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Lofredo et al.

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[54] **PROCESS AND SYSTEM FOR THE PRODUCTION AND PURIFICATION OF HELIUM**

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[52] **U.S. Cl.** **62/22,** 62/14, 62/39, 62/18, 62/24

[51] **Int. Cl.** **F25j 3/00,** F25j 3/06, F25j 5/00

[58] **Field of Search** ..... 23/1 E, 4; 62/9, 11, 22, 62/23, 24, 27, 28, 39, 51, 14, 18; 423/659

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[57] **ABSTRACT**

An overall process comprising a series of three successive operations for deriving helium from natural gas and purifying it to 99.9969 mol percent, minimum. In the initial operation, the natural gas feed, containing 15 to 40 mol percent of helium, is cooled and flashed in two stages. Both the crude helium product and the vaporized condensate from the flash stages serve to refrigerate the feed stream. The condensate by-product provides commercial grade fuel gas. In the intermediate operation, an input stream having a helium content from 60 to 80 mol percent is enriched to a purity of 85 to 97 mol percent by cooling it to below liquid nitrogen temperature, and passing it through a helium separator. The effluent helium-rich vapor is optionally reheated, then work expanded, and used as a refrigerant for the feed stream. Dissolved helium is removed from the condensate in the helium separator by auxiliary flashing, and returned to the recycle stream, the condensate from the latter stage ultimately serving as a refrigerant. In the final operation, the enriched helium undergoes purification by catalytic action to remove hydrogen. It is then dried, compressed, cooled, passed through several stages of flashing and additional cooling, and through carbon adsorbers to remove the balance of nitrogen. The cold, nearly pure product is finally heat exchanged with a feed stream prior to storage. Condensate from the flashing stages, and liquid from an auxiliary nitrogen system provide refrigeration for the final purification operation.

44 Claims, 12 Drawing Figures

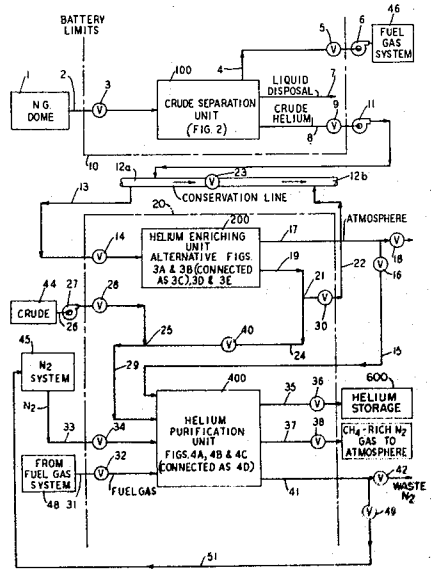
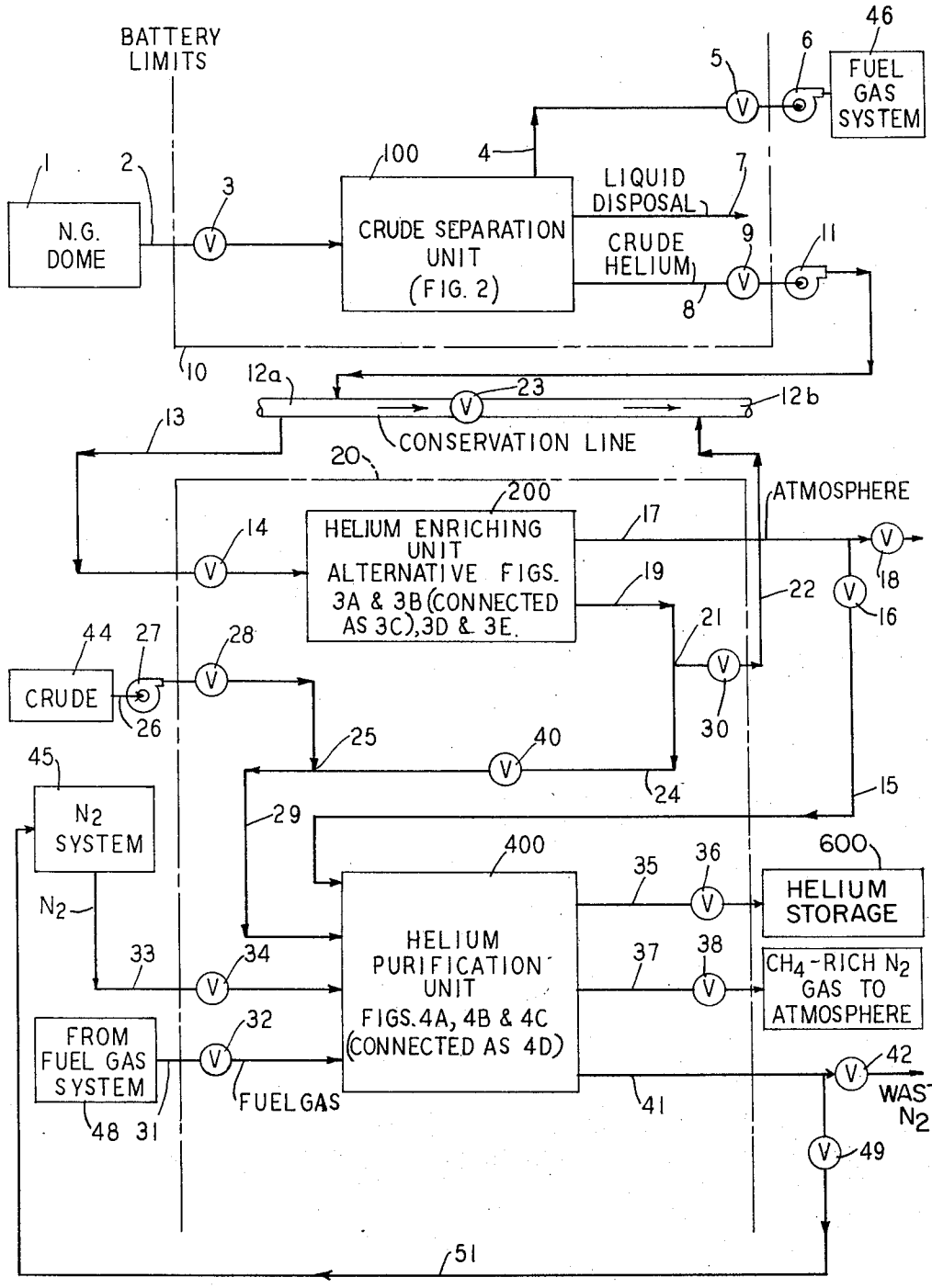


FIG. 1



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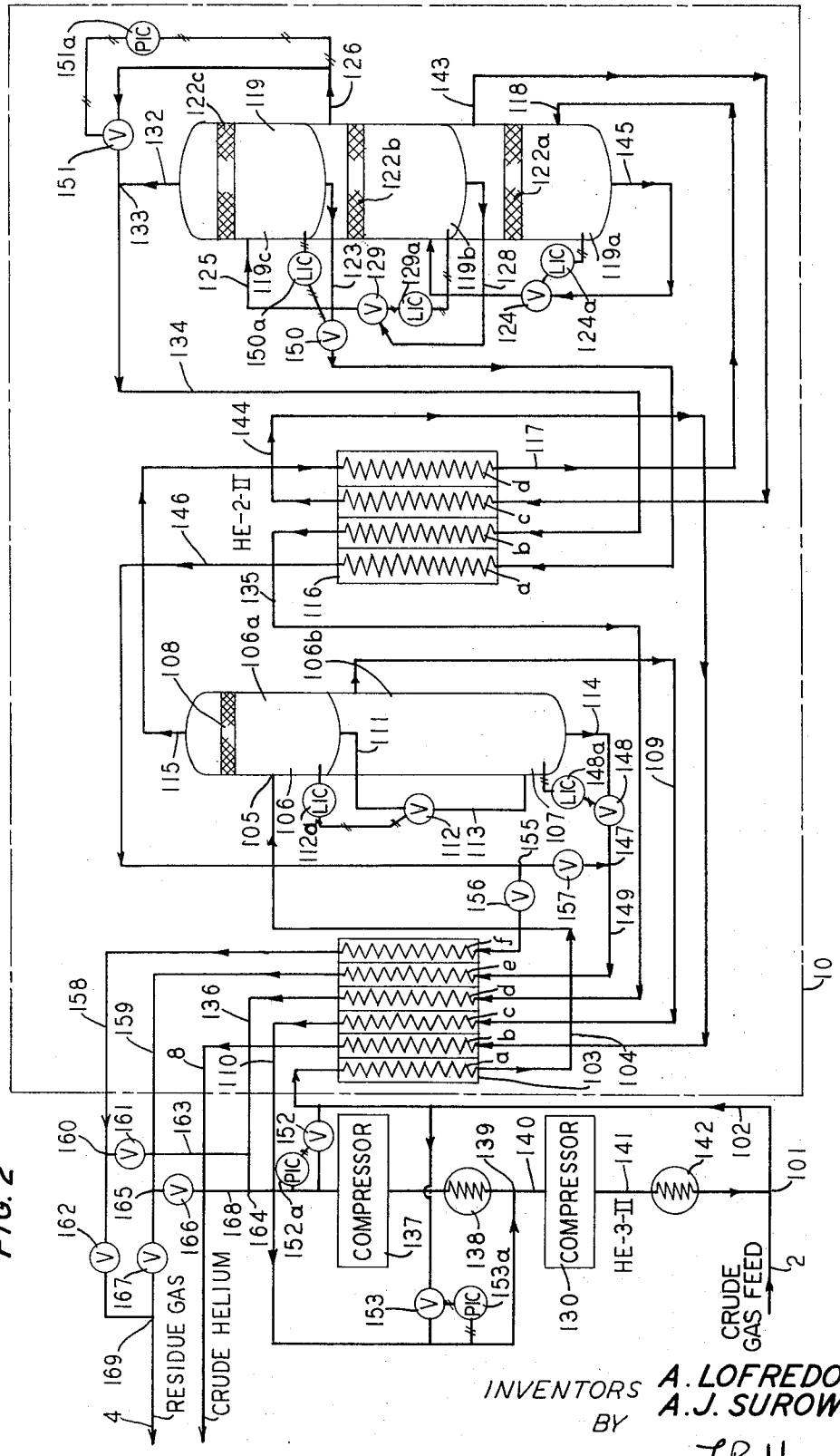


FIG. 2

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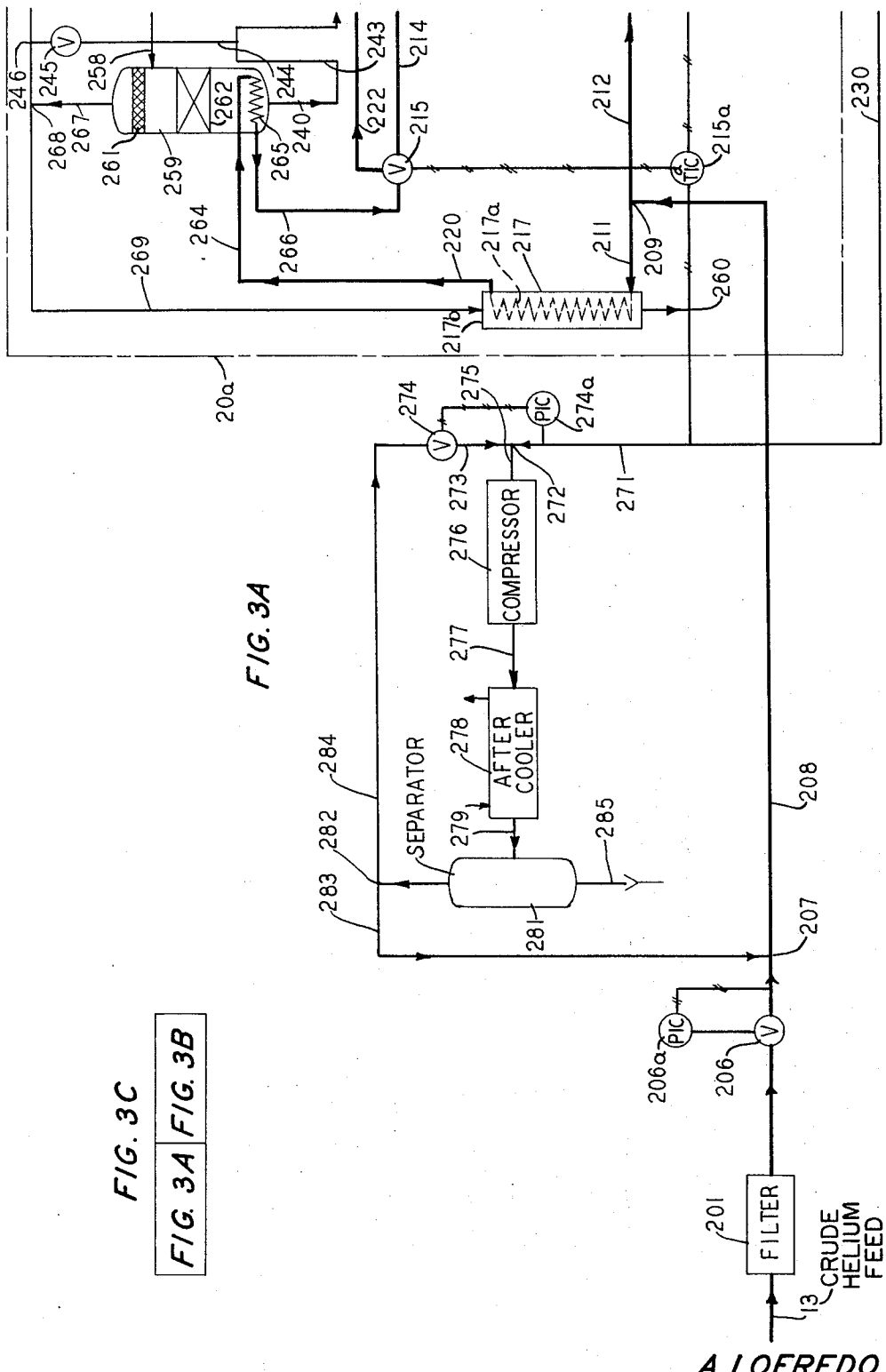


FIG. 3A

FIG. 3C

FIG. 3A	FIG. 3B

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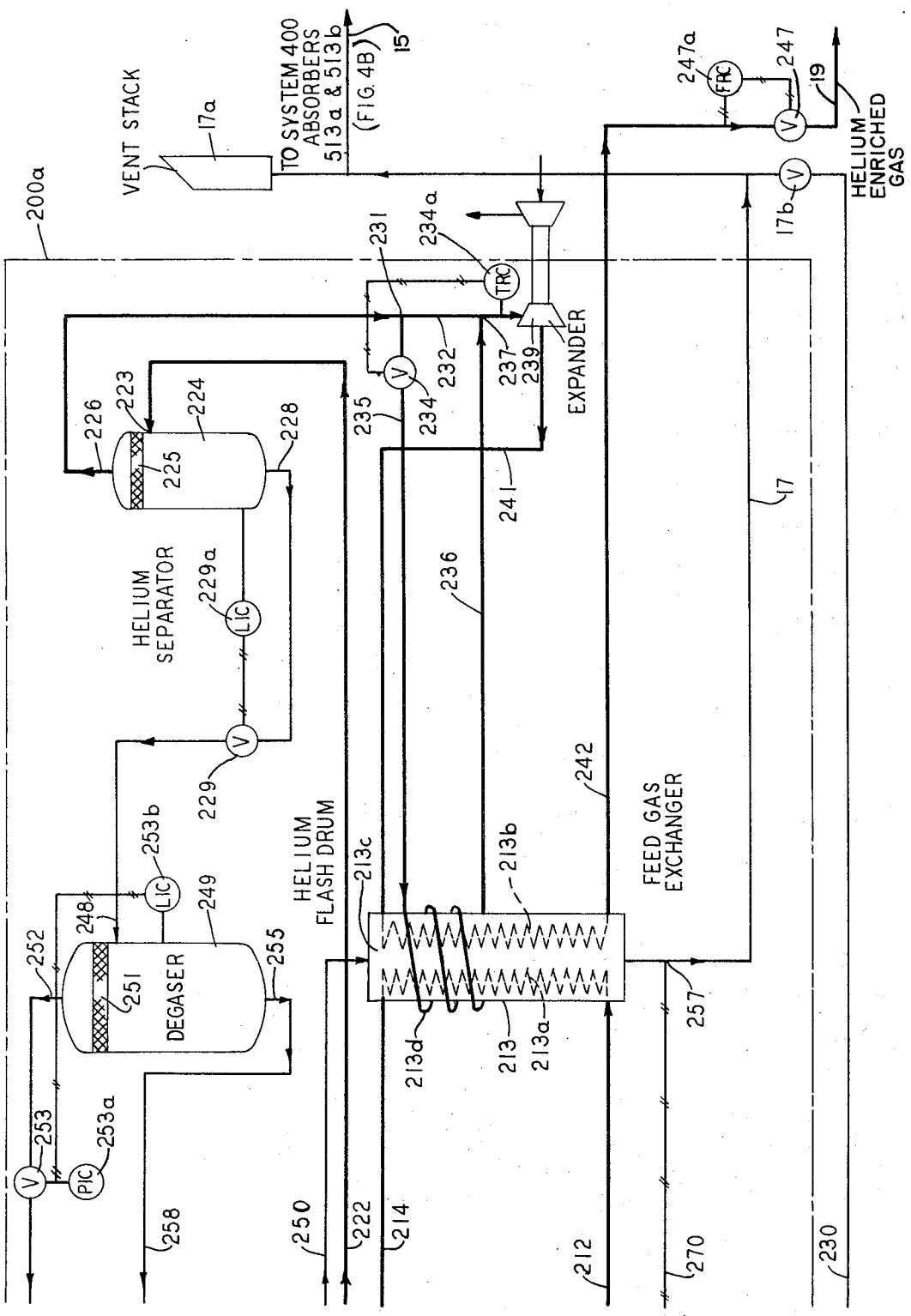


FIG. 3B

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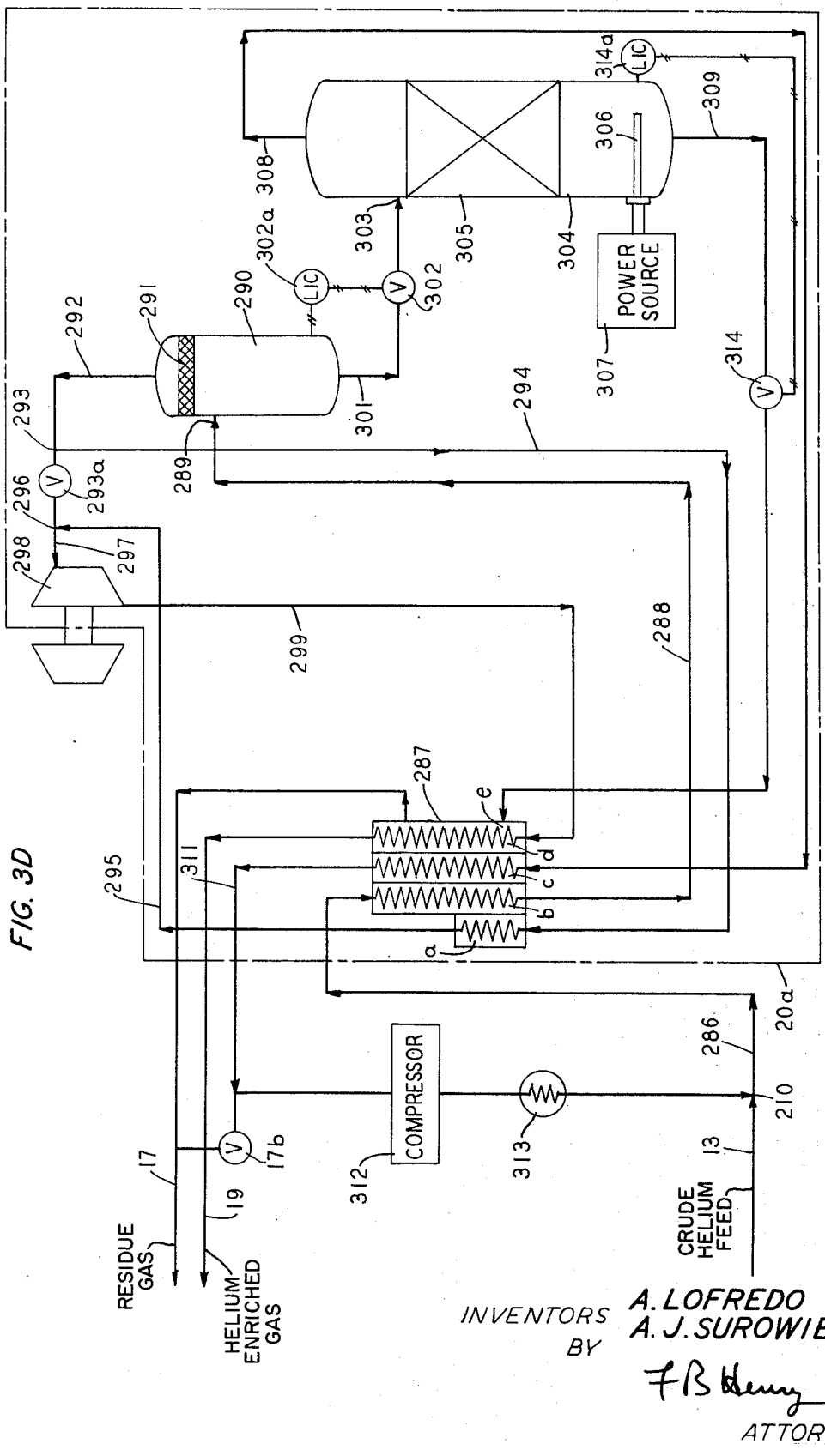


