and of most of the water content of the mixture being treated. Water can be discharged through line 64 and light oils are passed through line 65 to be admixed with material introduced into separator 56. Deoiler 63 is operated with a kettle temperature in the range of about 260 to about 310° F. and a top temperature in the range of about 64 to about 107° F.

Uncondensed gases from deoiler 63 are removed through line 66, partially cooled in heat exchanger 67 by indirect heat exchange with a cold stream of hydrogen gas from flash tower 82 passing through line 84, the resulting warm hydrogen gas passing from heat exchanger 67 through line 70, compressor 71 and line 72 to a low point of deoiler 63. Preferably this hydrogen gas is introduced beneath the surface of the liquid in the reboiler portion of deoiler 63. Such an introduction of this gas serves to strip ethylene from the liquids being removed from deoiler 63. The partially cooled gases passing through heat exchanger 67 are further cooled and partially condensed in cooler 73 and the resulting gas-liquid mixture is introduced into reflux accumulator 74. Cooler 73 cools the gases to a temperature of about 20 to about 56° F. by means of a propane refrigerant. A resulting liquid condensate is passed through line 75 as a liquid reflux to the top of deoiler 63. Uncondensed gases are passed through line 76 to one or the other of driers 77 and 78. These driers contain a mass of granular adsorbent desiccant such as activated charcoal, activated alumina, activated silica gel, activated bauxite, or the like. As soon as one of the driers has become spent, as a result of accumulation of adsorbed water, a regenerated drier is switched into use and the water is removed from the spent mass of desiccant by contact with a heated dry gas, as will be more fully discussed hereinafter. In some instances it may be found to be more desirable to dry the gases passing through line 66 to heat exchanger 67, instead of drying the gases further downstream as shown. The dried gases, under a pressure between about 550 and 650 pounds per square inch absolute are passed through heat exchanger 80, cooler and condenser 81, and line 79 to flash tower 82. In heat exchanger 80 the gases are cooled by indirect heat exchange with cold hydrogen and methane, from flash tower 82 and demethanizer 100. In cooler 81, this material is further cooled to a temperature in the range of about -140 to about -100° F. to effect substantially complete condensation of the ethylene and heavier hydrocarbons and a condensation of the major portion of the methane. Usually this is accomplished by a plurality of coolers. Thus,

satisfactory operation has been obtained by use of a first cooler with liquid propane as a refrigerant, followed by a second cooler using liquid ethane or liquid ethylene as a refrigerant.

In flash tower 82, which is equipped with a few standard bubble trays, most of the hydrogen and some of the methane is separated as a gas from a liquid comprising the remaining portion of the methane, and the ethylene and heavier hydrocarbons. The top of flash tower 82 is maintained at a temperature in the range of about -170 to about -140° F., by means of cold expanded gaseous methane passing through cooling coils 95. Cold gaseous hydrogen is removed through line 83. A minor portion thereof, about 10 to 20 per cent, is passed through line 84 to heat exchanger 67 for use as a stripping gas in deoiler 63, as previously discussed. The remainder of the hydrogen is used as a cooling agent in heat exchanger 80, and can then be discharged from the process through line 85.

Liquid material, at a temperature of about -140 to about -100° F., is removed from the bottom of flash tower 82 through line 86, and passed to a high point of demethanizer 100. This is a conventional fractionating column, provided with a substantial number of bubble trays. It is operated at a pressure between about 500 and about 600 pounds per square inch absolute. Gases are removed from the top of demethanizer 100, at a temperature of about -140 to about -110° F., through line 87 and are cooled and substantially condensed in condenser 88 by indirect heat exchange with vaporizing liquid ethane or ethylene. A liquid reflux is returned through line 90 to maintain the desired low temperature in the top of demethanizer 100. Gaseous methane is removed through line 91 and a substantial portion is expanded through expansion valve 92 to a pressure between about 50 and about 100 pounds per square inch absolute, with a resulting temperature between about -235 and about -185° F. This cold expanded gaseous methane is then passed through cooling coils 95 to furnish the necessary refrigeration to maintain the desired reflux in flash tower 82. The gaseous methane is then passed through line 93 and mixed with the gaseous hydrogen stream immediately before it is introduced into heat exchanger 80. The material discharged through line 85, therefore, is a mixture of hydrogen and methane.

