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(54) RECOVERY OF HELIUM FROM NITROGEN-RICH STREAMS

(57) Overall power consumption in a cryogenic distillation process for recovering helium from nitrogen-rich gases comprising helium may be reduced if the feed to the distillation column system is at least substantially condensed by indirect heat exchange against a first bottoms liquid at first pressure, and a second bottoms liquid at a second pressure that is different from the first pressure.

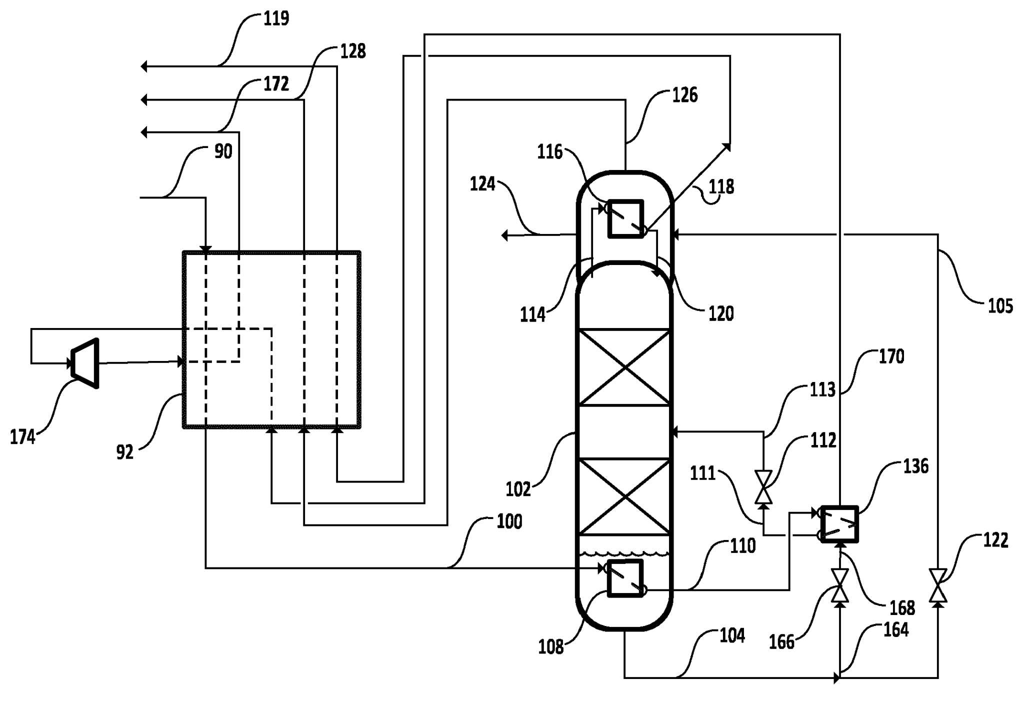

FIG. 2

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Description

[0001] This invention relates to the recovery of helium from nitrogen-rich gas. The invention has particular application in the recovery of helium from nitrogen-rich natural gas from an underground source.

[0002] US5167125A discloses a process for recovering light gases such as hydrogen, neon and helium, from gas stream containing higher boiling components such as nitrogen and C_{1-2} hydrocarbons. According to the embodiment depicted in Fig. 1 of US5167125A, a stream 100 of feed gas is cooled by indirect heat exchange and the cooled feed gas 110 is reduced in pressure across valve 112 and fed to a distillation column 102 where it is separated into bottoms liquid depleted in light gas(es), and overhead vapor enriched in light gas(es). The bottoms liquid is reboiled using the feed gas in reboiler 108 to provide vapor for the column. Nitrogen in the overhead vapor is condensed in the overhead condenser 116 by indirect heat exchange against a stream 104 of bottoms liquid that is expanded across valve 122, and the resultant liquid nitrogen is recycled to the column as reflux 120. A stream 118 of impure helium gas is removed from condenser 16.

[0003] It is an objective of the present invention to provide an improved process for recovering helium from nitrogen-rich gases comprising helium, particularly nitrogen-rich natural gas from underground sources.

[0004] It is an objective of preferred embodiments of the present invention to reduce the overall power required to recover helium from such gases. Helium may be recovered at a higher pressure than in prior proceses and with reduced loss.