FIG. 3D

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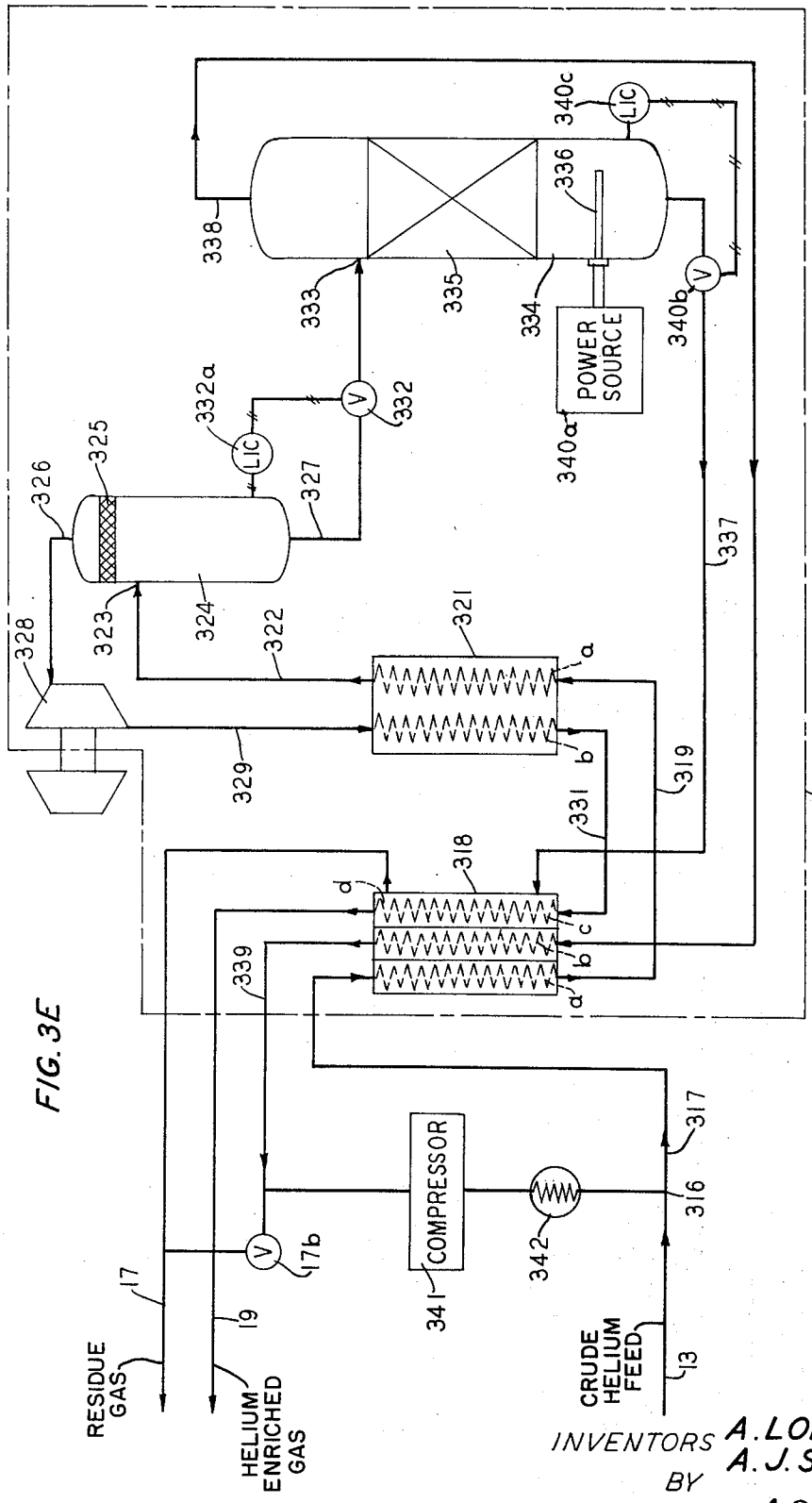


FIG. 3E

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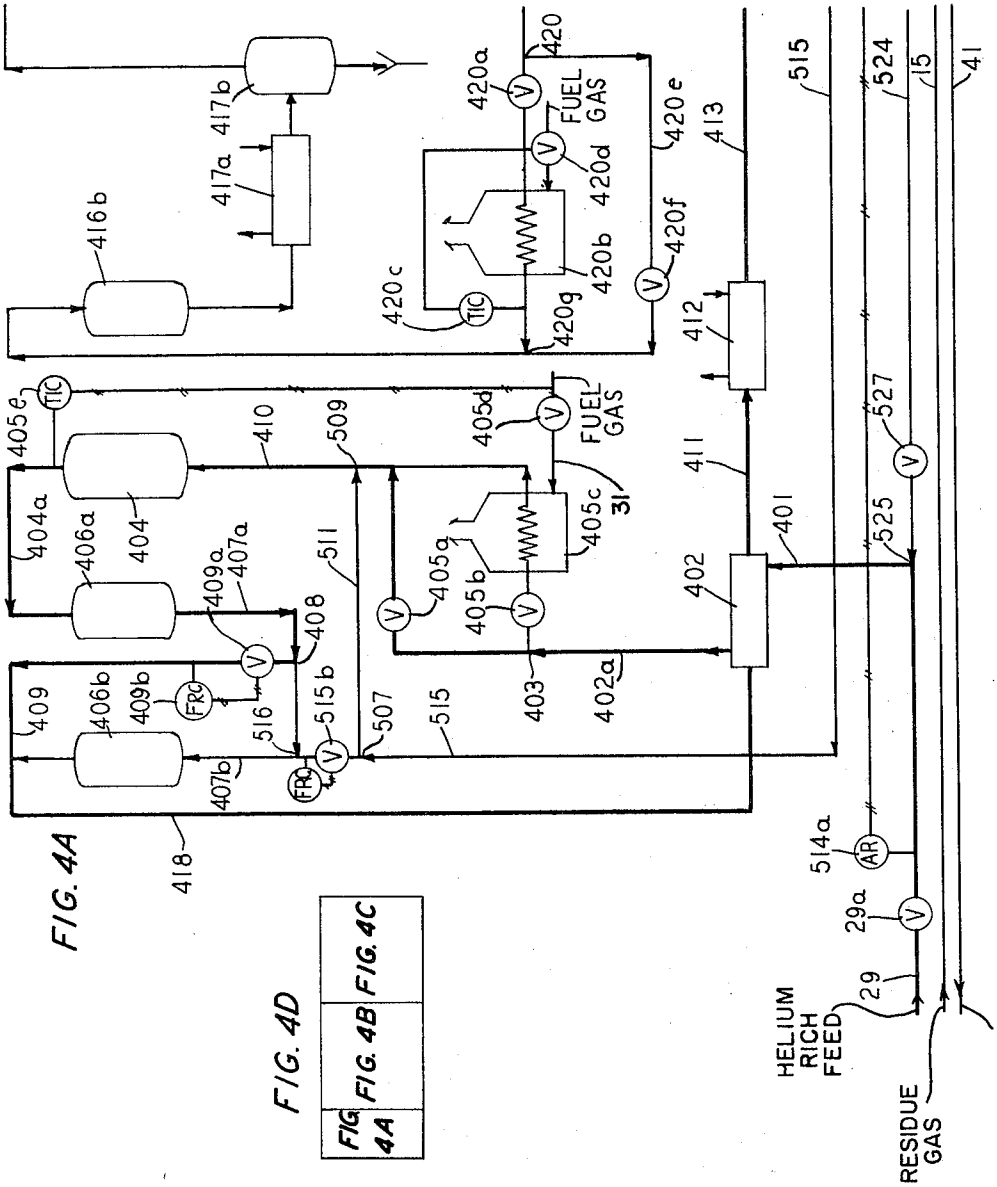


FIG. 4A

FIG. 4D

FIG 4A	FIG. 4B	FIG. 4C

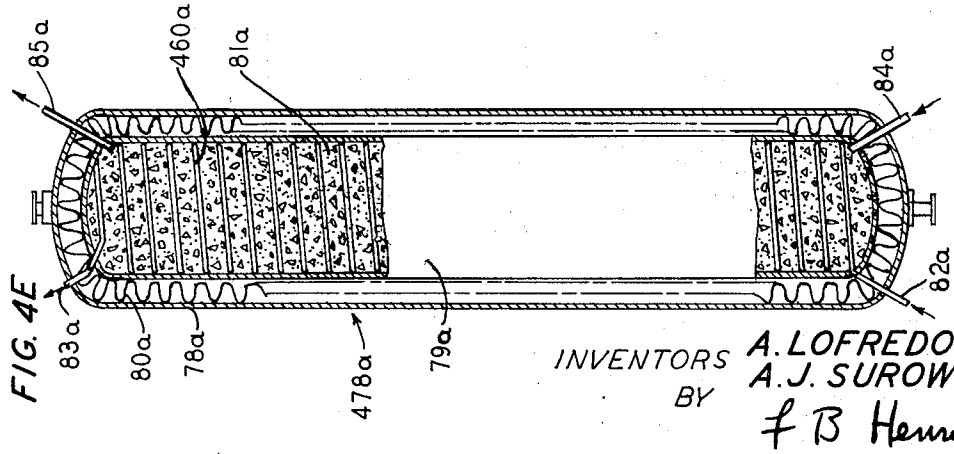


FIG. 4E

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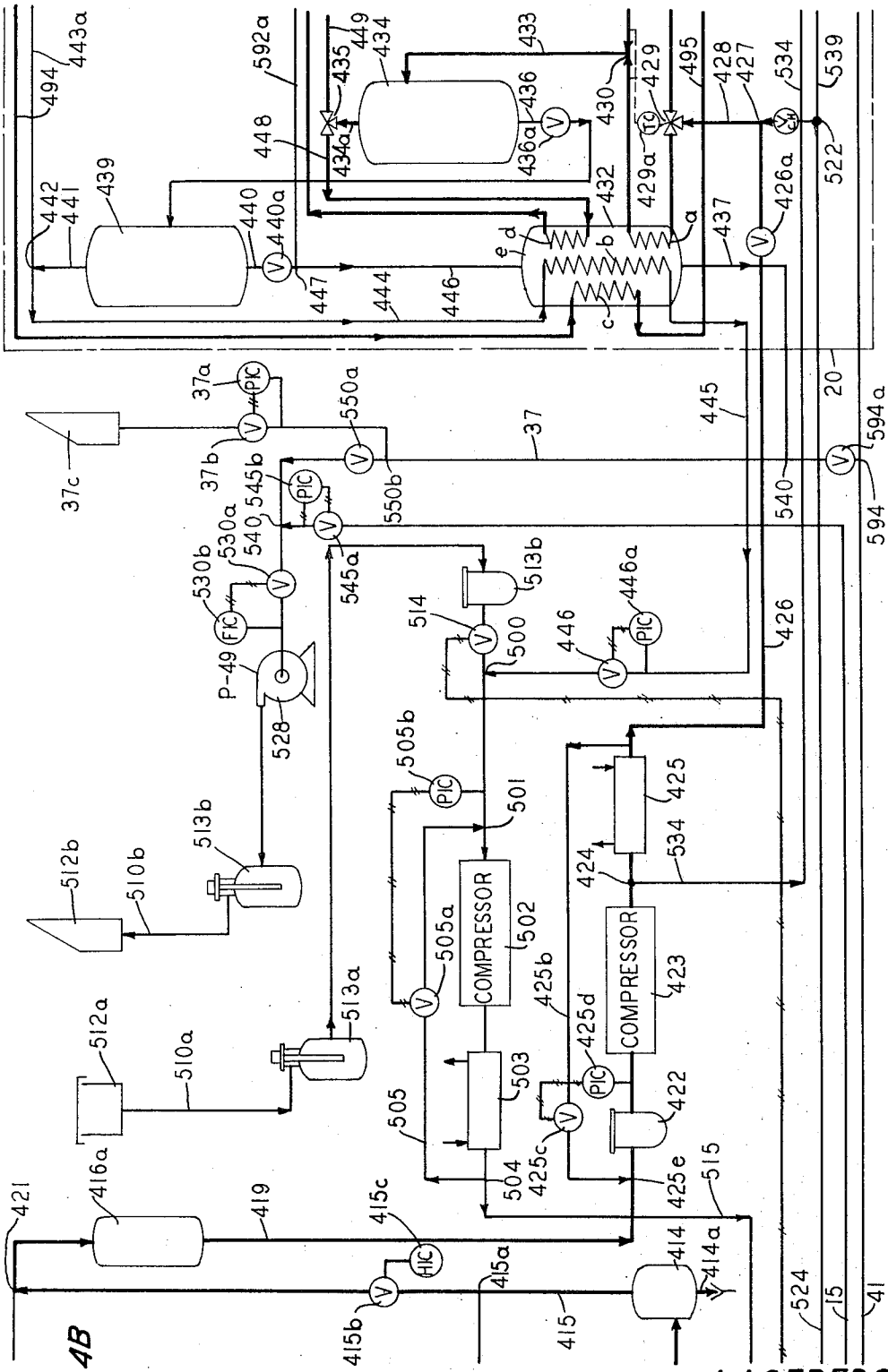


FIG. 4B

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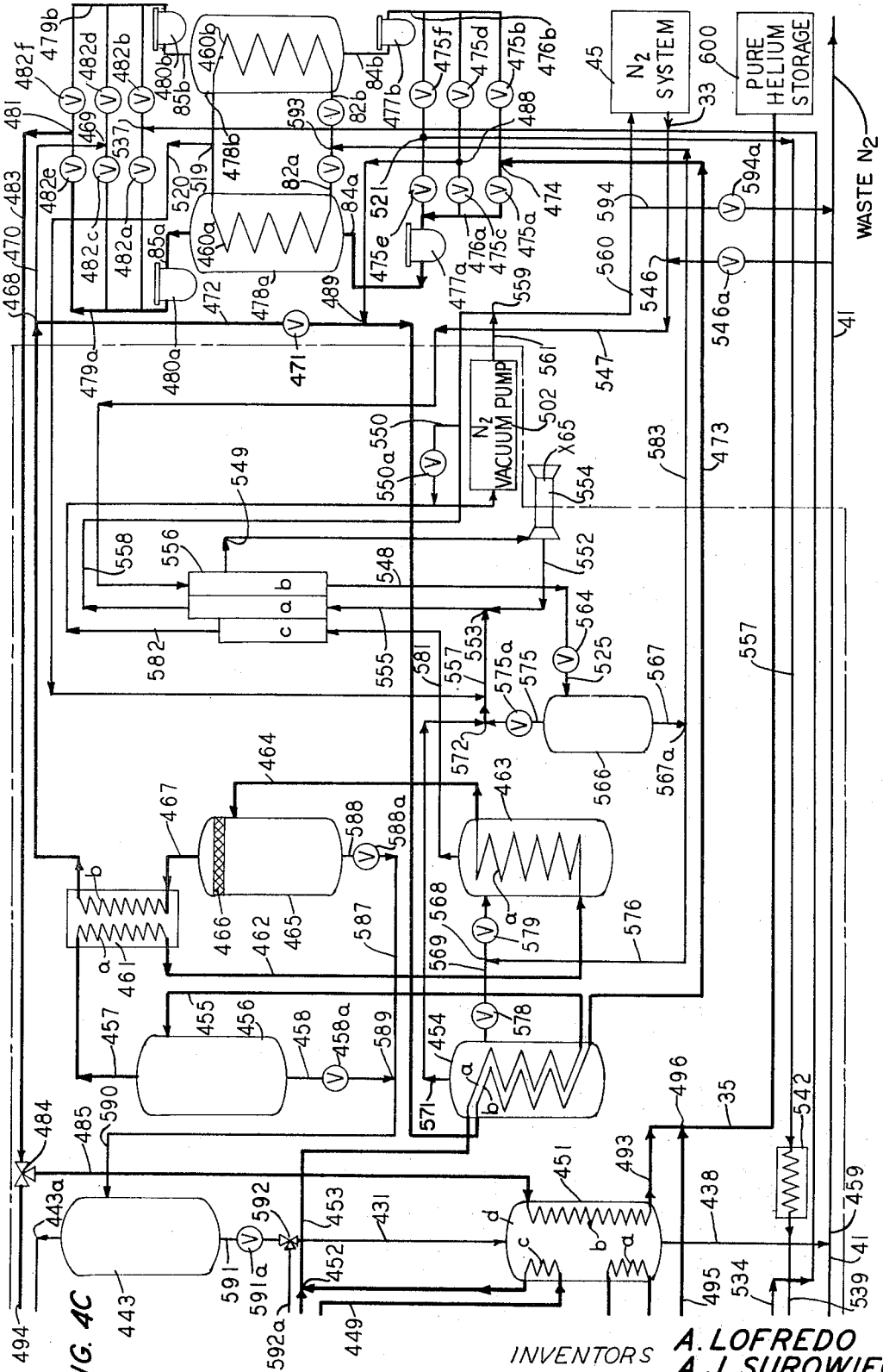


FIG. 4C

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PROCESS AND SYSTEM FOR THE PRODUCTION AND PURIFICATION OF HELIUM

BACKGROUND OF THE INVENTION

This relates in general to the purification and processing of low boiling gases, and more particularly, to a process comprising a series of operations for producing highly purified helium, together with commercial by-products, from natural gas.

Helium has many applications in modern technology, the most important of which are in the aircraft and missile industry, where it is used to pressurize missile and rocket propellant tanks. An important metallurgical use of helium is in the shielded-arc welding of metals, and in plasma-jet torches used for cutting. In addition, it has many refrigeration applications, such as providing low temperature environments for certain types of masers and lasers, and also for superconductor systems. Other important commercial applications are in providing a controlled inert atmosphere for growing germanium and silicon crystals, for cooling vacuum furnaces, and in processing fuel elements for nuclear reactors. Helium is also used in many laboratory and research operations.

These many present-day applications require substantial amounts of helium, which in most cases must be of a high grade of purity, substantially free of contaminants. Although small amounts of helium are present in the earth's crust, and atmosphere, the most important source is in natural gas. Inasmuch as the helium would ordinarily be lost if not extracted before the gas burns, it is important that it be separated from natural gas before the latter is consumed and marketed. Therefore, it is important to provide highly economical separation processes of high thermodynamical efficiency in which the losses of helium are minimized. It is also important that the residue gas from the helium separation process be available as fuel, and at temperatures and pressures suitable for piping in a natural gas system.

It is, accordingly, the principal object of this invention to improve the processing of low boiling gases, more particularly helium.

A more specific object is to provide more efficient production of high purity helium with the simultaneous production of refrigerants and fuel by-products in usable commercial form.

BRIEF DESCRIPTION OF THE INVENTION

These and other objects are attained in accordance with the present invention in a three-part system, each of whose units may be used as separate entities without the other two, or combined as a single integrated system. The first unit effects a crude separation of helium to between **60** and **80** mol per cent purity from a natural gas feed source. The second unit may use the output of the first, or the output of an independent source, or a combination from several sources, to produce an enriched helium product purified to between about **85** and **97** mol percent. The third unit may use the enriched product from the second unit, or from an independent source, to produce a final helium product of Grade A purity in which the contaminants are reduced to as low as three parts per million.

The process in the first unit produces crude helium from natural gas by a series of steps which comprise cooling the feed gas by heat exchange with the product

and residue streams, separating out condensed hydrocarbons, subcooling the remaining feed gas, stripping the feed gas of helium by flashing in a plurality of stages, deriving the crude helium product as vapor from the primary helium stripping stage, recycling the partially-helium vapor derived from the secondary helium stripping and flashing stages, and vaporizing the condensate so obtained to provide refrigeration for the process.

The process carried out in the intermediate unit enriches the crude helium stream having an initial pressure of about 80 atmospheres and a temperature of between about 45° and 100°F, by cooling it to below liquid nitrogen temperature, and using a helium separator to separate out the helium-enriched vapor product, which is returned, through the system, after a work-expansion step, to cool the feed stream. The liquid condensate from the helium separator is passed through one or more stages of flashing to remove dissolved helium vapor, which, together with other vapor components, including nitrogen, is returned through the system for recycling. Both the streams returned for recycling and the liquid condensate are used for refrigerating the feed streams. In a preferred embodiment, the enriched helium vapor passes through a reheat step to eliminate liquid, prior to work expansion in a turboexpander. In accordance with another feature, the liquid condensate from the flashing stages may be distilled in a reboiler in a heat exchange with a small component of partially liquefied feed stream to derive additional dissolved helium. In another embodiment, a subcooler is interposed between the feed gas heat exchanger and the helium separator.

In the process in the final unit, the helium-rich feed stream may be derived from the enriching unit, or from an independent source, at about 60 atmospheres pressure. This is passed through a platinum catalyst in the presence of air for the removal of all but a slight excess of hydrogen, which is removed subsequently in a copper oxide bed. The stream is dried, further compressed to about **186** atmospheres, and further cooled, after which it is passed through three stages of flashing with intervening cooling in heat exchangers, two of which include boiling nitrogen. The resultant helium vapor containing less than 1 mol percent nitrogen is passed through a low temperature, high pressure carbon adsorber which functions to remove the balance of the nitrogen from the helium stream. The nearly 100 percent pure product stream is then heat exchanged with feed streams, before passing to storage. Part of the refrigeration for the system is derived from the streams of liquid condensate flowing out of the flashing stages; whereas, the refrigeration for the carbon adsorber and nitrogen boilers is furnished by an off-site source of compressed nitrogen gas which is liquefied in an ancillary system. Nitrogen for the off-site source may be derived from condensate from flashing stages in earlier parts of the system which has been appropriately processed.

The principal advantage of the systems of the present invention is that they provide for the production of a helium product of 99.9969 minimum mol percent purity by processes of improved thermal efficiency, in which the need for external refrigeration is minimized, and in which the hydrocarbon by-product gases are of commercial quality.

Other objects, features, and advantages of the system of the present invention will be apparent to those

skilled in the art after a study of the detailed specification hereinafter with reference to the attached drawings.

SHORT DESCRIPTION OF THE DRAWINGS

FIG. 1 is a showing of the combined three-unit system in accordance with the present invention for producing grade A purity helium from natural gas;

FIG. 2 is a detailed showing in schematic diagram of the crude separation unit **100** of FIG. 1 of the drawings;

FIGS. 3A and 3B, combined as indicated in block diagram 3C, form a detailed showing in schematic diagram of the preferred embodiment of a helium enriching unit corresponding to block **200** in FIG. 1 of the drawings;

FIG. 3D is a detailed showing of an alternative form of the helium enriching unit shown in block **200** of FIG. 1;

FIG. 3E is a modification of the helium enriching unit shown in FIG. 3D;

FIGS. 4A, 4B, and 4C, combined as shown in diagram 4D, form a detailed showing in schematic of the final helium purification unit corresponding to block **400** in the block diagram of FIG. 1; and

FIG. 4E is a detailed showing of one of the carbon adsorbers of FIG. 4C.

Referring to FIG. 1 of the drawings, there is shown in a compilation of block diagrams, an overall system in accordance with the present invention, including a crude separation unit **100,** a helium enrichment unit **200,** and a helium purification unit **400.** Each of these units shown in block diagram will be described in substantial detail hereinafter. It will be noted that cooling water and instrument air lines have been omitted for simplification.

Unit **100** is retained in an insulated cold box **10,** the outer walls of which comprise a carbon-steel structure, having dimensions 9.5 feet by 9.5 feet by 30 feet. The space between vessels is filled with a heat insulating material, such as inorganic wool, the space being maintained by means of nitrogen gas at a pressure slightly above barometric pressure, for example at a pressure of about 5 inches of water.