Liquid substantially free from methane and hydrogen and comprising ethylene and heavier hydrocarbons is passed from the bottom of demethanizer 100, at a temperature of about 40 to about 55° F., through line 101 to fractionator 102. Fractionator 102 is also a conventional fractional distillation column operated under a pressure between about 325 and about 450 pounds per square inch absolute. Vapors are removed through line 103, passed through cooler and condenser 104, and the resulting mixture of condensate and uncondensed ethylene is passed to reflux accumulator 105. The liquid condensate is returned through line 106 to the top of fractionator 102, as a liquid reflux, to maintain a temperature between about -10 and +12° F. Substantially pure ethylene is removed from accumulator 105, as a gaseous product of the process, through line 107. A liquid mixture of ethane and C₃ hydrocarbons is removed from the bottom of fractionator 102 through line 108 and passed to line 10 to be reintroduced into the cracking coils. If desired, cooling and reflux for demethanizer 100

can be obtained as shown for ethylene fractionator 102, or vice versa.

As will be appreciated, the gaseous hydrogen and methane passing through line 85 is exceptionally dry. It therefore serves as an excellent drying gas to remove water from the spent mass of desiccant in drier 77 or drier 78. As is necessary, a portion of this gaseous material is removed from line 85 through line 110, heated to a temperature of about 400 to 450° F. in heater 111 and passed through one of pipes 112 or 113 to whichever of driers 77 and 78 contains a spent mass of desiccant. A resulting water-laden gas is removed through line 114 or line 115 and discharged from the process. This operation can be conveniently carried out under a pressure of about 75 to about 100 pounds per square inch. After the water has been removed, supply of heat to heater 111 is discontinued and a stream of unheated cold gas is then passed through the desiccant mass to cool the mass back to a temperature of about 35 to about 60° F.

As will be appreciated by one skilled in the art, the drawing is entirely diagrammatical, and in actual operation various additional pieces of conventional equipment, such as pumps, control valves, temperature indicating, recording, and control devices, heaters for reboilers, coolers, heat exchangers, insulation for transfer lines and fractional distillation columns, accumulators and the like, containing materials at subatmospheric temperatures, will obviously be included and can be readily incorporated in any specific case. The temperatures used in the various steps will be closely dependent upon the pressures used, and both will be somewhat dependent upon the compositions of the materials being treated.

My invention will be further illustrated by the following example. In connection with this example, reference is made back to various pieces of equipment illustrated in the drawing. A gaseous C₂-C₃ hydrocarbon mixture is introduced through valve 9, having a composition shown in the accompanying table. To this is added a recycle stream from line 108, also shown in the accompanying table, and steam to give a total furnace feed passing through expansion orifice 11 as shown in the table. This mixture is subjected to cracking at a maximum temperature of about 1470° F. under an exit pressure of about 20 pounds per square inch absolute, to give an effluent through line 17 having the composition shown in the table, prior to the introduction of quench water. This material is treated in deoiler 63 at a pressure of about 600 pounds per square inch absolute, and a temperature in reflux accumulator 74 of 50° F. The gaseous ethylene-containing stream is passed through line 76, dried in dryer 77 and has a composition as shown in the table, as it is passed through line 79 to flash tower 82. The hydrogen discharged through line 83, has the composition shown, when flash tower 82 is operated with a reflux temperature of -160° F. The demethanizer 100 is operated at a pressure of about 550 pounds per square inch absolute and a reflux temperature of -122° F. The methane fraction discharged through line 91 has the composition shown in the table. Ethylene fractionator 102 is operated at a pressure of about 400 pounds per square inch absolute and a reflux temperature of 0° F. The resulting gaseous ethylene stream, removed as a product of the process through line 107 is substantially pure, having the composition shown in the table. Compositions in the table are in mol per cent.