[0005] It is also an objective of preferred embodiments of the present invention to reduce the capital and operating costs of apparatus for recovering helium from such gases.

[0006] According to a first aspect of the present invention, there is provided a process for recovering helium from a nitrogen-rich feed gas comprising helium. The process comprises cooling a pressurized nitrogen-rich feed gas comprising helium to produce cooled feed; and separating said feed in a first distillation column system operating at an elevated operating pressure to produce helium-enriched overhead vapor and nitrogen-enriched bottoms liquid. The process is characterized in that the feed to the first distillation column system is at least partially condensed; and that the cooling of the feed gas is achieved by indirect heat exchange against at least a first bottoms liquid at a first elevated pressure and a second bottoms liquid at a second elevated pressure that is different from the first elevated pressure.

[0007] According to a second aspect of the present invention, there is provided apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid;

an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;

a first heat exchange system for cooling feed gas by indirect heat exchange with a first nitrogen-enriched bottoms liquid to produce cooled feed gas and vapor for the column system;

a first pressure reduction device for reducing the pressure of a second nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid;

a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against said reduced pressure bottoms liquid to produce at least partially condensed feed gas and at least partially vaporized bottoms liquid; and

a second pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

[0008] According to an alternative arrangement of the second aspect of the present invention, there is provided apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid;

an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;

a first pump for pumping a second nitrogen-enriched bottoms liquid to produce pumped bottoms liquid;

a first heat exchange system for cooling feed gas by indirect heat exchange with said pumped bottoms liquid to produce cooled feed gas and nitrogen-enriched vapor;

a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against a first nitrogen-enriched bottoms liquid to produce at least partially condensed feed gas and vapor for the column system; and

a pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

[0009] All references herein to pressure are references to absolute pressure and not gauge pressure unless expressly stated otherwise. In addition, references to the singular should be interpreted as including the plural and vice versa, unless it is clear from the context that only the singular or plural is meant. Further, unless expressly stated otherwise, fluid compositions are calculated in mol. % on a "dry" basis, *i.e.* excluding any water content from the calculations. In reality, to avoid operating problems, water content must be low enough, typically no more than 10 ppm, to avoid freeze-out and/or hydrate formation at the cold end of the process.

[0010] The terms "elevated pressure" and "pressurized" are intended to mean a pressure that significantly more than atmospheric pressure. The terms are intended to exclude insignificant increases in pressure, *e.g*. produced by a fan, simply to overcome pressure drop in apparatus that is operating at about atmospheric pressure. By use of the terms "elevated pressure" and "pressurized", the Inventors are typically referring to absolute pressures of at least 1.5 bar, *e.g.* at least 2 bar.

[0011] The term "indirect heat exchange" means that sensible and/or latent heat as appropriate is transferred between fluids without the fluids in question coming into direct contact with each other. In other words, heat is transferred through a wall of a heat exchanger. The term is intended to include the use of an intermediate heat transfer fluid where appropriate.

[0012] The term "distillation" is intended to include rectification and fractionation.

Overview of the process

[0013] The process involves cooling a pressurized nitrogen-rich feed gas comprising helium to produce cooled feed; and separating the feed in a first distillation column system operating at an elevated operating pressure to produce helium-enriched overhead vapor and nitrogen-enriched bottoms liquid. The feed to the first distillation column system is at least partially condensed. The cooling of the feed gas is achieved by indirect heat exchange against at least a first nitrogen-enriched bottoms liquid at a first elevated pressure and a second nitrogen-enriched bottoms liquid at a second elevated pressure that is different from the first elevated pressure.

[0014] The first and second nitrogen-enriched bottoms liquids are typically taken from the sump of the first distillation column system and may be different portions of the same bottoms liquid. However, one of the bottoms liquids could be taken from a different point at the bottom of the distillation column system.

[0015] The compositions of the first and second nitrogen-enriched bottoms liquids are usually at least substantially identical although slight variations may be observed depending on the precise location in the sump of the distillation column system at which the bottoms liquid is used to cool the feed, or from which the bottoms liquid is extracted for use in cooling the feed. However, any variations in composition that may be present would be too small to have any significant effect on the operation of the process.