Helium enriching unit **200** and helium final purification unit **400** can either be insulated separately in in structures of the general form of cold box **10,** or the two units **200** and **400** can be enclosed in spaced-apart relation in a single overall cold box **20.** The space between the walls of box **20** is also maintained at a pressure of about 5 inches of water, above barometric pressure.

The system shown in block **100** is indicated in detail in a preferred embodiment shown in FIG. 2 of the drawings. Unit **200** is shown in alternative configurations in the detailed diagrams of FIGS. 3A, 3B combined as FIG. 3C, and in FIGS. 3D and 3E of the drawings. Unit **400** is shown in detail in FIGS. 4A, 4B, and 4C, combined as FIG. 4D of the drawings.

Crude gas fed from the source **1,** which may be, for example, an underground natural gas dome, is piped through a 6-inch conduit under control of the valve **3,** into the crude separation unit **100.** The feed gas enters the unit through conduit **2** at a pressure of about 30 atmospheres and at a temperature of 80°F. This input stream comprises natural gas containing between 15 and 40 mol percent of helium.

After processing the natural gas feed in the crude separation unit **100,** in a manner to be described in detail hereinafter, a residue of processed fuel gas, which is methane-rich gas from which the helium has been removed, passes off through a 10-inch diameter conduit **4** under control of the valve **5,** at a pressure of about 5 atmospheres, and at a temperature between about 65° and 75°F. This stream has a heat rating of between 896 and 960 British Thermal Units per cubic feet, depending on the composition of the natural gas input. The stream is forced by centrifugal pump **6** to pass to fuel gas storage system **46.** Liquid disposal from the water coolers in the unit **100** flows off through the conduit **7.** The crude helium output, which is 60 to 80 mol percent pure, flows through the 6-inch diameter conduit **8** at a pressure of about 30 atmospheres and at a temperature of 65°F, through the normally open valve **9,** from which it is forced by the compressor **11** through a conduit system leading into the conservation pipeline **12*a*** for storage at about 80 atmospheres.

A feed stream from the conservation pipeline **12*a*** passes through the 6-inch diameter conduit **13** under control of valve **14,** from which it passes into the helium enrichment unit **200.** The latter can assume any one of the forms indicated in FIGS. 3A and 3B, combined as 3C, FIG. 3D or FIG. 3E. The stream through conduit **13** enters at the storage pressure of about 80 atmospheres and at a temperature of between 45° and 100°F. This feed stream consists of between about 60 and 80 mol percent helium, with a balance of additional components including hydrogen, nitrogen, and methane.

After the processing in one of the three alternative enrichment units **200,** a residue gas comprising waste nitrogen, including a small percentage of methane, either passes back into the system for recycling, or flows out through the conduit **17** at atmospheric pressure and at a temperature between 19° and 30°F, under control of the valve **18,** flowing at a flow-rate depending on the nitrogen in the input stream and the specific enrichment process, and passes off into the atmosphere or for other application, as explained hereinafter. The enriched product, which is now between 85 and 97 mol percent helium, depending on the composition of the input stream and the exact process used, includes as impurity small amounts of hydrogen and nitrogen. It flows out at a pressure of about 58 atmospheres and at a temperature somewhere between 19° and 100°F, depending on the specific one of the alternative processes used, through the 4-inch diameter conduit **19** to the junction **21.** At this point part of the flow may be returned, at the above pressure, by opening valve **30** leading through the 4-inch diameter conduit **22** to the conservation pipeline **12*b***, where it is maintained or stored at a pressure of approximately 59 atmospheres at ambient temperature.

The balance of the stream passes through the **4**-inch diameter conduit **24** to the junction **25.** An additional or alternative input stream of crude gas from a source **44,** alternative to the crude separation unit **100** or one of the helium enriching units **200,** may be directed through conduit **26,** under the force of pump **27** and under control of the valve **28.** For example, this may comprise a stream of 87.7 mol percent helium with a balance of nitrogen and hydrogen impurity, flowing at a pressure of about 59 atmospheres or a little less and at a temperature a little above ambient. The combined

stream from the junction **25** flows through the **4**-inch diameter conduit **29,** serving as input to the helium purification unit **400.** The merged input flow has an initial temperature of about 80°F and an input pressure of about **58** atmospheres. In the examples under description, this input stream has between 85 and 97 mol percent of helium, with impurities including 2 to 11 mol percent of nitrogen, less than 2 mol percent of hydrogen, and trace amounts of other impurities, including methane.

Also entering the helium purification unit **400,** through the intake conduit **31,** is a stream of fuel gas which is controlled by valve **32,** and which serves as fuel for preheat of the copper oxide beds for the reduction of hydrogen, as will be explained in detail hereinafter. In addition, a stream of nitrogen from an off-site source **45** passes at a pressure of about 41 atmospheres and at a temperature of 90°F, through the normally open valve **34,** into the purification unit **400** to function as a refrigerant in the process. The fuel gas supply **48** may be derived from fuel gas system **46**; and the nitrogen supply for source **45** may be derived from appropriately processed condensate from unit **400,** as will be described hereinafter.

After the final purification process has taken place in the unit **400,** which is described in detail hereinafter with reference to FIG. **4** of the drawings, Grade A product helium, having a minimum purity of 99.9969 mol percent, passes out through the 1-inch diameter conduit **35** under control of valve **36,** at a pressure of about 180 atmospheres and at a temperature of about 90°F, to storage **600,** pending loading in vessels or tanks for transportation to the points for use.

A stream of methane-rich nitrogen passes out of the system at about atmospheric pressure and at a temperature of 80°F, through the normally open valve **38.** Waste nitrogen, returning from the system at about atmospheric pressure and at a temperature of about 85°F, passes out through the 4-inch diameter conduit **41,** through the valve **42,** and into the atmosphere, or alternatively, through valve **49,** and conduit **51** to the nitrogen storage system **45** for reprocessing.

The several units which are components of the overall purification and enrichment system will now be described individually in detail. Each unit will be described, for the purposes of illustration, with reference to the processing of one or more input streams of preselected composition which in each case enters the unit at a stated pressure, temperature, and flow rate. However, it will be understood that the units described are not limited to the processing of feed streams of the specific characteristics recited by way of example.

The crude helium separation unit **100** is shown in detail in FIG. **2** of the drawings. Operation of unit **100** will be described with reference to two illustrative feed streams having the following characteristics.

Table 1

Feed Stream		
Flow Rate* (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	Case A	Case B
Composition (Mol %)	10,417	10,417
Helium	16.40	36.2
Nitrogen	24.20	26.8
Methane	53.50	33.7
Ethane	3.30	1.8
Propane	1.50	0.9
Butane	1.10	0.6

*The Flow Rates indicated in Table 1 above, and in the text and tables hereinafter are computed at 70°F at a pressure of one atmosphere.

In this system a feed gas having the general composition and flow rate indicated in Table 1, flows in at a pressure of 463 pounds per square inch absolute and a temperature of 80°F, through the conduit **2** to the junction **101,** where it merges with the stream returning for recycle. The combined stream flows from junction **101** through the conduit **102** to the column **103***a* of the five-column heat exchanger **103,** which may be of a brazed aluminum plate-and-fin type, where it is cooled to below −140°F by a heat exchange with the product and residue streams passing out through the other four columns.

The cooled stream then flows through the conduit **104** to the inlet **105** of the first stage **106***a* of the flash drum **106.** The latter is a conventional cylindrical flash vessel of stainless steel or the like, 10 to 12 feet tall and about 2 feet in diameter, bowed outwardly at the top and bottom ends, and having two compartments of about equal height, superposed vertically. The vessel **106***a* is designed to have approximately a 2 minute residence time. At the top of vessel **106***a* is interposed a demister **108,** comprising a pancake-shaped wire mesh 6 inches thick, for removing entrained liquid from the rising vapor. The pressure in the first-stage flash drum **106***a* is maintained at 447.7 pounds per square inch absolute, and the temperature at −147.8°F in Case A and 143°F in Case B, so that the low boiling gases, including helium, nitrogen, and some methane, rise to the top, whereas most of the heavy hydrocarbons become liquefied and fall to the bottom of the vessel. Leading out from the dome of the first-stage flash drum **106***a* is a conduit **115** through which the low boiling helium-rich vapor passes out. This vapor has the composition and flow rate indicated in the following Table 2.

Table 2

Flash Gas From Flash Drum 106*a*		
Flow Rate (Std. Cubic Ft./Min.) [at 70°F, 1 atm. pressure]	Case A	Case B
Composition (Mol %)	9,699	10,358
Helium	17.82	36.54
Nitrogen	28.29	28.62
Methane	52.74	33.49
Ethane	1.09	1.21
Propane	0.05	0.13
Butane	0.01	0.01

The helium-rich vapor passing out through conduit **115** from the first-stage flash drum **106***a*, flows at a pressure of 447.68 pounds per square inch absolute, and at a temperature of −148.7°F in Case A and −143°F in Case B.

Conduit **115** passes the vapor stream, substantially free of hydrocarbons and consisting essentially of helium, nitrogen, and methane, to column *d* of the four column heat exchanger **116.** In the heat exchanger **116,**

the vapor stream is further cooled to a temperature of −257.8°F in Case A, and −253.3°F in Case B, by countercurrent heat exchange with the streams of returning condensate, helium product, and helium bearing vapor returning for recycle. The cooled helium-rich stream then passes through the conduit **117** to the inlet pipe **118** of the first stage **119***a* of helium stripper **119.**

The helium stripper **119,** which is a stainless steel cylindrical vessel 23 feet high and 32 inches in diameter, is separated vertically into three substantially equal length compartments, the first stage **119***a* at the bottom, the second stage **119***b* in the middle, and the third stage **119***c* at the top. At the top of each stage is interposed a de-mister comprising a stainless steel wire mesh 4 inches thick, which serves to entrap liquid passing up with the vapor through the top stage.

The bottom stage **119***a* is maintained at an internal pressure of about 446 pounds per square inch, absolute, and at a temperature of −257.8°F, in case A, and −253.3°F, in case B. The partial-liquid entering through inlet pipe **118** is flashed in vessel **119***a*. The residue, which includes primarily nitrogen and methane, sinks to the bottom of the vessel; while the vapor phase, consisting of about 60 mol percent helium, rises to the top of this stage. The vapor then passes upward and out through the conduit **143,** leading from the top of the lower stage of helium stripper **119** at a temperature of −257.8°F, in case A, and −253.3°F, in case B, and a pressure of 445.68 pounds per square inch, absolute. This crudely purified product stream, comprising about 63 mol percent of helium, passes from conduit **143** to the passage *c* of the heat exchanger **116,** where it is warmed up to a temperature of −190°F, in case A, and −168° F, in case B, in a heat exchange with the stream being cooled. The warmed crude-product stream then passes out of heat exchanger **116** through the conduit **144** and into passage *b* of the feed gas heat exchanger **103.** In the latter, the stream is warmed up to a temperature of 69°F, in case A, and 72°F, in case B; and passes out of the unit **100** at a pressure of 440 pounds per square inch, absolute for storage in the conservation pipeline **12***a* of FIG. **1,** from which it may either be transported for immediate use, or piped to helium enriching unit **200** for further purification. The flow rate and composition analysis of the crude product stream is shown in Table 3 below.

Table 3

Crude Helium Product

	Case A	Case B
Flow Rate (Std. Cubic ft./min. [at 70°F, 1 atmos. pressure]	2705	5992
Composition (Mol %)		
Helium	63.15	62.92
Nitrogen	31.87	31.27
Methane	4.97	5.80
Ethane	0.006	0.01

Returning now to the auxiliary systems, the liquid condensate, comprising primarily the heavy hydrocarbons, which collects in the bottom of the first-stage flash drum **106***a*, flows off through pipe **111** through valve **112,** at a pressure of 447.7 pounds per square inch, absolute. Valve **112** is pneumatically controlled by a liquid level device **112***a* in a manner well-known in the art, to maintain the liquid in the bottom of the flash vessel at a preselected level. The temperature, flow rate, and composition of the condensate are given in the following Table 4.

Table 4

Liquid Condensate — First-Stage Flash Drum

	Case A	Case B
Temperature (°F)	−148.7	−143.0
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atmos. pressure]	1238	326
Composition (Mol %)		
Helium	0.235	0.46
Nitrogen	7.364	7.09
Methane	51.576	29.65
Ethane	19.37	19.19
Propane	12.24	24.77
Butane	9.217	18.84

The hydrocarbon liquid stream passes through valve **112** and conduit **113** to the second-stage flash drum **106***b*, which is maintained at an internal pressure of about 197.68 pounds per square inch, absolute. Methane-rich liquid forms in the bottom of this vessel, the vapor, including some dissolved helium, rising to the top of the second-stage **106***b* and passing out through the conduit **109** at a pressure of 197.68 pounds per square inch, absolute. The temperature, flow rate, and composition of this partial helium stream are given below in Table 5.

Table 5

Vapor - Second-Stage Flash Drum

	Case A	Case B
Temperature (°F)	−169.5	−149.6
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	198	33
Composition (Mol %)		
Helium	1.42	4.43
Nitrogen	28.95	39.70
Methane	68.58	54.25
Ethane	1.02	1.49
Propane	0.04	0.13

This partially helium vapor stream passes into the passage *c* of heat exchanger **103** where it is warmed up to a temperature of 69°F, in case A, and 72°F, in case B, passing out through the conduit **110** and into the junction **139,** at which point it is joined by a partially helium stream returning from the helium stripper, as will be described hereinafter. The pressure of this returning recycle stream is retained at a preselected value by means of valve **153** connected to the feed stream, which is operated pneumatically by the pressure-responsive device **153***a* when the pressure in conduit **110** becomes low.

The methane-rich liquid condensate in the second-stage flash drum **106***b* passes out through conduit **114** to junction **147,** under control of valve **148,** which is operated pneumatically by the liquid level control device **148***a* to maintain the liquid in the vessel at a predetermined level. This liquid stream has a temperature and composition indicated in Table 6, below.

Table 6

Methane-Rich Condensate — Second-Stage Flash Drum

	Case A	Case B
Temperature (°F)	−169.5	−149.6
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atmos. pressure]	1040	293
Composition (Mol %)		
Helium	0.0030	0.018
Nitrogen	3.241	3.456
Methane	48.327	26.908
Ethane	22.874	21.167
Propane	14.572	27.515
Butane	10.978	20.936

From junction **147,** this methane-rich stream passes to column *e* of heat exchanger **103,** either alone, or merged with a stream of liquid condensate from helium stripper **119,** as will be described hereinafter.

Returning now to helium stripper **119,** liquid condensate, including nitrogen and methane, collects in the bottom of the lowest stage **119***a*. This liquid is drawn off through conduit **145** under control of the valve **124,** which is pneumatically (or electrically) regulated to keep the level of the liquid in the lowest stage **119***a* at a preselected level. This stream passes off at a pressure of 445.7 pounds per square inch absolute. Its temperature, flow rate, and composition are given in Table 7, below.

Table 7

Condensate — First-Stage Helium Stripper

	Case A	Case B
Temperature (°F)	−257.8	−253.3
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atmos. pressure]	6994	4366
Composition (Mol %)		
Helium	0.286	0.316
Nitrogen	26.907	24.986
Methane	71.219	71.518
Ethane	1.513	2.856
Propane	0.068	0.299
Butane	0.007	0.025

The stream drawn through the valve **124** passes into the second stage **119***b* of the stripper through conduit **120.** This vessel is maintained at an internal pressure of 197.7 pounds per square inch absolute. The process is again repeated with the vapor, including dissolved helium, rising to the top of the second-stage vessel **119***b,* and the major portion of the condensate, including nitrogen and methane, becoming liquefied in the bottom.

The vapor from this stage rises through the demister **122***b*, and passes out through the outlet **126.**

The stream in conduit **126** passes out through the valve **151** to the junction **133,** where it is joined by a stream from the third stage **119***c* of the helium stripper from conduit **132.** Valve **151** is controlled by a pneumatically actuated control device **151***a* which responds to the pressure in line **126.** Vessel **119***c* is maintained at an internal pressure of **80** pounds per square inch absolute.

The stream of liquid from stage **119***b* passes out from the bottom through conduit **128** at a pressure of 185 pounds per square inch gauge, through valve **129,** which is pneumatically controlled by a liquid level device **129***a* to maintain the liquid level in vessel **119***b*.

The liquid passes into the upper stage **119***c* of the helium stripper through intake **125.** The temperature, flow rate, and composition of this condensate are given in Table 9 below.

Table 9

Condensate — Second-Stage Helium Stripper

	Case A	Case B
Temperature (°F)	−258.4	−254.1
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atmos. pressure]	6952	4334
Composition (Mol %)		
Helium	0.072	0.072
Nitrogen	26.761	24.776
Methane	71.571	71.948
Ethane	1.522	2.877

As in the stage below, the helium-rich vapor rises to the top of stage **119***c* and passes out through demister **122***c* and conduit **132** at a pressure of 80 pounds per square inch absolute, the temperature, flow rate, and composition being given in Table 10, below.

Table 10

Vapor — Third-Stage Helium Stripper

	Case A	Case B
Temperature (°F)	−264.4	−261.1
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	280	202
Composition (Mol %)		
Helium	1.740	1.53
Nitrogen	84.080	82.15

The liquid collected in the bottom of the third-stage vessel **119***c* passes off through the conduit **123** under control of valve **150** and pneumatic liquid level device **150***a*, at a pressure of 80 pounds per square inch, absolute. The temperature, flow rate, and composition are given below in Table 11.

Table 11

Liquid — Third-Stage Helium Stripper

	Case A	Case B
Temperature (°F)	−264.4	−261.1
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	6672	4132
Composition (Mol %)		
Helium	0.002	0.001
Nitrogen	24.355	21.978
Methane	73.980	74.661
Ethane	1.585	3.017
Propane	0.071	0.316
Butane	0.007	0.027

The stream flowing from the conduit **123** passes back through the column *a* of heat exchanger **116** where it is heated up to a temperature of −190°F in case A and −167°F in case B, in a heat exchange with the cooling liquids. Conduit **146** leads out from column *a* of heat exchanger **116** to the junction **155.**

In accordance with one alternative, valve **157** is closed and the methane-rich stream passes through open valve **156** to passage *f* of heat exchanger **103,** where it is heated up to a temperature of 69°F and 72°F in cases A and B, respectively. This stream then flows through conduit **158** to junction **160.** If valve **161** is open and valve **162** is closed, the stream flows through

conduit **163** to junction **164**, where it joins the recycle stream through compressor **137**. If valve **161** is closed and **162** is open, the stream flows to junction **169** where it joins the stream through valve **167**, as will be described, being pumped to fuel gas system **46** for use commercially, or in other units of the purification system.

Assuming valve **157** to be closed, the stream of liquid condensate which has passed out of conduit **114** in flash vessel **106***b*, passes through valve **148** and moves as a separate stream into conduit **149** and column *e* of heat exchanger **103**, in which it is heated up to 69°F, in case A, and 72°F in case B. It subsequently passes through conduit **159** to junction **165** where, assuming valve **167** to be closed and valve **166** to be open, it passes through conduit **168** to junction **164** to join the recycle through the system. Assuming valve **167** to be open and valve **166** to be closed, the methane-rich stream passes into junction **169** where it merges with the stream from valve **162**, the merged stream passing out through conduit **4**, as previously described. Such a system provides flexibility in control of the volume and composition of the recycle stream. The increased recycle stream obtained in this manner supplies additional refrigeration through joule-thompson expansion after compression.

Assuming valve **156** to be closed and valve **157** to be open, the two methane-rich streams merge at junction **147** ahead of heat exchanger **103**, the merged stream passing through column *e*, conduit **159**, through open valve **167**, and to outlet conduit **4** through junction **169**. This merged stream of residue gas, which passes through outlet **4** at a pressure of 73 pounds per square inch, absolute, has the following temperature, flow rate, heat capacity, and approximate analysis, as indicated in Table 12, below.

Table 12

Residue Gas

	Case A	Case B
Temperature (°F)	−190	−167
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	7712	4425
Heat Capacity (B.T.U. per Cubic ft.)	902.8	896.2
Composition (Mol %)		
Helium	0.0023	0.0024
Nitrogen	21.5085	20.7498
Methane	70.5221	71.4957
Ethane	4.4552	4.2203
Propane	2.0261	2.1191
Butane	1.4858	1.4127

This gas, which is suitable for fuel, is passed to fuel gas system **46** of FIG. **1**.

Returning now to the helium stripper, the partially helium vapor streams coming out the tops of the second and third stages **119***b* and **119***c* which are combined at junction **133**, pass as a combined stream through the conduit **134**, being warmed up to −190°F, in case A, and to −168°F, in case B, in column *b* of heat exchanger **116**. The stream then returns through conduit **135** and column *d* of heat exchanger **103** where it is warmed up to 69°F, in case A, and 72°F, in case B, passing out through the conduit **136**. At this point the stream has a pressure of 75 pounds per square inch, absolute. The pressure in this line is maintained at a preselected value by the pressure controlled valve **152**, which connects this line to the feed stream **102**. The temperature, flow rate, and composition are given in Table 13.

Table 13

Recycle from Helium Stripper

	Case A	Case B
Temperature (°F)	69	72
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	322	334
Composition (Mol %)		
Helium	6.197	5,879
Nitrogen	79.844	78.077
Methane	13.956	16.040
Ethane	0.003	0.004

As previously described, this recycle stream may be merged with one or both, in whole or in part, of the hydrocarbon streams from conduits **158** and **159**. The recycle stream passes into the compressor **137** where the pressure is raised to 190 pounds per square inch, absolute. The compressor **137** may be of any of the types well-known for helium separation systems. (See page 122 et. seq., Technology of Liquid Helium, National Bureau of Standards, Monograph, 1968). The compressor **137** is followed by a water-cooled after cooler **138**, which compensates for the heat generated during the compression. The combined streams from the junction **139** pass through the conduit **140**, where the merged stream is again compressed in the compressor **130**, of similar type to **137**, passing out of the latter through the conduit **141** and through the after cooler **142**, for further cooling. The returning, recycled stream, at this point, has a temperature of 100°F and a pressure of 463 pounds per square inch, absolute. The temperature, flow rate, and analysis are shown in Table 14.

Table 14

Combined Recycle

	Case A	Case B
Temperature (°F)	100	100
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	520	567
Composition (Mol %)		
Helium	4.37	5.70
Nitrogen	60.42	73.37
Methane	34.81	20.72
Ethane	0.39	0.19
Propane	0.01	0.02

The combined recycle stream is passed into the junction **101** where it combines with the feed system for recycling through the system.