	Valve 9 Net Furnace Feed	Orif. 11 Total Furnace Feed	Line 17 Furnace Effluent	Line 60 Charge to Deoiler	Line 79 Effluent From Drier	Line 83 Flash Tower Overhead	Line 91 Demeth- anizer Overhead	Line 107 Ethylene Product	Line 108 C ₂ -C ₃ Recycle
H ₂			16.0	19.2	22.4	61.8	1.7		
CH ₄	5.7	4.3	26.9	32.3	33.6	30.9	95.3	2.1	
C ₂ H ₆			0.1	0.1	0.1			0.4	
C ₃ H ₈	5.5	4.1	24.8	29.7	29.4	8.4	2.6	95.0	4.7
C ₄ H ₁₀	14.4	10.8	5.0	6.0	5.9	0.9	0.4	2.5	34.1
C ₅ H ₁₂	29.3	21.9	5.4	6.3	5.8				41.4
C ₆ H ₁₄	44.1	33.1	1.1	1.3	1.2				8.5
C ₇ H ₁₆			0.1	0.1	0.1				0.6
C ₈ H ₁₈			1.5	1.7	1.2				8.7
C ₉ H ₂₀	1.0	0.8	0.2	0.3	0.2				1.4
Oils			2.1	2.4	0.1				0.7
Tars			0.3						
H ₂ O		25.0	16.5	0.6					
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

It will be appreciated that various modifications of my invention can be practiced, by one skilled in the art, without departing from the scope or spirit of the disclosure or claims.

I claim:

1. An improved process for the recovery of substantially pure ethylene from a mixture comprising free hydrogen, ethylene, propane and lighter paraffins, together with not more than minor amounts of heavier hydrocarbons, which comprises passing such a mixture to a separation zone, removing from said separation zone as a liquid substantially all hydrocarbons heavier than propane, introducing into said zone gaseous hydrogen from a first fractional distillation step as hereinafter recited to aid in stripping light material from said heavier liquid, removing as a gas from said separation zone propane and lighter material, partially cooling same by indirect heat-exchange with said hydrogen prior to introduction of said hydrogen into said zone, further cooling and partially condensing said mixture, passing a resulting condensate back to said separation zone as a reflux liquid, drying resulting uncondensed gases, cooling said dried uncondensed gases and condensing the major portion of the methane and substantially all the heavier hydrocarbons, passing said cooled and partially condensed mixture to a first fractional distillation zone, removing from said first zone a gaseous fraction comprising substantially all the hydrogen and a minor portion of the methane contained in said partially condensed mixture, passing a portion of said gaseous fraction into indirect heat-exchange with the first said gaseous propane and lighter material and subsequently into said separation zone as aforesaid, removing from said first zone a liquid fraction comprising methane and heavier hydrocarbons and passing same to a second fractional distillation zone, removing from said second zone a gaseous fraction comprising substantially methane, expanding at least a portion of said gaseous methane to a colder gas at a lower pressure, passing said expanded colder gas in indirect heat-exchange with vaporous material in the top of said first distillation zone, removing from said second zone a liquid fraction substantially free from methane and comprising ethylene and heavier hydrocarbons and passing same to a third fractional distillation zone, removing from said third distillation zone as a liquid fraction a substantially ethylene-free hydrocarbon material comprising ethane and heavier hydrocarbons, and removing also from said third zone as a product of the process a gaseous fraction comprising substantially pure ethylene.

2. An improved process for the recovery of substantially pure ethylene from a mixture compris-

ing free hydrogen, ethylene, propane and lighter paraffins together with not more than minor amounts of heavier hydrocarbons, which comprises passing such a mixture at a pressure in the range of about 400 to about 625 pounds per square inch absolute and a temperature in the range of about 75 to about 130° F. to a separation zone, removing from said zone as a liquid at a temperature in the range of about 260 to about 310° F. substantially all hydrocarbons heavier than propane, introducing into said zone a gaseous hydrogen fraction from a subsequent separation step as hereinafter recited to aid in stripping ethylene from said heavier liquid, removing as a gas from said separation zone propane and lighter material, partially cooling same by indirect heat-exchange with said hydrogen prior to its introduction into said zone, further cooling said mixture without substantial reduction in pressure to a temperature in the range of about 20 to about 56° F. thereby affecting a partial condensation, passing a resulting condensate back to said separation zone as a reflux liquid in an amount such as to maintain a temperature in the range of about 64 to about 107° F. at the point where said gaseous effluent is withdrawn, drying resulting uncondensed gases, cooling said dried uncondensed gases while under a pressure in the range of about 550 to about 600 pounds per square inch absolute to a temperature in the range of about -140 to about -100° F. thereby condensing the major portion of the methane and essentially all the heavier hydrocarbons, passing said cooled and partially condensed mixture to a first fractional distillation zone at said pressure, removing from said first zone at a temperature in the range of about -170 to about -140° F. a gaseous fraction comprising substantially all the hydrogen and a minor portion of the methane contained in said partially condensed mixture, passing a portion in the range of about 10% to about 20% of said gaseous fraction into indirect heat exchange with the first said gaseous propane and lighter material and subsequently into said separation zone as hereinbefore recited, removing from said first zone at a temperature in the range of about -140 to about -100° F. a liquid fraction comprising methane and heavier hydrocarbons and passing same to a second fractional distillation zone at a pressure in the range of about 500 to about 600 pounds per square inch, removing from said second zone at a temperature in the range of about -140 to about -110° F. a gaseous fraction comprising substantially methane, expanding said gaseous fraction to a pressure such that the temperature is in the range of about -235 to about -185° F., passing at least a sufficient portion of said expanded colder gas in indirect heat-exchange with vaporous material in the top of said