Cooling and condensing the feed

[0016] The first elevated pressure is typically equal to the elevated operating pressure of the first distillation column system. In this regard, the first elevated pressure is usually from about 2 bar to about 35 bar, and preferably from about 10 bar to about 30 bar.

[0017] The second elevated pressure may be more than or less than the elevated operating pressure of the first distillation column system. The difference between the first and second elevated pressures is typically at least 1 bar, e.g. at least 2 bar, for example at least 5 bar or, in some embodiments, at least 10 bar.

[0018] Typically, the vaporization pressure of the second nitrogen-enriched bottoms liquid is relatively close to the feed pressure (either pumped or expanded) whether it is taken as pressurized product or it gets expanded.

[0019] In embodiments in which the second elevated pressure is less than the elevated operating pressure of the first distillation column system, the second elevated pressure is typically significantly more than 1 bar, e.g at least 1.5 bar or from 2 bar to about 30 bar, and preferably from 5 bar to 25 bar. In such embodiments, bottoms liquid is expanded to produce the second bottoms liquid.

[0020] In embodiments in which the second elevated pressure is greater than the elevated operating pressure of the first distillation column system, the second elevated pressure may be from about 3 bar to about 150 bar, and preferably from about 10 bar to 100 bar. In such embodiments, bottoms liquid is pumped to produce the second the bottoms liquid.

[0021] The feed gas may be at subcritical pressure or supercritical pressure.

[0022] In embodiments in which the feed gas is at subcritical pressure, the feed gas may cooled (and possibly partially condensed) by indirect heat exchange against the first bottoms liquid and then at least partially condensed (or a further portion condensed) by indirect heat exchange against the second bottoms liquid.

[0023] In embodiments in which the feed gas is at supercritical pressure, the feed gas may be cooled by indirect heat exchange against the first bottoms liquid and then further cooled by indirect heat exchange against the second bottoms liquid. The pressure of the cooled feed is let down prior to being fed to the first distillation column system.

[0024] The feed to the first distillation column system typically has a vapor fraction of no more than 0.5, *e.g.* no more than 0.3 or no more than 0.2 or even no more than 0.05. In some preferred embodiments, the feed to the first distillation column system is at least substantially fully condensed.

[0025] The pressurized nitrogen-rich feed gas is usually at a pressure greater than the elevated operating pressure of said first distillation column system. In this regard, the pressurized feed is typically taken from a natural underground source. Where the pressurized feed is at a pressure greater than the elevated operating pressure of the first distillation column system, the process comprises expanding the at least partially condensed feed prior to separation.

[0026] The pressure of the pressurized nitrogen-rich feed gas may be from about 2 bar to about 200 bar, and is typically from about 10 bar to about 100 bar.

[0027] The elevated operating pressure of the distillation column system is usually from about 2 bar to about 35 bar, and preferably from about 10 bar to 30 bar.

Additional refrigeration requirement

[0028] Throughout this specification, the term "expanding" is intended to include expanding to produce work ("work expansion") and expanding isenthalpically, typically across a Joule-Thomson (or "J-T") valve. Gases are typically work expanded in an expander whereas liquids are usually expanded isenthalpically across a valve.

[0029] The process may comprise expanding vaporized bottoms liquid, or a fluid derived therefrom, to produce expanded nitrogen-enriched gas and using the expanded gas to provide a part of the refrigeration duty of the process. The vaporized bottoms liquid is usually work expanded in an expander.

[0030] The second nitrogen-enriched bottoms liquid is usually at least partially vaporized as a result of the indirect heat exchange against the feed gas. In such embodiments, the process may comprise warming the vaporized bottoms liquid by indirect heat exchange to produce warmed nitrogen-enriched gas; expanding the warmed nitrogen-enriched gas to produce expanded nitrogen-enriched gas; and cooling the feed gas by indirect heat exchange with the expanded nitrogen-enriched gas to produce cooled feed gas. The warmed nitrogen-enriched gas is usually work expanded in an expander.