From the crude helium unit **100**, as previously described, the crude product stream may pass into conservation pipeline **12***a*, for storage at a pressure of about 80 atmospheres. From pipeline **12***a* the stream may be directed into enriching unit **200**. This may take the form of any of the systems indicated in FIGS. **3C**, **3D** or **3E** of the drawings.

Let us refer, now, to the system shown in FIGS. **3A**, **3B**, assembled as shown in FIG. **3C**, which is preferred for the purposes of the present invention. The crude helium stream passes in through the conduit **13** at a pressure of 1,213 pounds per square inch gauge. The temperature, flow rate, and composition are given in Table 15, below.

Table 15

Feed Stream — Preferred Enriching Unit

	Case A	Case B
Temperature (°F)	45	100
Flow Rate (Std. Cubic ft./min.) [70°F, 1 atm. pressure]	15,278	15,278
Composition (Mol %)		
Hydrogen	1.26	0.98
Helium	64.92	75.66
Nitrogen	32.85	22.90
Methane	0.97	0.46

This stream initially passes through a filter **201** comprising, for example, sintered stainless steel for removing any entrapped solids. The flow into the system is under control of a conventional cryogenic valve **206**, electronically or pneumatically controlled by a pressure control system **206***a* which may be any one of the types well-known in the art. The incoming feed stream flows into the junction **207** where it is joined by the recycled stream **283**, returning through a circuit which will be described in detail hereinafter.

The combined feed and recycle stream in conduit **208** flows at a pressure of 1,213 pounds per square inch, absolute. The temperature, flow rate, and composition are given below in Table **16.**

Table 16

Feed and Recycle Stream — Preferred Enrichment Unit

	Case A	Case B
Temperature (°F)	46	100
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	15,494	15,404
Composition (Mol %)		
Hydrogen	1.5613	1.1140
Helium	64.4151	75.3063
Nitrogen	33.0668	23.1234
Methane	0.9568	0.4563

This combined stream passes into the junction **209**, at which point between about 1 and 2 percent by volume flows through the arm **211** and the remaining major portion, flows into the arm **212.** The conduit **212** passes into the conventional wound-tube type heat exchanger **213** through the coil **213***a* where it is cooled down to a temperature slightly below −316°F. The stream, having substantially the same analysis as previously indicated, then flows into the temperature controlled valve **215.** The latter is electronically or pneumatically controlled by a temperature-differential indicating circuit **215***a*, which is connected between the outlet **257** of the heat exchanger **213** and outlet **260** of heat exchanger **217**, and which responds to maintain the temperature differentials across the heat exchangers **213** and **217** substantially constant. The valve **215** is a three-way valve having, in addition to the lead-in from the conduit **214**, a lead-in for the minor component through conduit **266.** This minor component stream is derived from a path which passes through the conduit **211** through the passage **217***a* of heat exchanger **217**, where it is separately cooled down to between −264°F and −281°F (depending on the composition of the input), through conduit **264**, reboiler coil **265**, returning through conduit **266** to the left-arm of the three-way valve **215.** The reboiler coil **265** serves to further cool the minor component in a heat exchange with evaporating condensate in helium stripper **259**, as will be further described hereinafter.

The streams combine at the output of valve **215** to form a single stream which passes through the conduit **222** at a pressure of 1,181 and 1,184 pounds per square inch, absolute, in cases A and B, respectively, and at a temperature of −315.7°F, the flow rate and composition being indicated in the foregoing Table **16.**

The conduit **222** passes to the intake **223** of the helium separator **224.** The latter is a conventional flash vessel maintained at an internal pressure of about **1181** and **1184** pounds per square inch absolute, in Cases A and B, respectively. This vessel comprises, for example, stainless steel 1½ feet in diameter and 9 feet high, having a de-mister **225**, which comprises a disc of wire mesh, say 6 inches thick. The enriched helium vapor rises to the top of this vessel, the higher boiling components, including most of the nitrogen, which have become liquefied, falling to the sump where they collect. The liquid level in the sump is maintained constant by means of the output valve **229**, connected to the conduit **228** at the bottom of the vessel. Valve **229** is either electronically or pneumatically controlled by a liquid-level control circuit **229***a*, most likely taking the form of a differential pressure sensing element.

The vapor output from the dome of the vessel **224** passes out through the conduit **226** at a pressure of 1181 and 1184 pounds per square inch, absolute, in cases A and B, respectively, and at a temperature of −315.7°F, the flow rate and composition being indicated below in Table **17.**

Table 17

Enriched Product Stream

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	10,364	12,015
Composition (Mol %)		
Hydrogen	1.7276	1.1902
Helium	95.6954	96.2061
Nitrogen	2.5571	2.5896
Methane	0.0199	0.0141

This helium-enriched product stream passes into the junction **231.** Assuming temperature-regulated control valve **234** to be closed, the entire stream passes through conduit **232** to junction **237** at the intake of the turbo-expander **239.** If the probe from the electronic or pneumatic temperature-regulating circuit **234***a*, which is interposed in the expander intake stream, indicates that the stream is likely to be below the condensation point after turbo-expansion, valve **234** is opened to allow part of the stream to pass through conduit **235** to the outer channel **213***d* of the wound-tube heat exchanger **213**, where it undergoes a reheat step, being warmed up from −315.7°F to 33°F and 99°F, in cases A and B, respectively. The reheated stream flows at pressures of 1,180 and 1,183 pounds per square inch, absolute in cases A and B, respectively, back into the main stream in conduit **232**, where it serves to maintain the stream at a preselected temperature.

The temperature-controlled stream flows into the conventional turbine expansion device **239**, where it is expanded with the performance of work to a pressure of about 875 pounds per square inch, absolute, being thereby cooled to a temperature of just below −319°F. The function of the reheat step is to prevent the forma-

tion of a liquid phase as the stream passes into the turbo-expansion device, thereby reducing the errosive effect on the latter. The expansion operation may, in some embodiments, serve to supply energy to other parts of the system, including the compressor stages **276** and **278** of the circuit with which the expander may be connected for tandem operation, in a manner well-known in the art.

After work-expansion, the cooled low-pressure stream returns through the inner tube **213***b* of heat exchanger **213** where it is heated up to a temperature of 33°F, in case A and 94°F in case B, passing into the conduit **242** and through the flow-regulated valve **247.** The latter is operated under control of conventional electronic or pneumatic means **247***a* to regulate the flow into the output channel **19** in accordance with a predetermined flow rate. The stream flows out from the channel **19** at a pressure of about 863 pounds per square inch, absolute, the temperature, flow rate, and composition being given below in Table 18.

Table 18

Enriched Product Output

	Case A	Case B
Temperature (°F)	33	94
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	10,364	12,015
Composition (Mol %)		
Hydrogen	1.7276	1.1902
Helium	95.6954	96.2061
Nitrogen	2.5571	2.5896
Methane	0.0199	0.0141

Returning now to the helium stripper **224,** the liquid settling in the bottom, as previously indicated, passes out through the vent **228** and the valve **229** which is electronically or pneumatically regulated by liquid level control circuit **229***a* to keep a preselected liquid level in vessel **224.** The liquid stream passes to the intake **248** of the helium flash drum **249,** serving as a degaser, which is maintained at an internal pressure of about 30.0 pounds per square inch, absolute. The liquid in this entering stream flows into the vessel **249** at a pressure of about 1,181 or 1,184 pounds per square inch, absolute, in cases A and B, respectively, the temperature and flow rate being given in Table 19, below.

Table 19

Liquid Condensate from Helium Separator

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	5,130	3,389
Composition (Mol %)		
Hydrogen	1.2253	0.8438
Helium	1.2143	1.2211
Nitrogen	94.7107	95.9112
Methane	2.8497	2.0239

The helium flash drum **249,** which is a stainless steel cylindrical vessel with bowed ends, is, say, 6 feet high and 3 feet in diameter. It includes a de-mister **251** of wire mesh, which may be, for example, 6 inches thick, located at the top, for entrapping liquid rising with the vapor. The liquid falls to the bottom of the drum where it passes out through the conduit **255,** as will be described hereinafter.

The vapor rising through the de-mister **251** passes out through the conduit **252** in the dome at a pressure of about 30 pounds per square inch, absolute. The temperature, flow rate, and composition of this vapor are given in Table 20, below.

Table 20

Vapor from Helium Flash Drum

	Case A	Case B
Temperature (°F)	−325.2	−324.8
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	216	126
Composition (Mol %)		
Hydrogen	22.8713	17.3051
Helium	28.7058	32.5680
Nitrogen	48.4017	50.1148
Methane	0.0212	0.0121

This stream flows into the valve **253** which is actuated by the circuit means **253***a* and **253***b*, respectively, responsive to changes in the pressure and liquid level in the flash drum **249.** Output from the valve **253** passes into the junction **268** where it may be joined by additional amounts of vapor including trace amounts of hydrogen and helium derived from the conduit **267** out of the helium stripper **259,** in the case of vapor rising out of that vessel, as the result of reboiler action, as will be described. The returning stream, at a pressure of 20 pounds per square inch, absolute, in case A, flows at a temperature of −325.2°F and at the rate of 216 standard cubic feet per minute; whereas, in case B, the temperature is −324.8°F and the flow rate, 126 standard cubic feet per minute. This stream passes through the conduit **269** and into the outer channel **217***b* of heat exchanger **217** where it absorbs heat from the incoming feed stream, being thereby warmed up to a temperature of 33°F, in case A, and 94°F in case B, as it passes into the junction **260.**

The liquid passing out of the bottom of the helium flash drum **249** through conduit **255** typically flows at a pressure of 30 pounds per square inch, absolute. The temperature, flow rate, and composition are given in Table 21 below.

Table 21

Liquid from Bottom Helium Flash Drum

	Case A	Case B
Temperature (°F)	−325.2	−324.8
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	4914	3263
Composition (Mol %)		
Hydrogen	0.2736	0.2060
Helium	0.0056	0.0066
Nitrogen	96.7468	97.6856
Methane	2.9740	2.1018

This stream passes through the inlet **258** where it is introduced into the helium stripper reboiler **259** at a point just below the wire mesh de-mister **261.** The helium stripper **259** is a cylindrical stainless steel vessel with bowed ends, 6 feet high and 1½ feet in diameter. The wire mesh de-mister **261,** which is 4 inches thick, is located on the top. The mid-section of the vessel is filled with stainless steel packing, such as shown, for example, in the following referenec: Office of Scientific Research & Development, Rept. 3768, June 13, 1944, Liquid Air Fractionation.

In the bottom of the vessel **259**, coil **265** is connected between conduits **264** and **266**. The liquid from conduit **258** flows into the helium stripper-reboiler **259**, where the internal pressure is maintained at about 20 pounds per square inch absolute. Liquid settling in the bottom of the vessel **259** is boiled in a heat exchange with the small component of feed gas passing through coil **265**, cooling the latter and causing any remnants of helium to rise to the top of the vessel. Liquid mixed with any rising vapor is entrained in the wire de-mister **261**, above. The liquid which settles in the bottom passes off through conduit **240**, which is connected to a siphon arrangement **243**. The latter, is U-shaped, so that the inverted closed end protrudes physically above the horizontal plane coinciding with the liquid level in vessel **259**, providing a head which keeps the liquid in the vessel at a desired level. The closed top end of siphon **243** is connected through a pipe **244** which leads to normally open valve **245**, leading to junction **246**. Valve **245** may be manually adjusted to control the pressure differential across helium stripper **259**, and to allow it to be emptied.

The liquid which typically passes off through conduit **240** at a reduced pressure of 20 pounds per square inch, absolute, has the same temperature and composition as indicated in Table 21 above, except that, depending on the composition of feed from input conduit **258**, additional amounts of dissolved helium and hydrogen may pass out of conduit **267** and through junction **268** to conduit **269**.

The liquid stream of the composition described in Table 21 flows out through conduit **250**, through the inner tube **213***c* of heat exchanger **213**, where it is warmed up to 33°F in case A, and 94°F in case B, at a pressure near atmospheric, thereby cooling down the feed streams through heat exchange. This waste stream comprising mostly nitrogen then passes out through conduit **17** and to the atmosphere through vent **17***a*, or alternatively, for use in parts of the final helium purification system **400**, in a manner set forth hereinafter. In accordance with a further alternative, the nitrogen from conduit **17** is passed through valve **17***b* open for the purpose, and conduit **230**, to join conduit **271** for compression with the recycle stream.

At the junction **260**, the stream from the helium flash drum **249**, including any trace amounts from the helium stripper **259**, or any amounts of waste nitrogen from conduit **230**, passes into conduit **271** to junction **272** at a pressure slightly above atmospheric pressure and at a temperature of 33°F in case A, and 94°F in case B, the composition of this stream being substantially that given in Table **20** above.

At the junction **272**, the gaseous stream flows into the conduit **275** to compressor **276** where it is compressed from a pressure slightly above atmospheric pressure to a pressure of **1213** pounds per square inch, absolute. The stream passes out of compressor **276** and through the compressor aftercooler **278**, which is water cooled to compensate for the heat of compression. The compressed stream passes through the conduit **279** to the separator **281**, which comprises a flash drum, where any liquid, such as trace amounts of water, passes out through a drain **285**, in the bottom. The remaining gas passes out through a vent at the top of separator **281** and into the junction **282**. This stream, which has a pressure of **1213** pounds per square inch, absolute and a temperature of 100°F, has a composition substantially the same as that given in Table 20 above. This stream then flows back into the junction **207** where it joins the feed system for recycling through the system. A small component may be returned through the conduit **284** and the valve **274**, which is controlled by the pressure-sensitive circuit **274a** interposed in the conduit **271**. The function of this device is to keep the pressure in the conduit **271** at a preselected level.

Alternative to the helium enrichment circuit of unit **200** just described are the modified arrangements for helium enrichment shown in FIGS. 3D and 3E of the drawings.

Referring in detail to FIG. 3D, a crudely purified helium feed stream flows into the unit through the conduit **13** at a pressure of about 1,205 pounds per square inch, absolute. The temperature, flow rate, and composition, for alternative cases, are given in Table 22, below.

Table 22

Feed Stream — First Alternate Enrichment Unit

	Case A	Case B
Temperature (°F)	45	100
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	15,278	15,278
Composition (Mol %)		
Hydrogen	1.26	0.98
Helium	64.92	75.66
Nitrogen	32.85	22.90
Methane	0.97	0.46

This feed system flows into the junction **210** where it is united with a recycle stream, the combined stream then flowing into column *b* of heat exchanger **287** where it is cooled down in each case to a temperature of −315.7°F by counterflowing waste and product streams. This combined, cooled stream has approximately the composition indicated in Table 23, below.

Table 23

Cooled Feed and Recycle Stream

Composition (Mol %)	Case A	Case B
Hydrogen	1.5613	1.1140
Helium	64.4151	75.3063
Nitrogen	33.0668	23.1234
Methane	0.9568	0.4563

After being cooled, it flows into the intake **289** to the helium flash drum **290**. The latter is a cylindrical stainless steel drum with bowed ends, 9 feet high and 1½ feet in diameter, which is maintained at an internal pressure of about 80 atmospheres. At the top of the dome, there is interposed a wire mesh de-mister **291** which is 6 inches thick.

As in previously described flash drums, the vapor rises to the top, passing through the de-mister wire mesh **291** and out through the conduit **292** at a pressure of about 1,200 pounds per square inch, absolute, and at a temperature of −315.7°F. The entrapped liquid flows down to the bottom of the drum, where it passes out through the conduit **301**.

The vapor stream which passes into junction **293** has a flow-rate and composition indicated in Table 24, below.

Table 24

Flash Drum Vapor

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	10,364	12,015
Composition (Mol %)		
Hydrogen	1.7276	1.1902
Helium	95.6954	96.2061
Nitrogen	2.5571	2.5896
Methane	0.0199	0.0141

Assuming valve **293***a* to be closed, this stream passes into conduit **294** and through column *a* of heat exchanger **287** where it is warmed up to −308.7°F at the intake to the expander **298.** Assuming that the valve **293***a* is open, all or a portion of the stream from junction **293** may be taken directly to the junction **296** without passing through heat exchanger **287,** entering the expander at a temperature of −315.7°F, in which case the nitrogen and methane components may be liquefied in the expander. In either case, the stream from the junction **296** flows at a pressure of about 1,200 pounds per square inch, absolute, at substantially the flow rates and composition indicated in Table 24 hereinbefore.

Turbo expander **298** is substantially similar in form to expander **239,** described with reference to the system of FIGS. 3A and 3B combined as 3C. In accordance with a variation of present practice, this may be connected in tandem to compressor **312** so that the energy derived from expansion is utilized to drive the compressor.

In the turbo expansion chamber **298** where the pressure drops to about 895 pounds per square inch, absolute, with the performance of work, the temperature is decreased to −319.3°F or −319.8°F in cases A and B, respectively. The cooled, low pressure stream passes from the expander **298** through the conduit **299,** and through column *d* of heat exchanger **287,** where it serves to refrigerate the feed stream, ultimately passing out through the conduit **19** at a pressure of about 890 pounds per square inch, absolute. The final temperature of the stream depends on whether or not it undergoes the reheat step through the heat exchanger prior to entering the turbo expander **298.** Assuming the product stream has been reheated prior to expansion, which is the preferred case, the final temperature of the enriched product stream, after passing through column *d* of heat exchanger **287,** is 19°F in case A, and 94°F in case B. The composition of the emerging enriched product stream is as indicated in Table 24 hereinbefore.

The residue liquid in the bottom of the helium flash drum **290** passes out through the conduit **301** under control of the valve **302,** the latter being controlled electrically or pneumatically by the conventional liquid-level control circuit **302***a* to maintain the liquid level in the vessel **290.** This liquid stream flows out at a pressure of 1,200 pounds per square inch absolute and at a temperature −315.7°F. The flow rate and composition are indicated on Table **25,** below.

TABLE 25

Liquid from Flash Drum

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	5,130	3,389
Composition (Mol %)		
Hydrogen	1.2253	0.8438
Helium	1.2143	1.2211
Nitrogen	94.7107	95.9112
Methane	2.8497	2.0239

This stream passes into the intake **303,** which is directed to the upper portion of the helium stripper **304.** The latter is a cylindrical stainless steel drum with bowed ends, 6 feet high and 3 feet in diameter, which is maintained at an internal pressure of about 20 pounds per square inch, absolute. The cylindrical body portion is filled with 2 feet of stainless steel packing **305,** similar to **262,** FIG. 3A, which provides a surface for degassing the descending liquid and freeing dissolved helium vapors.

The liquid residue in the bottom of the container **304** is boiled slightly by means of a conventional bayonet heater **306** which is connected to a 2 kilowatt source of power **307.** Conduit **308** in the dome takes off the vapor rising to the top of the vessel, the liquid portion being entrained in the stainless steel packing **305** in the body portion of the container.

The vapor rising from the conduit **308,** has a pressure of about 20 pounds per square inch, absolute. The temperature, flow rate, and composition are indicated below in Table 26.

Table 26

Vapor from Helium Stripper

	Case A	Case B
Temperature (°F)	−320	−331
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	128	186
Composition (Mol %)		
Hydrogen	22.8713	17.3051
Helium	28.7058	32.5680
Nitrogen	48.4017	50.1148
Methane	0.0212	0.0121

The vapor from helium stripper **304** ultimately flows into column *c* of heat exchanger **287** where it is heated up to a temperature of 32.8°F, in case A, and 94°F in case B, passing out through the conduit **311** and into the compressor **312** where the pressure is raised from atmospheric pressure to about 1,205 pounds per square inch absolute, ultimately passing through the water-cooled after cooler **313,** to compensate for the heat of expansion. In one alternative, this recycle stream may be combined with waste nitrogen passing from conduit **17** through valve **17***a*. The compressed stream then passes into the junction **210** to merge with the feed stream for recycling through the system.

The liquid condensate in helium stripper **304** passes out through conduit **309** at a pressure of about 20 pounds per square inch absolute. The flow through valve **314** is controlled by conventional electronic or pneumatic means **314***a* to retain a liquid level in the bottom of helium stripper **304.** The temperature, flow rate, and composition of this stream are indicated in Table 27, below.

Table 27

Liquid from Helium Stripper

	Case A	Case B
Temperature (°F)	−325.2	−324.8
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	4914	3263
Composition (Mol %)		
Hydrogen	0.2736	0.2060
Helium	0.0056	0.0066
Nitrogen	96.7468	97.6856
Methane	2.9740	2.1018

This stream comprising mostly liquid nitrogen, passes through column *e* in the heat exchanger **287,** where it serves to refrigerate the incoming feed stream. It then passes out through conduit **17,** where it may either pass into the atmosphere, or through valve **17***b* for recompression and recycle. Alternatively it may be used in the final purification stage described with reference to unit **400,** hereinafter.

The system just described has been modified in accordance with the system indicated in FIG. 3E of the drawings by interposing in the path between the heat exchanger and the helium flash drum a second heat exchanger or subcooler which serves to further cool the stream prior to flashing.

In the system in FIG. 3E, the incoming feed stream in the feed conduit **13,** which contains 64 and 76 mol percent of helium, in cases A and B, respectively, has substantially the same flow rate, temperature, pressure, and composition as described with reference to the feed stream of the enrichment circuit of FIG. 3D (see Table 22). This feed stream flows through the junction **316** where it is united with the recycle stream, and then into the conduit **317** leading to the column **318***a* of heat exchanger **318,** where it is cooled down to a temperature of −312.3°F in case A and −312.3°F in case B. It then passes through the conduit **319** to the column *a* of heat exchanger **321** where it is further cooled down to −315.7°F in case A and −315.7°F in case B. The cooled, partially liquid stream passes through the inlet **323** of the helium flash drum **324,** which is a cylindrical stainless steel vessel similar to flash drum **290** of FIGS. 3A and 3B, containing a de-mister mesh **325.** The helium-enriched product vapor passes up through the wire mesh **325** where it is stripped of liquid, and out through the conduit **326.** This vapor, which is at the dew point, flows at a pressure of about 1,200 pounds per square inch, absolute, and has the same temperature, flow rate, and composition indicated with reference to Table **24** in the description of FIG. 3D.