first distillation zone to maintain the aforesaid temperature in the range of about -170 to about -140° F., removing from said second zone at a temperature in the range of about 40 to about 55° F. a liquid fraction comprising ethylene and heavier hydrocarbons and substantially free from methane and passing same to a third fractional distillation zone at a pressure in the range of about 325 to about 450 pounds per square inch, and removing overhead from said third zone as a product of the process at a temperature in the range of about -10 to about +12° F. a gaseous fraction comprising substantially pure ethylene.

3. An improved process for the recovery of substantially pure ethylene from a mixture comprising free hydrogen, ethylene, propane and lighter paraffins together with not more than minor amounts of heavier hydrocarbons, which comprises passing such a mixture at a pressure of about 600 pounds per square inch absolute and a temperature of about 110° F. to a separation zone, removing from said zone as a liquid at a temperature of about 300° F. substantially all hydrocarbons heavier than propane, introducing into said zone a gaseous hydrogen fraction from a subsequent separation step as hereinafter recited to aid in stripping ethylene from said heavier liquid, removing as a gas from said separation zone propane and lighter material, partially cooling same by indirect heat-exchange with said hydrogen prior to its introduction into said zone, further cooling said mixture without substantial reduction in pressure to a temperature of about 50° F. thereby effecting a partial condensation, passing a resulting condensate back to said separation zone as a reflux liquid in an amount such as to maintain a temperature of about 100° F. at the point where said gaseous effluent is withdrawn, drying resulting uncondensed gases, cooling said dried uncondensed gases while under a pressure of about 575 pounds per square inch absolute to a temperature of about -125° F. thereby condensing the major portion of the methane and essentially all the heavier hydrocarbons, passing said cooled and partially condensed mixture to a first fractional distillation zone at said pressure, removing from said first zone at a temperature of about -160° F. a gaseous fraction comprising substantially all of the hydrogen and a minor portion of the methane contained in said partially condensed mixture, passing about 16% of said gaseous fraction into indirect heat exchange with the first said gaseous propane

and lighter material and subsequently into said separation zone as hereinbefore recited, removing from said first zone at a temperature of about -125° F. a liquid fraction comprising methane and heavier hydrocarbons and passing same to a second fractional distillation zone at a pressure of about 550 pounds per square inch, removing from said second zone at a temperature of about -122° F. a gaseous fraction comprising substantially methane, expanding said gaseous fraction to a pressure such that the temperature is about -215° F., passing at least a sufficient portion of said expanded colder gas in indirect heat-exchange with vaporous material in the top of said first distillation zone to maintain the aforesaid temperature of about -160° F., removing from said second zone at a temperature of about 46° F. a liquid fraction comprising ethylene and heavier hydrocarbons and substantially free from methane and passing same to a third fractional distillation zone at a pressure of about 400 pounds per square inch, and removing overhead from said third zone as a product of the process at a temperature of about 0° F. a gaseous fraction comprising substantially pure ethylene.

4. The process of claim 1 in which said drying of uncondensed gases is effected by contact under desiccating conditions with alternated masses of solid adsorbent desiccants and in which adsorbed water is removed from a spent mass of desiccant by contact with a heated stream of admixed gaseous hydrogen and methane fractions from said desiccating conditions with alternated masses of first and second fractional distillation zones.

KARL H. HACHMUTH.

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The following references are of record in the file of this patent:

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Certificate of Correction

Patent No. 2,498,806

February 28, 1950

KARL H. HACHMUTH

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows:

Column 8, line 28, after the word "separation" strike out "step"; column 10, line 33, strike out "desiccating conditions with alternated masses of";

and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 29th day of August, A. D. 1950.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.