The helium-enriched vapor stream then passes into the turbo expander **328,** of the type indicated in FIG. 3D, where it is work-expanded to a pressure of about **895** pounds per square inch gauge, being thereby cooled to a temperature of −319.3°F in case A and −319.8°F in case B. The cooled, low pressure enriched product stream then passes through the conduit **329** and into the channel *b* of the sub-cooler **321** where it serves to cool the incoming stream. From the subcooler, it ultimately passes through conduit **331** and into the column *c* of heat exchanger **318,** flowing out through the conduit **19** at a pressure of about 890 pounds per square inch absolute. The temperature, flow rate, and composition of the enriched product are as indicated with reference to the enriched helium product of FIG. 3D. As in the previous figure, the waste methane and nitrogen collect as liquid in the bottom of the helium flash drum, passing out through the conduit **327** under control of the valve **332** which is regulated by the device **332***a*, to maintain the liquid in the vessel at a substantially preselected level. The flow rate, temperature, pressure, and analysis of this stream is the same as that indicated in Table 25 for the circuit of FIG. 3D. Similarly, the condensate stream, comprising primarily liquid nitrogen, flows in through the inlet **333** to the helium stripper **334.** The latter, which corresponds to helium stripper **305** of FIG. 3D, contains a bayonet heater **336,** connected to a source of power **340***a*, for reboiling the liquid condensate. As in the helium stripper of FIG. 2D, the body portion of the vessel **334** is packed with stainless steel packing **335.**

Also, as previously described, vapor comprising the low boiling components, including about 30 mol percent of helium, and the balance nitrogen with a small amount of hydrogen, rises to the top of vessel **334,** passing out through the conduit **338** in the dome, flowing at a pressure of about **20** pounds per square inch, absolute. The temperature, flow rate, and composition of this helium stripper vapor are the same as set forth in Table 26 with reference to the previous figure. This stream flows back into column *b* of heat exchanger **318** where it is heated up to a temperature of 32.8°F in case A and 94°F in case B. It is then compressed to about 1,200 pounds per square inch absolute, the compressor heat being compensated for in the after cooler **342.** The compressed stream passes into the junction point **316,** where it is recycled through the system.

As in FIG. 3D, the condensate liquid, comprising primarily nitrogen collecting in the bottom of the vessel **334,** passes out through the conduit **337** through valve **340***b* under control of liquid level device **340***c*. The composition, temperature, pressure, and flow rate are substantially as indicated with reference to that figure, and in Table 27. This stream flows into the outer shell of heat exchanger **318** where it serves as a refrigerant, in a countercurrent heat exchange with the cooling feed streams; after evaporation, the condensate is heated up to 19°F in case A and 94°F in case B. As previously indicated, this stream, which is about 97 mol percent nitrogen, may be passed out to the atmosphere at atmospheric pressure, or through valve **17***b* for recycle. In accordance with another alternative, it may be used in the final purification stage of the system.

As previously indicated, each one of the three helium enrichment units indicated in FIGS. 3A and 3B, combined as FIGS. 3C, 3D and 3E, may be employed as a separate entity; or, they may be employed in three alternative arrangements, each serving as the intermediate unit between the crude separation unit **100** described with reference to FIG. **2,** and the final helium purification unit **400** to be described with reference to FIGS. 4A, 4B and 4C.

Assuming one of the helium enrichment circuits of FIGS. 3A and 3B, combined as FIGS. 3C, 3D or 3E, is

connected as part of the greater helium purification system, as previously indicated in FIG. **1**, the helium-enriched stream passes out of the enrichment circuit **200** through the conduit **19** to the junction **21** where a proportion of the flow may be returned through the conduit **22** to the conservation pipe line **12***b* for storage at an absolute pressure of about 800 pounds per square inch. The remaining component of the stream then passes through conduit **24** to the junction **25**, where it may be joined by a stream of enriched helium from an off-site source derived through the conduit **26** under control of the valve **28.** The enriched helium stream may flow in, for example, at a pressure of 863 pounds per square inch, absolute, having a purity below that of the helium stream derived from the enriching unit through conduit **24.**

The combined stream, having a purity within the range 87 to 97 mol percent, then flows into the final stage, comprising the helium purification system **400.**

The relationship of the input streams into the junction **25** may be adjusted in order to provide a stream flowing in the conduit **29** of the desired analysis, temperature, pressure, and flow rate. For the purposes of this description, the input stream flowing into the system **400** through the conduit **29** will be assumed to have a pressure of 863 pounds per square inch, absolute, and a temperature of 80°F. The flow rate and analysis of the composition of the feed stream is as shown below in Table 28.

Table 28

Feed to Final Purification Stage

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	866.6	790.8
Composition (Mol %)		
Hydrogen	1.00	1.2017
Helium	87.78	96.1943
Nitrogen	10.92	2.5900
Methane	0.30	0.014

This feed stream flows through valve **29***a* and junction **525**, FIG. 4A, where it may be joined by a purge stream returning through conduit **524**, if valve **527** is open, as will be explained hereinafter. From junction **525**, the stream flows into the outer chamber of standard shell and tube-type heat exchanger **402** where the temperature is raised to 225°F, passing into the conduit **402***a*, through which it flows to junction **403.** At this point, it passes through the normally open valve **405***a*. In the early phases of the system operation before heat exchanger **402** is operating in such a manner as to heat the feed stream to the desired temperature for the input of the dehydrogenator **404**, valve **405***a* is closed; and, the stream from **402***a* is diverted through valve **405***b* and heater **405***c*. The latter is fueled by fuel gas, such as may be derived from the fuel gas system **46** (see FIG. **1**), which receives a by-product of the crude helium unit. Fuel gas valve **405***d* to heater **405**c is operated pneumatically (or electrically) by a temperature-sensitive probe **405***e* interposed in the output of the catalytic dehydrogenator **404.**

The stream from the heater or heater by-pass circuit then flows into junction **509**, where it is combined with a recycle stream passing in through conduit **511**, as will be explained hereinafter.

The combined feed and recycle stream then flows into the catalytic dehydrogenator **404.** This unit, which serves to remove most of the excess hydrogen in the stream by oxidation to water vapor, is of a type well-known in the art, which may be of the general form indicated, for example, in Rosenblatt U.S. Pat. No. 2,582,885, Jan. 15, 1952, in which the catalyst comprises pellets coated with palladium. Other catalysts suitable for this purpose include finely divided platinum. The oxygen to be combined with the hydrogen impurity is supplied from a stream of air passing into the feed stream through conduit **511** in a manner to be described hereinafter. A one mol percent hydrogen content in the feed stream causes a temperature rise of 200°F during the reaction in the dehydrogenator **404**, so that the effluent gas has a temperature approaching 425°F.

Although the dehydrogenator **404** converts most of the hydrogen to water vapor, a small excess of hydrogen remains in the stream which passes out through the conduit **404***a* and to the connected one of the twin hydrogenator adsorbers **406***a*, **406***b*, which operate in alternate shifts of 66 hours. These comprise beds of comminuted copper oxide and are well known in the art. The copper oxide beds in the hydrogenator adsorbers **406***a*, **406***b* require operating temperatures in excess of 390°F which are usually amply supplied by the operation in the catalytic dehydrogenator **404.** However, during the start-up operation, or in periods of low hydrogen concentration in the feed stream, this function is performed by heater **405***c*.

The traces of hydrogen remaining when the stream reaches connected hydrogen adsorber **406***a*, combine with the oxygen in the copper oxide, forming copper and additional water vapor. The gas stream, including the additional water vapor, passes out through the conduit **407***a* and into the junction **408.** Conduits **407***a* and **407***b* are in alternative operation, depending on which one of the hydrogen adsorbers **406***a* and **406***b* is in operation. Adsorber **406***b* is regenerated by flowing through the off-cycle vessel approximately 20 mol percent of the hydrogen-free feed gas from conduit **407***a* through junction **408**, junction **516** and conduit **407***b*, together with a stream of oxygen-bearing gas derived through flow-regulated valve **515***b*, in a manner to be described hereinafter. The oxygen in this regenerative stream is held at a concentration of approximately 0.25 mol percent until the regeneration is satisfactory.

The hydrogen-free feed stream passes through the conduit **409** merging with the output from regenerating adsorber **406***b*, and through conduit **418** into the inner shell of heat exchanger **402**, which serves to lower the temperature of the dehydrogenated stream in a heat exchange with the incoming feed stream passing through conduit **401.** The stream then passes out of the inner shell of heat exchanger **402** into the conduit **411** which leads to the inner shell of heat exchanger **412** of a similar type, in which the stream is further cooled as it reaches the inlet **413** of the separator **414.** The latter is a conventional type centrifugal separator which removes from the stream a large proportion of the water vapor formed in the dehydrogenation process, which drains out as liquid through the conduit **414***a* at the bottom.

The partially purified stream then passes out through conduit **41**, junctions **415***a* and **421** into drier **416***a*, which is the on-cycle one of twin driers **416***a*, **416***b*,

comprising beds of molecular sieve which operate on alternate 8-hour shifts to adsorb the remaining moisture from the stream to a concentration of less than 1 part per million of water. The feed stream enters the on-cycle drier **416***a* at a temperature of 90°F, a pressure of 854 pounds per square inch, absolute, and at flow rates of 900.2 and 801.7 standard cubic feet per minute, in cases A and B, respectively. The driers **416***a* and **416***b* operate in alternation. The off-cycle drier **416***b* is connected for reactivation in an auxiliary circuit extending between junctions **415***a* and **421** in parallel with the on-cycle circuit through valve **415***b*. The valve **415***b* in conduit **415** is controlled pneumatically (or electrically) by circuit **415***c* to force flow in the auxiliary circuit. The auxiliary circuit passing through a second junction **420**, includes, in one branch, valve **420***a* leading to the input of fuel-fired heater **420***b*, which is energized, for example, by fuel gas derived from the fuel gas system **46** of the crude purification unit **100** (see FIG. 1). The flow of fuel gas into heater **420***b* is controlled by valve **420***d*, responsive to circuit **420***c* connected to a probe in the heater output. A bypass for heater **420**b is provided by conduit **420***e* under control of normally closed valve **420***f*.

During the reactivation process, the small stream of gas passes from junction **420***g* into the drier **416***b*, removing the adsorbed water vapor. From the output of drier **416***b*, the stream passes through a water cooled heat exchanger **417***a*, and a centrifugal separator **417***b*, for removing the moisture. The remainder of the stream returns to junction **421** to join the main stream through drier **416***a*.

The stream passing out of drier **416***a* flows through conduit **419** at a temperature of about 90°F into filter **422**, for removing solid particles from the stream, from which it flows through compressor **423** where the pressure is raised from **845** to about 2,760 pounds per square inch, absolute. The flow rate and analysis of the composition of this stream are as shown in Table 29, below.

Table 29

Compressed Feed Stream

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	899.4	801.0
Composition Hydrogen	Less than 1 ppm	Less than 1 ppm
(Mol %)		
Helium	85.3134	95.0715
Nitrogen	14.3960	4.9147
Methane	0.2906	0.0138

Preferably, compressor **423** is of a type well known in the art which avoids oil contamination of the compressed stream.

The output from the compressor **423** passes through the junction **424**, the main stream flowing into the after cooler **425**, of a conventional water cooled shell and tube type, which absorbs the heat generated in the stream by compressor **423**, leaving the stream at a temperature of about 90°F. A small component of the flow may be returned through conduit **425***b* and valve **425***c* under control of the pressure-controlled circuit **425***d* to the junction **425***e* at the input to the compressor, to stabilize the pressure differential across the compressor.

Output from the after-cooler **425** flows through conduit **426** under control of normally open valve **426***a* to junction **427**, and through conduit **428** to the three-way valve **429**, the flow in the output arms of which is controlled by the temperature-differential responsive circuit **429***a* having probes interposed in the respective input arms of junction **430** above. One component, which may, for example, be a little over 50 percent by volume, flows into column *a* of heat exchanger **432**; whereas, the balance passes into column *a* of heat exchanger **451**. The pressures of the emerging components, which recombine at junction **430**, is 2,742 pounds per square inch, absolute; and the temperature is maintained at −280°F.

The combined stream passes through conduit **433** into an inlet above the bottom of the first stage flash drum **434**. This is a cylindrical stainless steel vessel, bowed at the ends, 5 ½ feet high and ½ foot in diameter, and maintained at a pressure of 2,742 pounds per square inch, absolute. From this inlet the low boiling helium vapor rises to the top of the flash drum **434**, passing out through the conduit **434***a* in the dome and into three-way valve **435**; whereas any liquid sinks to the bottom of the vessel, passing out through conduit **436** under control of liquid-level controlled valve **436***a*, at a pressure of 2,742 pounds per square inch, absolute and at a temperature of −280°F. It will be noted that the presence of liquid condensate depends on the composition of the input to the final helium purification system, so that the presence of such liquid would be expected in case A, where the input is 87 mol percent pure helium, but not in case B, where the input is 96 mol percent pure helium. The further disposition of this liquid condensate, largely nitrogen, as it passes through second-stage flash drum **439** is described hereinafter in a discussion of ancillary refrigeration cycles in unit **400**.

The vapor output from first-stage flash drum **434**, which passes out through the conduit **434***a* and into three-way valve **435**, is at a temperature of −280°F and a pressure of 2,742 pounds per square inch, absolute. The flow rate and analysis of the composition of this stream are as shown below in Table 30.

Table 30

Vapor from First-Stage Flash Drum

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	820	801
Composition (Mol %)		
Helium	92.9298	95.0717
Nitrogen	7.0454	4.9145
Methane	0.0248	0.0138

At three-way valve **435**, the stream again divides, part returning for a partial recool through column *d* of heat exchanger **432** through the conduit **448**, and the remainder passing through the conduit **449** and into column *c* for a partial recool through heat exchanger **451**. These two streams are recombined in junction **452**. The ratio of the streams in conduits **448** and **449** is determined by a temperature-sensitive control circuit which operates the respective right and left arms of valve **435** in accordance with indications of a dual temperature probe interposed into the branches connected

to junction **452.** At this point the combined stream has a pressure of **2739** pounds per square inch absolute, at a temperature of −315°F in case A, and −306°F in case B, and respective flow rates of 820 and 801 standard cubic feet per minute. The composition remains substantially the same as previously indicated.

The combined stream from junction **452** flows through the conduit **453** to inner tube *a* of the nitrogen boiler **454,** where, in a heat exchange with boiling liquid nitrogen, the temperature of the stream is further reduced and partially liquefied. The cooled partially liquid output stream then passes into the conduit **455** which leads into the secondstage flash drum **456.** The latter is a cylindrical stainless steel drum with bowed ends, 5 feet high and 0.5 feet in diameter. The vessel **456** is maintained at an internal pressure of about 2,730 pounds per square inch, absolute. The vapor, comprising the nearly purified helium, rises to the top, passing out through the conduit **457** in the dome; and, the liquid, primarily nitrogen, condenses in the bottom, and passes out in a course which will be described hereinafter with reference to the refrigeration cycles. After the flashing operation in the vessel **456,** the vapor stream passing out through the dome has a pressure of 2,732 pounds per square inch absolute, a temperature of −315°F, and the composition and flow rates of this stream are as shown below in Table 31.

Table 31

Vapor from Second-Stage Flash Drum

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	771.9	771.9
Composition (Mol %)		
Helium	98.5470	98.5464
Nitrogen	1.4522	1.4529
Methane	0.0008	0.0007

The helium stream from the dome of second-stage flash drum **456** passes into column *a* of heat exchanger **461** where the temperature is further reduced to −332.9°F and −333.8°F in cases A and B, respectively. The stream flows out of heat exchanger **461** through the conduit **462** and into the inner tube *a* of nitrogen boiler **463,** in which the temperature is further reduced to −340°F, at a pressure of 2,724 pounds per square inch, absolute, which is maintained at 2.3 pounds per square inch absolute. The cooled stream then passes through the conduit **464** and into the third-stage flash drum **465.** The latter is a flash vessel of a form similar to those previously described, being 5 feet high and ½ foot in diameter, and maintained at an internal pressure of 2,724 pounds per square inch absolute. This vessel includes a wire mesh **466** in the dome, which is say, 4 inches thick and serves to entrap liquid globules from the rising vapor, the entrapped liquid falling down to the bottom of the vessel, and the vapor passing out through the conduit **467** in the dome. The flash gas passing out has a pressure of 2,724 pounds per square inch absolute, a temperature of −340°F, and a flow rate of 767.4 standard cubic feet per minute. The analysis of the composition of this gas is as shown below in Table 32.

Table 32

Vapor from Third-Stage Flash Drum

Composition (Mol %)	Case A	Case B
Helium	99.1235	99.1234
Nitrogen	0.8763	0.8764
Methane	0.0002	0.0002

The flash vapor passes through column *b* of heat exchanger **461,** where it is slightly reheated to −320°F, passing to the junction **468,** at the intersection of conduits **470** and **472.**

Under normal conditions the stream then passes through conduit **472,** valve **471** and junction **489** from which it passes to column *b* of nitrogen boiler **454,** passing through conduit **473** at a temperature of −315°F into junction **474,** between open valve **475***a* to on-cycle adsorber **478***a*, and closed valve **475***b* to off-cycle adsorber **478***b* undergoing regeneration. It passes through valve **475***a* into the conduit **476***a* at the terminal of valves **475***c* and **475***c*, whose functions will be described hereinafter, through the solid particle filter **477***a*, and into the lower end of nitrogen adsorber **478***a* through intake **84**a. The nearly purified helium stream in the adsorber feed has a pressure of 2,712 pounds per square inch, absolute, a temperature of −315°F, and a flow rate of 767.4 standard cubic feet per minute, assuming none of the flow has been diverted to off-cycle adsorber **478***b*. The composition of this stream is as shown below in Table 33.

Table 33

Nitrogen Adsorber Feed

Composition (Mol %)	Case A	Case B
Helium	99.1235	99.1234
Nitrogen	0.8763	0.8764
Methane	0.0002	0.0002

The substantially identical nitrogen adsorbers **478***a* and **478***b*, typical construction of **478***a*, only, being shown in longitudinal section in FIG. **4E**, each contain 70.1 cubic feet of finely divided carbon in insulated containers which are cooled with liquid nitrogen. The function of these adsorbers is to remove substantially all of the remaining impurity content from the helium stream. They operate in alternation on a 24-hour cycle of adsorption, followed by a 24 hour period for reactivation. During the adsorbing period, the carbon bed is cooled with liquid nitrogen to remove the heat of adsorption. Reactivation of the beds is conducted in a manner which will be described hereinafter.

Each of the adsorbers **478***a* and **478***b* is formed of a shell which may be, for example, of SA–516–70 or equivalent grade steel. This outer shell **78***a*, **78***b*, is cylindrical, with bowed ends, 18½ feet high and 44½ inches in outer diameter. The steel walls are 2½ inches thick. Disposed symmetrically inside of the outer vessel **78***a*, **78***b* is an inner cylindrical stainless steel shield **79***a*, **79***b* which is three-sixteenths of an inch thick, the outer vessel and the shield being spaced apart 6 inches. In this intervening space is interposed insulation **80***a*, **80***b*, of unbonded fiber comprising, for example, glass fiber having a density of about 4.5 pounds per cubic

foot. The comminuted carbon bed **81***a*, **81***b*, is contained within the respective inner cylindrical stainless steel shield **79***a*, **79***b*. In this inner vessel is wound, at a 3 inch pitch, a refrigeration coil **460***a*, **460***b* having a ½ inch outer diameter and 0.095 inch wall thickness. The coil **460***a* has an intake connection **82***a* and an exhaust connection **83***a*, connectable to the auxiliary nitrogen system **45** for refrigeration, as will be explained hereinafter. Coil **460***b* has corresponding connections to the auxiliary nitrogen system **45.** Connections **84***a* and **85***a* are respectively connected for intake and exhaust of the low temperature product during the on-cycle period.

When the vessel **478***a* is connected for regeneration, in the manner in vessel **478***b*, as shown in FIG. **4**C, connections **84***a* and **85***a* serve as intake and exhaust connections to the carbon bed during the purging process. The vessel is designed to withstand a pressure of 3,035 pounds per square inch, absolute.

As presently shown, the adsorber **478***a* is connected into the operating system to adsorb the remaining traces of nitrogen from the helium product under purification; whereas the nitrogen adsorber **478***b*, as indicated in the drawing, is connected for reactivation. During the period of adsorption, the vessel **478***a* is maintained at a pressure of 2,745 pounds per square inch, absolute, and at a temperature of −315°F by liquid nitrogen flowing in the coil **460***a*.

The helium gaseous product, at a pressure of 2,707 pounds per square inch absolute and a temperature of −315°F, purified of the remaining trace amounts of nitrogen and other impurities, passes out of adsorber **478***a* through the exhaust connection **85***a*, solid particle filter **480***a*, conduit **479***a*, and open valve **482***e*, to junction **481,** one branch of which is connected to the closed valve **482***f*, leading from the circuit of reactivating adsorber **478***b*. The purified helium stream passes into the conduit **483,** which is in turn connected to three-way pneumatically operated valve **484.** One arm of the latter is connected to the conduit **485** leading through coil *b* of heat exchanger **451** and conduit **493** to junction **496.** The other arm of valve **484** is connected through conduit **494,** to coil *c* of heat exchanger **432,** and conduit **495** to junction **496.** Valve **484** is pneumatically actuated by flow-differential responsive probes in heat exchanger output conduits **493** and **495** to keep the flows in each of these branches substantially equal at 380.35 standard cubic feet per minute in each case. The two product streams are combined in junction **496** to form a purified helium output stream which passes out through conduit **35** at a temperature of 80°F, a pressure of 2,700 pounds per square inch absolute, and a flow rate of 760.7 standard cubic feet per minute, in the storage tank **600,** or directly to commercial use. The analysis of the purified product is helium at least 99.9969 mol percent, containing less than 30 parts per million of hydrogen, nitrogen, hydrocarbon impurities.

Let us return, now, to the reactivation of the off-cycle nitrogen adsorber **478***b*, which is similar in construction to the on-cycle coil **478***a*, and contains a cooling coil **460***b*, corresponding to cooling coil **460***a*. The other components are similarly corresponding.

Reactivation of the carbon bed of the nitrogen adsorber **478***b*, which has operated over a 24-hour period to adsorb the remaining impurities from the helium product stream including about 0.8763 mol percent nitrogen and 0.0002 mol percent methane, is carried out in the following steps. Liquid nitrogen from the auxiliary nitrogen circuit, which flows in the coil **460***b* at a temperature of −315°F during its period of active adsorption, is stopped by closing valve **82***b*. Valve **82**a has been previously opened so that coil **460***a* contains flowing liquid nitrogen. In synchronism with this operation, valve **475***b* is closed, and valve **475***a* is opened, diverting the nearly pure helium stream from junction **474** away from adsorber **478***b* and into **478***a*.

During the regeneration period, a component of the compressed feed stream is diverted from junction **424** at the output of compressor **423,** through conduit **534** and junction **537.** Assuming valve **482***a* to be closed, and valve **482***b* to be open for this purpose, the warm stream passes into the top of carbon adsorber bed **478***b* through particle filter **480***b* and inlet pipe **85***b*. The temperature, pressure, and flow rate of this intermittent stream are indicated in Table 34 below.

Table 34

Intermittent Stream for Warming Off-Cycle Adsorber

	Case A	Case B
Temperature (°F)	250	250
Pressure (Pounds per square inch, absolute)	2760	2760
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	153	153
Composition (Mol %)		
Helium	85.3134	95.0715
Nitrogen	14.3960	4.9147
Methane	0.2906	0.0138

This stream serves to warm up the off-cycle adsorber **478***b* so that gases adsorbed by the carbon bed during the on-cycle period are vented into the recycle stream.

During the reactivation process, the pressure within the carbon bed is maintained at about 2,760 pounds per square inch, absolute. The stream, including desorbed gases from the carbon bed of off-cycle nitrogen adsorber **478***b*, is allowed to vent through the outlet **84***b*, filter **477***b*, valve **475***f*, and junction **521** at the bottom of the vessel, valve **475***e* being closed during this process. The stream of vented gas, comprising 85 to 96 mol percent of helium, 4 to 15 mol percent of nitrogen, and a small amount of methane, flows through conduit **557,** heat exchanger **542,** and conduit **539** to junction **522,** at a pressure of about 2,757 pounds per square inch, absolute, a flow rate of 64.5 standard cubic feet per minute, and a temperature ranging anywhere from −300°F to +210°F, depending on the progress of the desorption process.

At junction **522,** assuming valve **527** to be closed, the stream, including the desorbed gases, flows into junction **427** where it joins the compressed feed stream passing from valve **426**a through conduit **428,** into the three-way valve **429.** If the returning vent stream contains hydrogen, of which it is desired to purge the stream before recycle, valve **527** is opened, permitting the stream to flow through junction **525** and conduit **401,** where it joins the fresh feed in heat exchanger **402** for passage through the catalytic dehydrogenator **404,** as previously described. A check valve automatically prevents flow from junction **427** into junction **522.**

It will be noted that all of the valves, **475***a* through **475***f*, at the bottom end of the nitrogen adsorber sys-

tem, and **482***a* through **482***f*, at the top end of the nitrogen adsorber system, may be so arranged that they are opened and closed automatically by well-known electronic or pneumatic mechanisms.

Near the end of the reactivation cycle when the temperature of the purged gases has risen to about 210°F, nearly pure helium at a temperature of −315°F is introduced into the bed of the vessel **478**b in the amount needed to displace one void bed volume. This nearly pure stream is derived through a path leading back from junction **468** and junction **469** through conduit **470,** valve **482***c* being closed, valve **471** partially closed, and valve **482***d* being open, so that some of the product stream flows into **478***b* through intake **85***b*. During this period, valve **482***b* is closed.

When the displaced gases from vessel **478***b* reach nearly pure helium specifications, as indicated by conventional analyzing means, valve **475***f* to junction **521** is closed. Valve **82***b* is opened, supplying liquid nitrogen from the auxiliary nitrogen refrigeration circuit, to be described hereinafter, to coil **460***b*. The flow of near pure helium to **478***b* is continued in this fashion until the temperature of the adsorber reaches −315°F as indicated by the temperature of the gases at outlet **84***b*. At this time valves **475***d* and **482**d are closed and valve **475***b* opened keeping adsorber **478***b* in stand-by condition for the next adsorption cycle.

During the crossover period from vessel **478** a to vessel **478***b* in the on-cycle connection, valves **82***a* and **82***b*, **475***a* **475***b*, and **482***a* and **482***b* can all be operated synchronously, permitting the nitrogen refrigeration stream to divide, cooling both of the adsorbers **478***a* and **478***b* simultaneously, and at the same time permitting the helium stream from junction **474** to divide between the adsorbers, and the divided components of purified product to join at junction **481** for flow into the output.

In any case, the product from junction **481,** which is practically 100 percent pure helium, flows at a pressure of 2,707 pounds per square inch, absolute, a temperature of −315°F, and a flow rate of 760.7 standard cubic feet per minute, through conduit **483** to the three-way valve **484,** and to the output in conduit **35** by a path previously described.

It will be noted that the helium purification operation described makes efficient use of the condensate from the flash drums for refrigeration purposes. Some of the ancillary and refrigeration circuits of the final purification operation will now be described.

In case A, the liquid stream of condensate sinking to the bottom of first stage flash drum **434** and passing out through conduit **436** has a pressure of 2,742 pounds per square inch, absolute, a temperature of −280°F. In case B, in which the input is of substantially higher puritiy, the condensate is practically nonexistent. The condensate stream is composed purity, shown below in Table 35.

Table 35

Condensate from First-State Flash Drum (Vessel 434)

	Case A	Case B
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	79.4	0
Composition (Mol %)		
Helium	6.6370	0
Nitrogen	90.3273	0
Methane	3.0357	0

This liquid stream then passes into conduit **436,** through the valve **436***a* to the inlet to fourth-stage flash drum **439.** The latter, which is maintained at an internal pressure of about 17.68 pounds per square inch, absolute, is a cylindrical stainless steel vessel with bowed ends, 2½ feet high and ½ foot in diameter. Valve **436***a* is electronically or pneumatically controlled by means well-known in the art, to maintain a preselected liquid level in the bottom of the firststage flash drum **434.**

The vapor, including helium and nitrogen, rises to the top of the vessel **439,** and passes out through the conduit **441** in the dome; and any liquid settles in the bottom of the vessel, passing out through the conduit **440.** The pressure of the emerging gas stream from second-stage flash drum **439** is about **17.68** pounds per square inch, absolute, at a temperature of −318°F, in case A, and −320°F, in case B, the flow being substantially zero in the latter case. The flow rate and composition are indicated in Table 36, below.

Table 36

Fourth-Stage Flash Vapor (Vessel 439)

	Case A	Case B
Flow Rate (Std. cubic feet/min.) [at 70°F, 1 atm. pressure]	22.1	0
Composition (Mol %)		
Helium	23.8699	0
Nitrogen	76.0673	0
Methane	0.0628	0

This stream flows through conduit **441** into the junction **442** where it is joined by the vapor output through conduit **443***a* from the helium stripper (fifth stage flash drum) **443,** as will be described hereinafter.

The combined flow from the junction **442** moves into the conduit **444.** This stream has a pressure slightly above atmospheric, about 17.68 pounds per square inch, absolute, a temperature of −319°F, and −324°F, in cases A and B respectively. It has a flow rate and composition as shown in Table 37 below.

Table 37

Combined Vapor from Stripper and Fourth-Stage Flash Drum

	Case A	Case B
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	25.3	1.9
Composition (Mol %)		
Helium	26.4068	46.1426
Nitrogen	73.5383	53.8547
Methane	0.0549	0.0027

This low pressure vapor stream passes back into the column *b* of heat exchanger **432,** where it also serves to cool down the incoming feed stream in a heat exchange in which the low pressure vapor is heated up to 80°F with a slight expansion to just below atmospheric

pressure. This stream then passes through the conduit **445** and pressure-controlled valve **446** past junction **500,** where, if valve **514** is open, it is combined with a stream of air flowing from air inlet **512***a* at 80+F and slightly below atmospheric pressure. Valve **514** may be pneumatically (or electrically) controlled in a manner well-known in the art, in response to an analysis of the incoming feed stream so that sufficient oxygen is provided to meet the stoichiometric requirement for removal of the hydrogen impurity in the feed stream as water vapor. The air passing from inlet **512***a* through conduit **510***a* has the usual composition, including about 77.10 mol percent of nitrogen and 20.50 mol percent of oxygen, in addition to 2.35 mol percent of water and about 0.05 mol percent of carbon dioxide. The heated vessel **513***a* through which the stream passes, contains an adsorbing bed comprising zeolites, or the like, for removing carbon dioxide to less than about 20 parts per million, and water vapor to less than about 5 parts per million. The adsorber **513***a* is operated on cycle, in alternation with adsorber **513***b*. The latter is shown connected for reactivation by a stream of waste nitrogen which may be derived from helium enrichment system **200** through conduit **17,** valve **16,** and conduit **15.** (See FIG. 1). Sufficient nitrogen is provided from enrichment system **200** for this purpose, provided there is sufficient nitrogen impurity in the feed stream being processed.

The stream flowing in conduit **15,** which comprises about 96.74 mol percent nitrogen and nearly 3 mol percent methane, flows at about ambient temperature and pressure through valve **545***a* controlled by pressure-actuated probe **545***b*, to junction **540.** It then passes through valve **530***a*, controlled by the flow-actuated probe **530***b*, and into the blower **528,** which forces the stream through adsorber **513***b*. The stream picks up the desorbed gases, and passes through conduit **510***b* to the vent **512***b*. Assuming insufficient nitrogen is provided from conduit **15,** valve **550***a* may be opened to provide waste nitrogen from conduit **37** through junction **550***b* for the reactivation process.

The combined recycle stream passes junction **501** and into compressor **502,** which is similar to compressor **423,** where it is compressed to 870 pounds per square inch, absolute. The pressure differential across compressor **502,** in series with after cooler **503,** is maintained substantially constant by a by-pass through valve **505***a* which is controlled by pressure-indicator device **505***b*. The compressed stream is then cooled in after cooler **503** to 100°F, passing through conduit **515** into junction **507.** This stream has a flow rate and typical analysis as shown below in Table 38.

Table 38

Combined Recycle and Air Stream

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	45.9	24.5
Composition (Mol %)		
Helium	14.5269	3.5476
Nitrogen	75.9952	77.0652
Methane	0.0302	0.0002
Oxygen	9.4477	19.3870

This stream flows through junction **507** where a portion of it passes through flow controlled valve **515***b*, to junction **516** and conduit **407***b*, providing oxygen to regenerate the off-cycle hydrogen adsorber **406***b*, the outlet of which flows into feed stream conduit **409.** Another component flows through conduit **511,** junction **509,** to join the feed stream in conduit **410,** entering dehydrogenator **404** to provide oxygen for the catalytic dehydrogenation process.

Returning now to the refrigeration system, additional refrigerant is supplied by the condensates from second and third stage flash drums **456** and **465.** Liquid nitrogen, better than 98 mol percent pure, including small amounts of helium and methane residue, which collects in the second stage flesh drum **465,** is drawn off from the bottom, at the rate of about 4.5 standard cubic feet per minute, at a temperature of −340°F, and at a pressure of 2,724 pounds per square inch, absolute. This passes into conduit **588** through liquid-level-controlled valve **588***a* and into conduit **587.** At junction **589,** it is joined by the liquid stream from second-stage flash drum **456.** This comprises liquid nitrogen, better than 96 mol percent pure, including helium and methane impurity, drawn off from the bottom of flash drum **456,** which passes through conduit **458** and liquid level controlled valve **458***a*. This stream flows at the rate of 48.1 or 29.1 standard cubic feet per minute, in cases A and B respectively, at a pressure of 2,732 pounds per square inch, absolute and a temperature of −315°F. The merged stream then passes through conduit **590** to the inlet of fifth stage vessel, helium stripper **443.** The latter, which is maintained at an internal pressure of about **17.68** pounds per square inch, absolute, is a cylindrical aluminum vessel, bowed at top and bottom, which is 2½ feet high and ½ foot in diameter. The low boiling vapor, including helium stripped from the flashed liquid, passes through the dome outlet **443***a* to junction **442** where it merges with the vapor stream from conduit **441,** for recycle, as previously described.

The condensate falls to the bottom of helium stripper **443** (fifth stage), and passes out through the conduit **591** and valve **591***a* which is controlled to maintain the liquid in the stripper at a preselected level. This liquid passes out at a pressure of about 17.68 pounds per square inch, absolute, a temperature of −323°F, in case A, and −324°F in case B. The flow rate and analysis of the stream are as shown below in Table 39.

Table 39

Condensate from Fifth-State Flash Drum (Helium Stripper)

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	49.4	31.7
Composition (Mol %)		
Helium	0.0087	0.0094
Nitrogen	99.5833	99.6475
Methane	0.4080	0.3431

This liquid stream consisting of better than 99 mol percent pure nitrogen, passes through the valve **591***a* to the three-way valve **592.** The two arms of the latter respectively lead to conduit **592***a* passing to junction **447,** and conduit **431,** leading to outer shell *d* of heat exchanger **451.** Three-way valve **592** is pneumatically (or electronically) controlled by the relative amounts of the stream flowing in conduits **437** and **438.** In case B, when the output of condensate from the fourth stage flash vessel falls off to zero, nitrogen condensate from

helium stripper **443** may be diverted through conduit **592***a* in sufficient quantity to maintain the heat balance in exchanger **432.**

The stream passing to column *d* of heat exchanger **451** is heated up to 80°F in a heat exchange with the helium feed stream under purification and returning product. It then passes out through conduit **438** and junction **459** to nitrogen outlet conduit **41** where it may serve for refrigeration uses in other parts of the system, such as making up losses in nitrogen system **45,** cold colld boxes, etc.

The liquid residue from the first stage flash drum **434** passes out through the conduit **436** in the bottom and under control of liquid level controlled valve **436***a* at a pressure of 2,742 pounds per square inch, absolute and at a temperature of −280°F. This stream, which is operative only in case A, has a flow rate and composition as shown below in Table 40.

Table 40

Condensate from First Stage Flash Drum (Vessel 434)

	Case A	Case B
Flow Rate (Std. cubic ft./min.) [at 70°F, 1 atm. pressure]	79.4	0
Composition (Mol %)		
Helium	6.6370	0
Nitrogen	90.3273	0
Methane	3.0357	0

This passes through conduit **436** and the valve **436***a* to the inlet of the fourth stage flash drum **439,** where further separation takes place at an internal pressure of 17.68 pounds per square inch, absolute. Flash drum **439** is a cylindrical aluminum vessel, bowed at the ends, 2½ feet high and ½ foot in diameter. As previously discussed, the highly volatile elements, including remaining helium vapor rise to the top, and pass out through conduit **441,** where they merge with the vapor from helium stripper **443,** passing to recycle. The liquid falls to the bottom, and passes out through conduit **440** and liquid level controlled valve **440***a*. This stream, which flows out at a pressure of 17.68 pounds per square inch, absolute, and at a temperature of −318°F in case A, and −320°F in case B, has a flow rate and composition as shown below in Table 41.

Table 41

Condensate from Fourth Stage Flash Drum (Vessel 439)

	Case A	Case B
Flow Rate (Std. Cubic ft./min.) [at 70°F, 1 atm. pressure]	57.3	0
Composition (Mol %)		
Helium	0.0045	0
Nitrogen	95.8156	0
Methane	4.1799	0

This stream passes through junction **447** and conduit **446** to column **3** of heat exchanger **432,** where it is heated up to a temperature of 80°F in a heat exchange with the feed stream, returning recycle, and product streams, passing out through outlet conduit **437** to junction **540.** This is connected through conduit **37** to atmospheric vent **37***a* through a pressure controlled valve **37***b*. Junction **540** is also connected through valve **594***a* to junction **594** to nitrogen line **41,** returning to the nitrogen supply system **45,** and the refrigeration systems in the other units.

In addition to the foregoing circuits, which make use of the impurity condensates and waste streams to supply refrigeration to the system, refrigeration is also furnished by a supplemental system which derives nitrogen from an off-site source **45,** or alternatively, from nitrogen generated in the earlier stages of the system and processed to the desired purity and pressure.

This supplemental nitrogen refrigeration system will now be described. A stream of practically 100 percent pure nitrogen, for this purpose, is derived from the nitrogen source **45** at a pressure of 612.68 pounds per square inch absolute, at a temperature of 90°F, and a flow rate of 644.7 standard cubic feet per minute in both cases A and B. The gaseous nitrogen from source **45** passes directly into the system through conduit **33,** past junction **546,** to which is connected normally closed valve **546***a* leading to the waste nitrogen outlet **41.**

The high pressure nitrogen stream passes through conduit **547** at the flow rate indicated in the preceding paragraph, into column *b* of heat exchanger **556,** where it is divided into two components. The major component, flowing at the rate of between 473.8 and 509.3 standard cubic feet per minute, is cooled to −170°F, and passes out of the heat exchanger into conduit **549** after a partial pass. This component passes into the expansion turbine **554,** where it is work-expanded to just above atmospheric pressure, and a temperature of −310°F. This component then passes out through conduit **552** and junction **553,** where it joins the vapor stream returning to column *a* of heat exchanger **556,** through conduit **555,** the cold low-pressure stream serving as refrigerant in the heat exchanger, where it is heated up to 85°F. This combined stream, at just above atmospheric pressure, then returns through conduit **558** to junction **559** at the output of nitrogen vacuum pump **502,** for return through conduit **560** to nitrogen source **45.**

The minor component from heat exchanger **556,** consisting of the remainder of the flow in column *b*, passes through the heat exchanger, and is cooled to −275°F with a slight pressure drop to 598 pounds per square inch, absolute. It then flows through pressure-controlled Joule-Thompson expansion valve **564** where it is expanded to a pressure of 26 pounds per square inch, absolute, and cooled to a temperature of −310°F, partially liquefying. The liquid and vapor phases are separated in nitrogen flash and storage vessel **566.** The vapor flows out through the top of vessel **566** at a pressure of 26 pounds per square inch, absolute, and a temperature of −310°F, through pressure-controlled valve **575***a* and into junction **572,** where the stream joins up with the vapor flow from conduit **571** from the nitrogen-boiler **454,** and from conduit **520,** returning from coils **460***a*, **460***b* of the carbon adsorbers **478***a* and **478***b*. These merged streams, having a flow rate of between 156.5 and 269.5 standard cubic feet per minute, pass through conduit **557** to junction **553** for return through conduit **555** and heat exchanger **556,** as previously described.

The liquid nitrogen, which is stored in the bottom of vessel **566,** passes out through conduit **567** at a flow rate between 131.4 and 230.4 standard cubic feet per minute to junction **567***a*, where the stream of liquid divides.

The major liquid component, flowing at the rate of between 93.4 and 170.1 standard cubic feet per minute, flows through conduit **583** to junction **593** between inlets **82***a* and **82***b* of the respective refrigeration coils **460***a* and **460***b* of the carbon adsorbers. The liquid then flows through one or both of coils **460***a* and **460***b*, depending on which of the carbon adsorbers **478***a* or **478***b* is on-cycle at the time, the vaporized stream returning through junction **519** and conduit **520**, as previously described.

The minor component from junction **567***a* below nitrogen flash vessel **566**, consisting of the remainder of the flow, passes through conduit **576** to junction **568**, where it again divides, the major component, between 23.6 and 45.9 standard cubic feet per minute flowing through valve **578** to the outer shell of nitrogen boiler **454**, where it is evaporated in a heat exchange with the feed stream in column *a*. The vapor passes out at just above atmospheric pressure, at a temperature of −319°F, returning through conduit **571** to junction **572** as previously described.

The remaining nitrogen liquid component from junction **568** passes through valve **579** to the outer shell of nitrogen boiler **463**, where it evaporates in a heat exchange with a cooling stream in column *a*. The nitrogen vapor passes out at below atmospheric pressure, at a temperature of −343°F, returning through conduit **581** and column *c* of heat exchanger **556**, where it is warmed up to 0°F. This stream then passes out of the heat exchanger through conduit **582** to nitrogen vacuum pump **502**. If desired, this stream may be merged at the compressor input with part of the stream from conduit **558**, passing through conduit **550** and valve **550***a* to maintain the desired pressure at the compressor input. The compressed stream passes out through conduit **561** to junction **559** for return through conduit **560** to the nitrogen source **45**. Conduit **560** is connected to nitrogen return conduit **41** through conduit **594** and the normally closed valve **594***a*, so that the returning nitrogen vapor through conduit **41** can be diverted to nitrogen source **45**, if desired; or, alternatively, returning nitrogen in conduit **560** can be shifted for refrigeration and purging of other stages of the system.

Start-up Procedure

To bring the crude separation unit **100**, of FIG. **2**, into operation from ambient conditions, a procedure combining Joule-Thompson expansion and recompression is employed in a recycle operation. Such start-up facilities are normally incorporated in the design of a plant. In the case of unit **100**, these facilities operate to permit the return of off-specification crude helium, after Joule-Thompson expansion, to junction **164**. Residue gas can be returned to junction **164** by opening valves **161** and **166**, while closing valves **162** and **167**. Thus, off-specification residue gas and off-specification crude helium product can be returned to compressor **137**, and the crude separation unit placed in total recycle. Refrigeration for the unit is gained by removing the heat of recompression of the expanded gases by means of water-cooled heat exchangers **138** and **142**.

Similarly, for the case of each of the enriching units **200**, indicated in FIG. **3C** and in FIGS. **3D** and **3E**, a total recycle separation is employed to bring the unit from ambient conditions to the desired condition of temperature and composition. For example, in the preferred unit shown in FIG. **3C**, off-specification waste nitrogen can be returned for recompression through valve **17***b*, and conduit **230** to junction **272**. Start-up facilities will provide a means for returning off-specification product after Joule-Thompson expansion to junction **272** for recompression. Such a total recycle operation is continued until product specifications are met. Refrigeration is gained by removing the heat of compression by means of after-cooler **278** and by work expansion in expander **239**. Similar start-up procedures are employed in the alternate systems shown in FIGS. **3D** and **3E**.

The helium purification unit **400**, disclosed in FIG. **4D**, is also brought on-stream from ambient conditions by a total recycle operation. Start-up facilities are provided so that helium which does not meet Grade A specifications can be returned to junction **525** for reprocessing. Such recycling is continued until the desired product specifications are reached. Refrigeration during start-up is supplied by liquid nitrogen from vessel **566** and augmented by the use of a recycled product which has been expanded through a Joule-Thompson expansion step and recompressed.

This invention is not confined to the particular construction and arrangement of parts herein illustrated and described, but embraces all such modified forms as may come within a scope of the following claims.

We claim:

1. A process comprising three successive operations for producing grade A purity helium from natural gas:

in a first of said operations, deriving crude helium from said natural gas by a plurality of steps comprising:

compressing a feed stream of said natural gas to about 30 atmospheres pressure,

cooling said compressed feed gas stream to below the condensation point of the major portion of hydrocarbons in said feed stream by heat exchange with returning crude product and condensate streams,

separating out one or more condensed hydrocarbons from said gas feed stream as a by-product and utilizing said condensed hydrocarbons for cooling said feed gas stream,

subcooling the remaining feed gas stream to at least about the condensation point of nitrogen in a further heat exchange with returning crude vapor product and condensate streams,

stripping the subcooled feed stream of helium by flashing in a plurality of successive stages at progressively lower pressures to produce crude helium product vapor from the first of said flash stages and partially helium vapor from the remainder of said flash stages,

separating out a stream of condensed hydrocarbons from the last of said successive stripping stages,

returning said crude helium product vapor through said heat exchange steps for warming said crude helium vapor, and cooling said compressed feed gas stream,

recycling the partially helium vapor obtained from the remainder of said flashing stages, and

vaporizing and utilizing the said streams of hydrocarbon condensate from said flashing and stripping stages to provide refrigeration for said heat exchange steps;

in a second of said operations, enriching the product of the foregoing crude helium operation by the steps of:

compressing a feed stream comprising said crude helium vapor product to about 80 atmospheres pressure,

cooling said feed stream in heat exchange with enriched helium vapor product, liquid condensate, and recycle streams to at least about the condensation temperature of nitrogen,

directing said feed stream through at least one flashing stage for separating out enriched helium vapor from liquid condensate,

work-expanding the said enriched helium vapor from said one flashing stage,

directing the work-expanded enriched helium vapor through at least one heat exchange step with said feed stream to provide an enriched helium product stream at a temperature above 0°F,

directing the liquid condensate from said one flashing stage through at least one additional flashing stage for stripping the helium from said condensate,

directing the vapor product of said additional flashing stage through at least one heat exchange step for cooling said feed stream,

subsequently recompressing said last-named vapor product for recycling with said feed stream, and

directing the liquid condensate from said additional flashing stage through at least one heat exchange step for cooling said feed stream;

in the final one of said operations purifying a feed stream comprising said enriched helium vapor product to helium of grade A purity by a series of steps which comprise:

deriving said enriched helium feed stream compressed to about 60 atmospheres pressure,

heating said enriched helium feed stream to a temperature substantially above room temperature by heat exchange with the partially dehydrogenated feed stream,

dehydrogenating said enriched helium feed stream by removing the major portion of hydrogen from said stream by oxidation in the presence of a catalyst to produce water vapor, and removing excess hydrogen from said partly dehydrogenated stream by an additional chemical step,

drying said dehydrogenated stream of said water vapor,

further compressing said dehydrogenated stream to about 185 atmospheres pressure,

cooling said compressed dehydrogenated stream to below the condensation temperature of liquid nitrogen,

alternately flashing and further cooling said compressed dehydrogenated helium stream through a plurality of flashing and cooling stages in heat exchange with boiling liquid nitrogen for removing nitrogen and hydrocarbon impurities from said stream,

passing said helium stream after said cooling and flashing steps through an adsorbing step in a carbon bed adsorber for removing residual nitrogen and other impurities from said stream, and

heat exchanging the purified helium product resulting from said adsorbing step with said enriched helium feed stream to warm said product to about ambient temperature.

2. A process in accordance with claim **1** comprising three successive operations wherein:

said natural gas contains between about 15 and 40 mol percent of helium,

said crude helium product contains between about 60 and 80 mol percent of helium,

said enriched helium product contains between about 85 and 97 mol percent of helium, and

said grade A helium product has an impurity content which is less than about 30 parts per million.

3. The process in accordance with claim **1** wherein in the second of said operations, the enriched helium vapor from said one flashing stage is passed through a reheating step prior to said work-expansion step for warming up said vapor to a temperature exceeding the condensation temperature of the components of said vapor by more than the expected temperature reduction during said work-expansion step whereby no liquid is formed in said vapor during said work-expansion step.

4. A process in accordance with claim **1** wherein in the second of said operations said cooling is carried out in two steps:

the first said step comprising a heat exchange with multiple streams, including returning enriched helium product and recycle streams, and

the second said step comprising a subcooling step in a heat exchange with only the work-expanded enriched helium product stream.

5. A process in accordance with the first of said operations set forth in claim **1** wherein said by-product condensed hydrocarbons are separated out as one or more streams employed to cool said feed gas stream, each of which hydrocarbon streams may optionally be returned for recycling with said partially helium vapor, wherein said by-product condensed hydrocarbons consist of commercial grade fuel having a heating capacity of between 850 and 950 British Thermal Units per cubic foot.

6. A process in accordance with claim **1** wherein refrigeration for the cooling steps in each of said operations is furnished entirely by condensate from the flashing stages and from work expansion in the said operation, with the exception of the final said operation wherein supplementary refrigeration for the cooling steps including refrigeration of said carbon desorbing means is furnished from an auxiliary nitrogen source.

7. A process in accordance with claim **1** wherein condensate comprising waste nitrogen from said first and second operations is optionally returned with the recycle stream in each of said operations.

8. A process in accordance with claim **6** wherein the liquid condensate from the additional stage of said second operation and from the later stages of said third operation is nitrogen having a purity in excess of 96 mol percent; and wherein said nitrogen is employed for reactivating (airpurifying) adsorbers in the final one of said operations.

9. A process in accordance with claim **1** wherein in the final one of said operations said enriched helium feed stream is heated up to a temperature in excess of **200°F** prior to dehydrogenation in the presence of a

catalyst, the step of deriving fuel gas from the separated stream of condensed hydrocarbons in siad first operation for heating up said feed stream in the preliminary stages of said third operation during the initial period or when said feed stream is hydrogen deficient.

10. A process for deriving crude helium from natural gas which comprises the steps of:

compressing a feed stream comprising said natural gas to about 30 atmospheres pressure,

cooling said compressed feed gas stream to below the condensation point of a major portion of hydrocarbons in said feed stream by heat exchange with returning crude helium product and condensate streams,

separating out a first stream of condensed hydrocarbons from said feed stream in at least one flashing step and utilizing said condensed hydrocarbons for cooling said feed gas stream,

subcooling the vapor from said flashing step to at least about the condensation point of nitrogen in a further heat exchange with returning crude vapor product and condensate streams,

stripping the subcooled feed gas stream of helium by flashing in a plurality of successive stripping stages at progressively lower pressures to produce crude helium product vapor from the first of said flash stages and partially helium vapor from the remainder of said flash stages,

separating out a stream of condensed hydrocarbons from the last of said successive stripping stages,

returning said crude helium product vapor through said heat exchange steps for warming said crude helium vapor, and cooling said compressed feed gas stream,

returning and recycling the partially helium vapor obtained from the remainder of said flashing stages, and

vaporizing and utilizing the said streams of hydrocarbon condensate from said flashing and stripping stages to provide refrigeration for said heat exchange steps, said streams of returning crude helium, partial helium and hydrocarbon condensate providing adequate internal refrigeration for said crude helium derivation process without the use of an auxiliary refrigeration cycle.

11. A process in accordance with claim **10** for deriving a crude product containing between 60 and 80 mol percent of helium from natural gas containing between 15 and 40 mol percent of helium wherein:

said compressed feed stream is cooled to about −140°F by heat exchange with returning crude helium product and condensate streams,

said first stream of condensed hydrocarbons is separated from said feed stream by a pair of successive flashing steps, the first at about 30 atmospheres pressure and the second at about 13.5 atmospheres pressure, the said stream from said second flashing stage is utilized for cooling said feed gas stream,

the vapor from said first flashing step is subcooled to a temperature of −254°F in a further heat exchange with returning crude vapor product and condensate streams,

the subcooled feed gas stream is stripped of helium by flashing in a plurality of successive stages in which:

the first of said stages is at a pressure of **30** atmospheres,

the second of said stages which receives the liquid condensate from the first of said stages is at a pressure just in excess of 13 atmospheres,

the third of said stages which receives the condensate from the second of said stages is at a pressure of just in excess of 5 atmospheres, and

said second stream of condensed hydrocarbons is separated out from the last of said successive stripping stages at about 5.3 atmospheres pressure.

12. A process for enriching a crude helium feed stream which comprises the steps of:

compressing said feed stream of crude helium to about 80 atmospheres pressure,

cooling said feed stream in a heat exchange with enriched helium vapor product, liquid condensate, and recycle streams to at least about the condensation temperature of nitrogen,

directing the said feed stream through at least one flashing stage for separating out enriched helium vapor from liquid condensate,

work-expanding the said enriched helium vapor from said one flashing stage,

directing the work-expanded enriched helium vapor through at least one heat exchange step with said feed stream to provide an enriched helium product stream at a temperature above 0°F,

directing the condensate from said one flashing stage through at least one additional flashing stage for stripping the helium from said condensate,

directing the vapor product of said additional flashing stage through at least one heat exchange step for cooling said feed stream,

subsequently recompressing said last-named vapor product for recycling with said feed stream, and

utilizing the condensate from said additional flashing stage for refrigerating said feed stream.

13. A process in accordance with claim **12** wherein the enriched helium vapor from said one flashing step is passed through a reheat step prior to said work-expansion step for warming up the said vapor to a temperature exceeding the condensation temperature of the components of said vapor by more than the expected reduction in temperature in said workexpansion process, whereby no liquid is formed in said vapor during said work-expansion step.

14. A process in accordance with claim **12** wherein said cooling is carried out in two steps:

the first said step comprising a heat exchange with multiple streams including returning product and recycle streams, and

the second said step comprising a subcooling step in a heat exchange with only the work-expanded enriched helium product stream.

15. In a process for enriching a crude helium feed stream containing between about 60 and 80 mol percent of helium to an enriched product containing between 85 and 97 mol percent helium which comprises the steps of:

compressing said feed stream of crude helium to about 80 atmospheres pressure at near ambient temperature,

cooling said feed stream in a heat exchange with enriched helium vapor product, liquid condensate, and recycle streams to at least about the condensation temperature of nitrogen, and

directing said feed stream through one high pressure flashing stage for separating out enriched helium

vapor from liquid condensate, and directing the condensate from said high pressure flashing stage comprising nearly 96 mol percent nitrogen and small amounts of hydrogen and methane through at least one additional low pressure flashing operation including a reboiling operation for stripping helium from said condensate:

during said cooling step dividing said feed stream into two components, a major component comprising at least about 95 percent by volume of the flow, and a minor component comprising less than about 5 percent by volume of the flow,

cooling said major component in a heat exchange with returning product and waste streams to at least about the condensation temperature of nitrogen,

partially cooling said minor component in a heat exchange with a returning recycle stream, and subsequently further cooling said minor component in a heat exchange with evaporating liquid condensate in said reboiling operation in said additional flashing stage to at least about the condensation temperature of liquid nitrogen,

the two said cooled component streams being combined at about said last-named temperature and directed as a combined stream to said high pressure flashing stage in which said enriched helium vapor is separated out,

reheating said enriched helium vapor from said high pressure flashing stage to a temperature at least above the boiling point of the highest boiling component thereof in a heat exchange within the major component of said feed stream for substantially eliminating liquid therefrom,

subsequently work expanding said enriched helium vapor to about 60 atmospheres pressure at a temperature of about −320°F,

directing said work-expanded enriched helium vapor through at least one heat exchange with the major component of said feed stream in which said enriched vapor is warmed with a slight reduction in pressure to provide a product having a near ambient temperature,

directing the vapor product from said additional flashing operation, containing about 30 mol percent of helium, and a balance of nitrogen, hydrogen, and trace hydrocarbons through at least one heat exchange step with a component of said feed stream prior to passing for recompression and recycling in said system,

utilizing the condensate consisting of more than **96** mol percent nitrogen derived from said additional flashing operation for refrigerating said feed stream,

and optionally merging the said condensate with the vapor product from said additional flashing operation for recompression and recycling in said system.

16. The combination in accordance with claim **15** wherein the level of condensate in said reboiler is maintained at a preselected level by syphoning the said condensate from the bottom of the reboiler through a syphon providing a liquid head slightly above the said preselected liquid level in said reboiler; and wherein the pressure at the head of said syphon is regulated in accordance with the output vapor pressure from said reboiler.

17. A process for enriching a crude helium stream containing between 60 and 80 mol percent of helium to an enriched product containing between 85 and 97 mol percent of helium which comprises the steps of:

compressing said crude helium feed stream to about 80 atmospheres pressure at near ambient temperature,

cooling said crude helium feed stream in a heat exchange with returning enriched helium vapor product, liquid condensate and recycle streams to at least about the condensation temperature of nitrogen,

subsequently directing said cooled feed stream through a high pressure flashing stage where enriched helium vapor which consists of at least 95 mol percent of the helium is separated from liquid condensate,

reheating said vapor from said high pressure flashing stage to a temperature at least above the boiling point of the highest boiling component thereof,

turbo-expanding said reheated enriched helium vapor to about **60** atmospheres pressure,

heating said turbo-expanded enriched helium vapor in said heat exchange with said feed stream to provide an enriched helium product at near ambient temperature,

further flashing the liquid condensate comprising about 95 mol percent nitrogen derived from said high pressure flashing stage in a helium stripper at about 1½ atmospheres pressure for stripping the helium from said condensate, and passing the condensate from said further flashing stage consisting of about 97 mol percent nitrogen through heat exchange means for cooling said feed stream and out to the atmosphere at near ambient temperature,

passing the said stream of vapor derived from said helium stripper comprising about 30 mol percent helium in heat exchange for cooling said feed stream and warming said vapor to near ambient temperature at near atmospheric pressure, and

subsequently recompressing said last-named vapor product for recycling with said feed stream.

18. The process in accordance with claim **17** wherein said liquid condensate from said further flashing stage consisting of about 97 mol percent nitrogen is optionally combined with said stream of vapor derived from said helium stripper for recompression and recycling in said system.

19. The process in accordance with claim **17** wherein:

said feed stream is cooled to −**316**°F in at least two heat exchange steps,

the first said step is a heat exchange with returning product and recycle streams, and

the second said step is a heat exchange with an expanded returning product stream.

20. A process for preparing grade A helium from an enriched helium feed stream containing 85 to 95 mol percent of helium comprising in combination the following steps:

deriving said feed stream at about **60** atmospheres pressure and ambient temperature,

heating said enriched helium feed stream to above 220°F prior to partly dehydrogenating said feed stream together with a recycle stream including at most about 20 mol percent oxygen in the presence

of a catalyst to convert all but a small excess of the hydrogen in said stream to water vapor,
passing said partly dehydrogenated stream through a bed containing a reducing agent for removing the remaining hydrogen from said stream,
drying and compressing said dehydrogenated stream to 185 atmospheres,
separating said compressed stream into two components, each of said components being cooled by heat exchange with returning product and recycle streams to about −280°F,
alternately flashing and further cooling said compressed dehydrogenated helium stream through a plurality of flashing and cooling stages in heat exchange with boiling liquid nitrogen for condensing nitrogen and hydrocarbon impurities from said stream, said stages comprising:
passing said partially cooled feed stream through a first high pressure flashing stage,
deriving helium vapor between about 92 and 96 mol percent pure from said first flashing stage,
cooling said helium vapor from said first flashing stage to about −315°F in a heat exchange with returning vapor and product streams, and a further heat exchange in boiling liquid nitrogen in a first liquid nitrogen stage,
passing said cooled partially purified helium through a second high pressure flashing stage at about −315°F,
deriving from said second flashing stage a helium vapor having a purity in excess of 98 mol percent, containing less than about 2 mol percent nitrogen impurity,
cooling the helium vapor stream from said second flashing stage to a temperature of about −340°F in a heat exchange with reheating vapor from a third high pressure flashing stage and boiling liquid nitrogen in a second liquid nitrogen stage,
passing said stream through said third high pressure flashing stage at a temperature of −340°F,
deriving helium vapor from said third flashing stage which exceeds 99 mol percent purity containing less than one mol percent nitrogen,
reheating the purified vapor from said third flashing stage up to a temperature of about −320°F in a heat exchange with cooling vapor from said second flashing stage,
recooling said vapor with boiling liquid nitrogen in a repass through said first liquid nitrogen stage,
directing said reheated vapor at a pressure of at least about 82 atmospheres through an on-cycle one of a plurality of vessels comprising carbon adsorbing means, where trace amounts of hydrogen, nitrogen, and hydrocarbons are adsorbed, said adsorbing means assuming on-cycle and off-cycle connections in alternation,
deriving a product stream of nearly 100 mol percent pure helium from said carbon adsorbing means,
passing said product stream through a heat exchange with feed streams being cooled wherein said product stream is warmed up to a temperature of about 80°F at a pressure of about 182 atmospheres,
deriving condensates from said first high pressure flashing stage and flashing said condensates through a fourth flashing stage at 1½ atmospheres pressure and −320°F, wherein the condensate from said fourth flashing stage includes in excess of 95 mol percent nitrogen,
passing the condensate from said fourth flashing stage in heat exchange with said feed stream for cooling said feed stream and vaporizing and heating said condensate up to about 80°F prior to venting said vaporized condensate,
deriving condensates from said second and third flashing stages containing in excess of 96 mol percent of nitrogen with more than 2 mol percent helium and less than 0.5 mol percent methane,
directing the condensates from said second and third flashing stages to a fifth stage flash vessel employed as a helium stripper maintained at a pressure of about 1½ atmospheres,
returning the vapor from said helium stripper containing in excess of 20 mol percent of helium through heat exchange with said feed stream for recycling through said system,
deriving the condensate from said helium stripper (fifth stage) including in excess of 99 mol percent of liquid nitrogen and less than 0.5 mol percent methane, and passing said condensate in heat exchange with feed streams undergoing cooling,
deriving nitrogen from an ancillary off-site source at a pressure of about 41 atmospheres and a temperature above ambient, and
liquefying and using said nitrogen stream for refrigerating the on-cycle one of said adsorbers, and for refrigerating the vapor streams from said first, second and third flashing stages.

21. The process in accordance with claim **20** wherein the method of purging the carbon bed of an off-cycle one of said adsorbing vessels of impurities adsorbed during said on-cycle connection which comprises:
deriving a small component of hot compressed feed stream prior to after cooling,
passing said component through the carbon bed of said off-cycle vessel, and
subsequently passing the purged gases into the stream under purification for at least a partial repass through said process for preparing grade A helium.

22. In the process in accordance with claim **20** providing refrigerant for the refrigeration coil of the on-cycle vessel of said plurality, and the coils of said boiling liquid nitrogen stages by the steps of:
deriving high pressure gaseous nitrogen from an off-cycle source at about 41 atmospheres pressure,
cooling the said high pressure nitrogen streams in two components in heat exchange with returning low pressure nitrogen streams,
the major said high pressure component being cooled in said heat exchange operation to about −170°F and subsequently work-expanded to a temperature of about −310°F at just above atmospheric pressure prior to returning in said heat exchange with said cooling high pressure streams,
the minor said high pressure component being cooled to about −275°F in said heat exchange with returning low pressure streams,
adiabatically expanding said cooled high pressure minor component to about atmospheric pressure and a temperature of about −310°F in a pressure-controlled valve,
subsequently flashing said cooled minor component to separate the liquid from the vapor,

the vapor from said flashing step returning in said heat exchange operation and merging with other returning low pressure streams,

and the liquid from said flashing step passing for evaporation in the refrigeration coils of said on-cycle carbon adsorber, and the coils of said boiling liquid nitrogen stages,

the resultant nitrogen vapor being returned for heat exchange with said high pressure nitrogen streams prior to being optionally returned to said off-cycle nitrogen source for refrigeration in other parts of said system.

23. A system for converting a feed stream comprising natural gas to grade A purity helium which comprises in combination:

a first unit for converting a feed stream comprising natural gas to crude helium which comprises in combination:

means for compressing said natural gas feed stream to about 30 atmospheres pressure,

a first heat exchanger for cooling said natural gas feed stream in a heat exchange with returning product and recycle streams and condensed liquid streams,

means for separating out the condensed hydrocarbons from said natural gas feed stream comprising a flash drum having a plurality of stages at progressively lower pressures,

a second heat exchanger connected to receive vapor from the first flash stage of said plurality of flashing stages for subcooling said vapor in a heat exchange with returning product and recycle streams,

means comprising a helium stripper having a first stage at high pressure, and a plurality of subsequent stages at progressively lower pressures for separating a major portion of the helium vapor from said subcooled vapor,

means for deriving crude helium product vapor from said first stage of said helium stripper and for returning said crude product vapor through said heat exchangers to means for transferring said crude helium product to the second unit of said system,

means for deriving recycle vapor including a substantial component of helium from said subsequent stages of said helium stripper and for returning said recycle vapor through said heat exchangers,

means for compressing said recycle vapor and returning said vapor to said feed stream,

means for deriving condensed hydrocarbons from said plurality of flashing stages and returning a stream of said condensed hydrocarbons through said first heat exchanger for refrigeration by vaporization, and to storage, and

means for deriving a stream comprising a high percentage of condensed hydrocarbons from the stages of said helium stripper and returning said stream through said first and second heat exchangers for refrigerating said heat exchangers;

a second unit for converting said feed stream comprising crude helium to enriched helium, which comprises in combination:

means for deriving a crude helium feed stream compressed to a pressure of about 80 atmospheres,

heat exchanger means for cooling said crude helium feed stream to a temperature below the condensation point of liquid nitrogen,

helium separation means connected to receive the cooled crude helium feed stream from said heat exchanger means for separating out enriched helium vapor from said feed stream,

work-expansion means connected to receive and workexpand the enriched helium vapor from said helium separator,

a system of conduits connected to receive the workexpanded enriched helium vapor from said work expansion means and to direct said enriched helium vapor product to return through said heat exchanger means for cooling said crude helium feed stream, and means for transferring said enriched helium product to the third unit of said system,

a conduit connected to the bottom of said helium separator for deriving liquid condensate therefrom,

auxiliary separation means connected to receive the liquid condensate from said helium separator,

means for deriving the partly helium vapor from said auxiliary separation means and returning said vapor through said heat exchanger means for cooling said crude helium feed stream,

compressing means connected to said heat exchanger means for compressing and returning said partly helium vapor to said crude helium feed stream for recycling in said system,

and means for deriving the primarily liquid condensate from said auxiliary separation means and for directing said condensate to pass through said heat exchanger means for evaporation therein to cool said crude helium feed stream; and

a third unit for converting a feed stream comprising enriched helium to helium of grade A purity, which comprises in combination:

means for deriving an enriched helium feed stream compressed to a pressure of about 60 atmospheres,

means comprising a first heat exchanger for heating said feed stream up to a temperature in excess of 220°F,

a source of a stream containing about 20 mol percent of oxygen for combining with said helium enriched feed stream,

means comprising a catalytic dehydrogenator connected to receive said enriched feed stream in combination with said oxygen-containing stream for converting a major portion of the hydrogen in said feed stream to water vapor,

means for receiving the output of said dehydrogenator comprising a chemical hydrogen adsorber,

said first heat exchanger connected to cool the output of said adsorber in a heat exchange with the enriched helium feed stream entering said dehydrogenator,

means for drying said dehydrogenated stream,

compressing means connected to compress said dried stream to about 185 atmospheres pressure,

means comprising an aftercooler connected to said compressing means,

a second and third heat exchanger connected in tandem to receive said compressed dehydrogenated enriched helium stream in two components and to cool said stream components to below the condensation temperature of liquid nitrogen in a heat exchange with returning purified helium product, recycle, and condensate streams,

flashing means comprising a first stage flash drum, for flashing the recombined components of said cooled enriched compressed dehydrogenated helium stream through a plurality of stages to separate out higher boiling liquid condensates from the helium containing vapor in said stream,

means for deriving the nearly purified helium vapor from said first stage flash drum and directing said helium vapor for a partial repass through said second and third heat exchangers,

a first liquid nitrogen boiler connected to receive and further cool the merged streams derived from repass of said second and third heat exchangers,

means comprising a second stage flash drum connected to receive the output from said first nitrogen boiler and to separate the partially purified helium vapor from the condensate in said stream,

a fourth heat exchanger for receiving and slightly cooling the helium vapor from said second stage flash drum,

a second liquid nitrogen boiler connected to receive the slightly cooled helium vapor from said fourth heat exchanger and to further cool said vapor to a temperature of about −340°F,

a third stage flash drum including means for separating the liquid condensate from helium vapor at a temperature of about −340°F,

means including said fourth heat exchanger for slightly reheating the output of said third stage flash drum,

nitrogen adsorbing means comprising a plurality of vessels each equipped with a bed of comminuted carbon connectable in alternation in respective on-cycle and off-cycle positions wherein said on-cycle vessel is connected to receive the nearly purified vapor from said third stage flash drum and said liquid nitrogen boiler for adsorbing residual impurities in said vapor including nitrogen,

means for deriving the helium product vapor of grade A purity from said nitrogen adsorber and directing said purified product vapor through said second and third heat exchangers in tandem to the output of said system,

auxiliary to the principal purification system, said fourth stage flash drum maintained at a pressure of slightly over one atmosphere and at a temperature not exceeding about −320°F for receiving and flashing the liquid condensate from said first stage flash drum,

means for deriving liquid condensate containing in excess of about 95 mol percent of nitrogen from said fourth stage flash drum and passing said liquid condensate through said second heat exchanger means for evaporation and heat exchange cooling said compressed enriched helium feed stream, wherein said condensate is heated up to above ambient temperature,

venting means connected to said second heat exchanger for venting said vaporized condensate from said system,

means for deriving the condensates from said second and third stage flash drums containing in excess of about 96 mol percent of nitrogen and more than 2 mol percent of helium,

means including a fifth stage vessel functioning as a helium stripper for receiving said condensates from said second and third flashing stages, said helium stripper maintained at slightly over one atmosphere pressure at a temperature of less than about −320°F for stripping the remaining helium from said condensates,

means connected to said helium stripper for returning the partially helium vapor from said helium stripper and said fourth stage flash drum through said second heat exchanger for recycling through said system, and

means for deriving the condensate from said helium stripper consisting of about 99 mol percent of liquid nitrogen, and passing said condensate through said third heat exchanger for evaporation and heat exchange with one component of said cooling compressed helium feed stream, wherein said condensate is heated up to near ambient temperature,

means for deriving any condensate from said fourth stage flask drum consisting of at least about 95 mol percent of liquid nitrogen and such condensate from said helium stripper as is required for heat balance in said second heat exchanger, and passing said condensate through said second heat exchanger for evaporation and heat exchange with the other component of said cooling compressed helium feed stream, wherein said condensate is heated up to about ambient temperature,

venting means connected to receive the evaporated condensate from said second heat exchanger for venting said last-named evaporated condensate consisting of nitrogen having a purity of at least about 95 mol percent,

an off-site source of nitrogen maintained at a pressure of about 41 atmospheres,

means comprising a nitrogen liquefaction circuit for deriving a stream of nitrogen gas from said off-site source at said last-named pressure at near ambient temperature and converting said stream to liquid nitrogen, and

means for directing the said liquefied nitrogen to the coils of said adsorbing means for refrigerating said means at least during the on-cycle connection thereof, and to said liquid nitrogen boilers for refrigerating the partially purified helium stream, and

means connected to receive the evaporated condensate from said third heat exchanger for returning at least a part of the evaporated condensate consisting of nitrogen having a purity of about 99 mol percent to said off-site nitrogen source to make up losses.

24. The combination in accordance with claim **23** wherein said first unit is designed to convert a feed stream of natural gas containing between 15 and 40 mol percent of helium to crude helium containing between 60 and 80 mol percent of helium; wherein said second unit is designed to convert a feed stream of crude helium containing between 60 and 80 mol percent of helium and to enriched helium containing between 85 and 97 mol percent of helium, and wherein said third unit is designed to convert a feed stream of enriched helium containing between 85 and 97 mol percent of helium to helium of grade A purity having an impurity content not exceeding about **30** parts per million.

25. The combination in accordance with claim **23** wherein the stream of condensed hydrocarbons in said

first unit has a heating capacity of between about 850 and 950 British Thermal Units per hour, and

wherein in said third unit for preparing grade A helium, said means for heating said enriched helium feed stream to in excess of 220°F includes a heater for application to said enriched helium feed stream before said first heat exchanger is warmed up, said heater including means for deriving a fuel comprising at least a portion of the stream of said condensed hydrocarbons from said first unit.

26. The combination in accordance with claim **23** wherein said source of a stream containing about 20 mol percent of oxygen includes adsorbing means for adsorbing water vapor and carbon dioxide from said stream, and means for reactivating said adsorbing means comprising a stream of waste nitrogen which is a by-product of said second or third units.

27. The combination in accordance with claim **23** comprising means for optionally returning waste nitrogen from said first and second operations in combination with the recycle stream in each of said operations.

28. A system for converting a feed stream comprising natural gas to crude helium without auxiliary means for providing internal refrigeration, which comprises, in combination:

means for compressing said feed stream to about 30 atmospheres pressure,

a first heat exchanger for cooling said feed stream in a heat exchange with returning product, recycle, and condensed liquid streams,

means for separating out the condensed hydrocarbons from said feed stream comprising a flash drum having a plurality of stages at progressively lower pressures,

a second heat exchanger connected to receive vapor from the first flash stage of said plurality of flashing stages for subcooling said vapor in a heat exchange with returning product and recycle streams,

means comprising a helium stripper having a first stage at high pressure, and a plurality of subsequent stages at progressively lower pressures for separating a major portion of the helium vapor from said subcooled vapor,

means for deriving crude helium vapor from said first stage of said helium stripper and for returning said vapor through said heat exchangers to storage,

means for deriving recycle vapor including a substantial component of helium from said subsequent stages of said helium stripper and for returning said recycle vapor through said heat exchangers,

means for compressing said recycle vapor and returning said vapor to said feed stream,

means for deriving a first stream comprising hydrocarbon condensate from said plurality of flashing stages and returning said first stream of condensate through said first heat exchanger for refrigeration and to storage, and

means for deriving a second stream of hydrocarbon condensate from the stages of said helium stripper and returning said second stream of condensate through said first and second heat exchangers for refrigerating said heat exchangers.

29. A system for converting natural gas into crude helium in accordance with claim **28** which comprises means for optionally combining said first and second streams of hydrocarbon condensate in said first heat exchanger into a by-product stream consisting of commercial grade fuel having a heating capacity of between 850 and 950 British Thermal Units per cubic foot.

30. A system for converting natural gas into crude helium in accordance with claim **29** which comprises means for optionally combining and returning one or both of said streams of hydrocarbon with said recycle vapor.

31. A system in accordance with claim **28** for converting a feed stream comprising natural gas including between 15 and 40 mol percent of helium to crude helium having a purity of 60 to 80 mol percent wherein said flash drum has at least two stages,

the first of said stages being maintained at a pressure of about 30 atmospheres and a temperature of about −148°F, and

the second of said stages being maintained at a pressure slightly in excess of 13 atmospheres at a temperature below −169°F;

and wherein said helium stripper comprises at least three stages,

the first stage being maintained at a pressure of about 30 atmospheres and a temperature of about −258°F,

the second said stage being maintained at a pressure slightly in excess of 13 atmospheres at said temperature, and

the third said stage being maintained at a pressure slightly in excess of 5 atmospheres at a temperature of about −264°F.

32. A system for converting a feed stream comprising crude helium to enriched helium which comprises in combination:

means for deriving a crude helium feed stream compressed to a pressure of in excess of about 80 atmospheres,

heat exchanger means for cooling said crude helium feed stream to a temperature below the condensation point of liquid nitrogen,

means comprising a helium separator connected to receive the cooled crude helium feed stream from said heat exchanger means for separating out enriched helium vapor from said feed stream,

work-expansion means connected to receive and work-expand the enriched helium vapor from said helium separator,

a system of conduits connected to receive the work-expanded enriched helium vapor from said work expansion means and direct said enriched helium vapor to return through said heat-exchanger means for cooling said crude helium feed stream,

a conduit connected to the bottom of said helium separator for deriving liquid condensate therefrom,

auxiliary separation means connected to receive the liquid condensate from said helium separator,

means for deriving the partly helium vapor from said auxiliary separation means and returning said vapor through said heat exchanger means for cooling said feed stream,

compressing means connected to said heat exchanger means for compressing and returning said partly helium vapor to said feed stream for recycling in said system,

and means for deriving said liquid condensate comprising primarily nitrogen from said auxiliary separation means and directing said condensate to pass

through said heat exchanger means for evaporation therein to cool said crude helium feed stream.

33. The combination in accordance with claim **32** which comprises means for optionally returning the evaporated condensate comprising primarily nitrogen from said suxiliary separation means through said compression means for recycling in said system.

34. The combination in accordance with claim **32** wherein a reheat cycle is connected ahead of said work-expansion means, said reheat cycle including a portion of said heat exchanger means for receiving the enriched helium vapor from said helium separator and reheating said vapor to a temperature exceeding the condensation temperature of any of the components of said vapor by more than the expected reduction in temperature in said work-expansion means, whereby no liquid is formed in said work-expansion means.

35. A system for converting a feed stream comprising crude helium having a purity of 60 to 80 mol percent to enriched helium having a purity of between 85 and 97 mol percent which comprises in combination:

means for deriving a crude helium feed stream compressed to in excess of about 80 atmospheres at an initial temperature in excess of about 40°F,

conduit means for dividing said compressed crude helium feed stream into two components, one said component comprising in excess of about 95 percent by volume and the other said component comprising less than about 5 percent by volume,

heat exchanger means for cooling said crude helium feed stream to a temperature below the condensation temperature of liquid nitrogen,

said heat exchanger means comprising:

a first heat exchanger connected to receive and cool said minor component in a heat exchange with a returning recycle stream,

means connected in series with said first heat exchanger comprising a reboiler coil interposed in the bottom of a helium stripper,

a second heat exchanger connected to receive and cool said major component in a heat exchange with returning enriched helium product and condensate streams,

means for combining the respective two crude helium components from said reboiler coil and said second heat exchanger at a temperature of about −316°F,

means comprising said helium separator connected to receive the combined cooled crude helium feed stream from said heat exchanger means for separating out enriched helium vapor from said crude feed stream,

work-expansion means comprising a turbo-expander connected to receive the enriched helium vapor from said helium separator and to work expand said vapor,

a system of conduits connected to receive the work-expanded enriched helium vapor from said work expansion means and direct said enriched helium vapor to return through said heat exchanger means for cooling said crude helium feed stream,

a conduit connected to the bottom of said helium separator for deriving liquid condensate therefrom,

auxiliary separation means connected to receive the liquid condensate from said helium separator,

wherein said auxiliary separation means comprises:

a first auxiliary flash drum maintained at about 2 atmospheres pressure,

a second auxiliary flash drum maintained at slightly above atmospheric pressure and serving as said helium stripper connected to receive the liquid condensate from said first auxiliary flash drum for boiling in heat exchange with the cooled feed stream in said reboiler coil to separate out any vapor including helium vapor therefrom,

means for deriving a partly helium vapor from said auxiliary flashing separation means comprising conduit means connected between the tops of said first and second auxiliary flash drums and said first heat exchanger for supplying refrigeration to said heat exchanger,

compressing means connected to said heat exchanger means for compressing and returning said partly helium vapor to said feed stream for recycling in said system, and

means for deriving said primarily liquid condensate comprising primarily nitrogen from said auxiliary separation means and directing said condensate to pass through said heat exchanger means for evaporation to cool said crude helium stream, said means comprising conduit means connected between the bottom of said second auxiliary flash drum and said second heat exchange means for supplying refrigeration thereto.

36. The combination in accordance with claim **35** wherein means comprising a syphon is connected to the bottom of said second auxiliary flash drum serving as said helium stripper, said syphon constructed and arranged to maintain a preselected liquid level in said helium stripper-reboiler, a pressure-actuated valve being connected between the vapor output of said helium stripper and the head of said syphon for regulating the pressure in said syphon in accordance with the output vapor pressure of said helium stripper.

37. The combination in accordance with claim **35** wherein:

a three-way valve is connected to receive streams from the cold ends of each of said first and second heat exchangers and to supply a merged stream to the input of said helium separator, and

means for controlling the ratio of the streams received from the path including said first heat exchanger and said reboiler coil, and the path including said second heat exchanger in accordance with the temperature differential between the warm ends of said heat exchangers.

38. The combination in accordance with claim **35** wherein a reheat cycle is connected ahead of said turboexpander, said reheat cycle being connected through a portion of said second heat exchanger for receiving the enriched helium vapor from said helium separator and reheating said vapor to a temperature exceeding the condensation temperature of any of the components of said vapor by more than the expected reduction in temperature of said vapor in said turboexpander.

39. A system in accordance with claim **38** for converting crude helium having a purity of 60 to 80 mol percent to enriched helium having a purity of between 85 and 97 mol percent:

wherein said heat exchanger means is constructed to cool said feed stream to a temperature of about −315°F in heat exchange with returning enriched helium product, recycle, and condensate streams,

wherein said work expansion means comprises a turboexpander for work expanding said enriched helium product to about 60 atmospheres pressure,

wherein said auxiliary separation means comprises a single helium stripper connected to receive the condensate from said helium separator, said stripper maintained at an internal pressure of slightly above 5 atmospheres and including packing means for separating liquid from vapor in said stripper, said stripper including a bayonet heater connected to a source of energizing power for reboiling liquid condensate collected in the bottom of said stripper, and

means for separately returning the liquid condensate and partially helium vapor from said helium stripper through said heat exchanger means, and for returning said partially helium vapor to said compressor for recycle.

40. The combination in accordance with claim **35** wherein said heat exchanger means comprises a single heat exchanger for cooling said feed stream in heat exchange with enriched helium product vapor, partly helium vapor, and liquid condensate from said helium stripper,

wherein the said single heat exchanger includes a reheat coil connected to receive and partly reheat said enriched product vapor from said helium separator ahead of said turboexpander.

41. A system in accordance with claim **35** wherein said heat exchanger means comprises a first heat exchanger for cooling said crude helium feed stream in heat exchange with expanded enriched helium product vapor and a second heat exchanger interposed between said first heat exchanger and said helium separator in the path of said cooled feed stream for subcooling said crude helium feed stream in a heat exchange with enriched helium product from said turboexpander.

42. A system for preparing grade A helium from an enriched helium feed stream which comprises in combination:

means for deriving an enriched helium feed stream compressed to a pressure of about 60 atmospheres,

means comprising a first heat exchanger for heating said feed stream up to a temperature in excess of 220°F,

a source of a stream containing about 20 mol percent of oxygen for combining with said helium enriched feed stream,

means comprising a catalytic dehydrogenator connected to receive said enriched feed stream in combination with said oxygen containing stream for converting a major portion of the hydrogen in said feed stream to water vapor,

means for receiving the output of said dehydrogenator comprising a chemical hydrogen adsorber,

said first heat exchanger connected to cool the output of said adsorber in a heat exchange with the enriched helium feed stream entering said dehydrogenator,

means for drying said dehydrogenated stream,

compressing means connected to compress said dried stream to about 185 atmospheres pressure,

means comprising an aftercooler following said compressing means,

a second and third heat exchanger connected in tandem to receive said compressed dehydrogenated enriched helium stream in two components and to cool said stream components to below the condensation temperature of liquid nitrogen in a heat exchange with returning purified helium product, recycle, and condensate streams,

flashing means, comprising a first stage flash drum, for flashing the recombined components of said cooled enriched compressed dehydrogenated helium stream through a plurality of stages to separate out higher boiling liquid condensates from the helium-containing vapor in said stream,

means for deriving the nearly purified helium vapor from said first stage flash drum and directing said helium vapor for a partial repass through said second and third heat exchangers,

a first liquid nitrogen boiler connected to receive and further cool the merged streams derived from repass of said second and third heat exchangers,

means comprising a second stage flash drum connected to receive the output from said first nitrogen boiler and to separate the partially purified helium vapor from the condensate in said stream,

a fourth heat exchanger for receiving and slightly cooling the helium vapor from said second stage flash drum,

a second liquid nitrogen boiler connected to receive the slightly cooled helium vapor from said fourth heat exchanger, and to further cool said vapor to a temperature of about −340°F,

a third stage flash drum including means for separating the liquid condensate from helium vapor at a temperature of about −340°F,

means including said fourth heat exchanger for slightly reheating the output of said third stage flash drum,

nitrogen adsorbing means comprising a plurality of vessels each equipped with a bed of comminuted carbon connectable in alternation in respective on-cycle and off-cycle positions, wherein said on-cycle vessel is connected to receive the nearly purified vapor from said third stage flash drum and said liquid nitrogen boiler for adsorbing residual impurities in said vapor including nitrogen,

means for deriving the purified helium product vapor from said nitrogen adsorber and directing said purified product vapor through said second and third heat exchangers in tandem to the output of said system,

auxiliary to the principal purification system, said fourth stage flash drum maintained at a pressure of slightly over one atmosphere at a temperature of about −320°F for receiving and flashing the liquid condensate from said first stage flash drum,

means for deriving liquid condensate containing in excess of about 95 mol percent of nitrogen from said fourth stage flash drum and passing said liquid condensate through said second heat exchanger means for evaporation to cool said compressed enriched helium feed stream, wherein said condensate is heated up to near ambient temperature,

venting means connected to said second heat exchanger for venting said vaporized condensate from said system,

means for deriving the condensates from said second and third stage flash drums containing in excess of about 96 mol percent of nitrogen and more than 2 mol percent of helium,

means including a fifth-stage vessel functioning as a helium stripper for receiving said condensates from said second and third flashing stages, said helium stripper maintained at slightly over one atmosphere pressure at a temperature of less than about −320°F for stripping the remaining helium from said condensates,

means connected to said helium stripper for returning the partially helium vapor from said helium stripper through said second heat exchanger for recycling through said system, and

means for deriving the condensate from said helium stripper consisting of about 99 mol percent of liquid nitrogen and passing said condensate through said third heat exchanger for evaporation to cool one component of said cooling compressed helium feed stream, wherein said condensate is heated up to near ambient temperature,

means for deriving any condensate from said fourth stage flash drum consisting of at least about 95 mol percent of liquid nitrogen and such condensate from said helium stripper as is required for heat balance in said second heat exchanger, and passing said condensate through said second heat exchanger for evaporation and heat exchange with the other component of said cooling compressed helium feed stream, wherein said condensate is heated up to about ambient temperature,

venting means connected to receive the evaporated condensate from said second heat exchanger for venting said last-named evaporated condensate consisting of nitrogen having a purity of at least about 95 mol percent,

an off-site source of nitrogen maintained at a pressure of about 41 atmospheres,

means comprising a nitrogen liquefaction circuit for deriving a stream of nitrogen gas from said off-site source at said last-named pressure at near ambient temperature and converting said stream to liquid nitrogen and

means for directing the said liquefied nitrogen to the coil of said on-cycle adsorbing means for refrigerating said means at least during the on-cycle connection thereof, and to said liquid nitrogen boilers for refrigerating the partially purified helium stream,

and means connected to receive the evaporated condensate from said third heat exchanger for returning at least a part of the evaporated condensate consisting of nitrogen having a purity of about 99 mol percent to said off-site nitrogen source to make up losses.

43. In a system in accordance with claim **42** for preparing grade A helium having an impurity content not exceeding about 30 parts per million from an enriched helium feed stream containing between 85 and 97 mol percent helium,

means for purging the carbon bed of an off-cycle one of said carbon adsorbing means which comprises in combination a purge connection to said compressing means ahead of said aftercooler for deriving from said compressing means a small component of hot compressed feed stream,

a system of valves for alternatively connecting said purge connection to the off-cycle one of said carbon adsorbing means,

and means simultaneously connectable to said off-cycle adsorbing means for venting the purged gases into said compressed feed stream for at least a partial repass through said system commencing just ahead of said second and third heat exchangers.

44. In a system in accordance with claim **42** wherein said nitrogen liquefaction system comprises in combination:

a heat exchanger connected to said off-site source to receive a high pressure stream of nitrogen gas from said off-site source, at a pressure of about 41 atmospheres at least at ambient temperature, and to cool said stream in two components in heat exchange with low pressure nitrogen streams returning through said heat exchanger,

means comprising a turboexpansion device connected to receive the major component of said high pressure nitrogen stream after partial cooling in said heat exchanger to about −170°F, and to expand said stream to about atmospheric pressure, and to further cool said component to a temperature of about −310°F,

conduit means for returning said cooled low pressure major stream through said heat exchanger for cooling said high pressure streams,

means comprising a pressure-controlled expansion valve for receiving the minor high pressure component of said stream cooled to about −275°F at a pressure of about 40 atmospheres and expanding said component to a pressure just above atmospheric pressure at a temperature of about −310°F,

a flash drum connected to said valve to receive said minor stream after expansion and separate the vapor phase from the liquid phase in said stream,

conduit means connected between the upper portion of said flash drum and said conduit means for returning said major stream for returning the vapor portion of said minor component through said heat exchanger,

conduit means connected between the lower end of said flash drum and the refrigeration coils of at least the on-cycle one of said carbon adsorbers for supplying refrigerant to said coils,

conduit means connected between the lower end of said flash drum and the coils of said liquid nitrogen boilers for supplying refrigerant to said boilers,

conduit means connected to the refrigeration coils of said carbon adsorber and to the refrigeration coils of said nitrogen boilers for returning the evaporated nitrogen from said coils through said heat exchanger,

and means for optionally returning said evaporated nitrogen to said off-cycle nitrogen source or to said system for recycle with the stream under purification.

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