[0031] In some embodiments, the process comprises expanding a third nitrogen-enriched bottoms liquid to produce expanded nitrogen-enriched fluid; vaporizing the expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in the first distillation column system to produce nitrogen-enriched gas; expanding the nitrogen-enriched gas to produce expanded nitrogen-enriched gas; and condensing nitrogen gas in the first distillation column system by indirect heat exchange against the expanded nitrogen-enriched gas to produce liquid reflux for the first distillation column system. The nitrogen-enriched gas is typically work expanded in an expander.

[0032] The pressure at which the expanded third bottoms liquid is vaporized is typically less than the pressure at which the expanded second bottoms liquid is vaporized.

[0033] The process may comprise expanding a fourth nitrogen-enriched bottoms liquid to produce further expanded nitrogen-enriched fluid; and vaporizing the further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in the first distillation column system to produce further nitrogen-enriched gas.

[0034] The pressure at which the expanded fourth bottoms liquid is vaporized is typically less than the pressure at which the expanded third bottoms liquid is vaporized.

[0035] Where the second bottoms liquid is vaporized as a result of the indirect heat exchange against the feed gas, the process may comprise expanding the vaporized bottoms liquid to produce expanded nitrogen-enriched gas; and condensing nitrogen gas in the first distillation column system by indirect heat exchange with the expanded nitrogen-enriched gas to produce liquid reflux for the first distillation column system and warmed nitrogen-enriched gas.

[0036] In such embodiments, the process may comprise expanding a third bottoms liquid to produce further expanded nitrogen-enriched fluid; and vaporizing the further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in the first distillation column system to produce further nitrogen-enriched gas. The vaporization pressure of the further expanded nitrogen-enriched fluid will typically be less than the vaporization pressure of the second bottoms liquid.

[0037] A fourth bottoms liquid may be expanded to form an expanded fluid which is then separated into a vapor phase and a liquid phase. The vapor phase may be warmed by indirect heat exchange to produce a gaseous nitrogen product.

[0038] Flash vapor may be formed on expanding bottoms liquid to form expanded bottoms liquid. Alternatively, the bottoms liquid could be subcooled prior to expansion and thereby avoid the formation of flash vapor. Such subcooling could be effected by indirect heat exchange against expanded nitrogen-enriched gas.

[0039] The bottoms liquid evaporated in the overhead condenser and not expanded in an expander is typically at the lowest pressure (*e.g.* from about 1 bar to about 10 bar) as it needs to boil at low temperature to condense as much nitrogen as possible from the helium.

[0040] The bottoms liquid evaporated in the overhead condenser and expanded in an expander is at an intermediate pressure (*e.g.* from about 2 bar to about 25 bar), and is typically only there if the vapour from the second bottoms liquid is taken as product and not expanded (e.g see Fig. 3), so there is no other source of expander refrigeration. This stream can be evaporated at an intermediate pressure and higher temperature to optimise the cooling in the condenser over the whole temperature range - most of the condensing duty is needed at the higher temperature where the nitrogen concentration in the helium is highest.

[0041] The third and fourth nitrogen-enriched bottoms liquids are typically taken from the sump of the first distillation column system and may be different portions of the same bottoms liquid. However, one or more of the bottoms liquids could be taken from a different point at the bottom of the distillation column system. In some embodiments, the first, second, third and fourth bottoms liquids are different portions of the same bottoms liquid.

[0042] The process is preferably autorefrigerated. The term "autorefrigerated" is intended to mean that all of the refrigeration duty required by the process is provided internally, *i.e.* by indirect heat exchange against fluid streams within the process. In other words, no additional refrigeration is provided from an outside source.

Origin of the feed

[0043] The feed gas may be taken from a natural gas liquid (NGL) recovery column. Thus, the process may comprise cooling and at least partially condensing pressurized dry, carbon dioxide-free natural gas to produce at least partially condensed natural gas; and separating the at least partially condensed natural gas in a second distillation column system operating at an elevated operating pressure to produce C_{2+}-depleted overhead vapor and C_{2+}-enriched bottoms liquid. In such embodiments, the C_{2+}-depleted overhead vapor is the nitrogen-rich feed gas.

[0044] The process may be integrated with a process for recovering helium and NGL from pressurized natural gas comprising predominantly nitrogen with smaller amounts of methane, C_{2+} hydrocarbons and helium.

[0045] The pressurized natural gas is usually extracted from an underground source, such as a geological deposit or a natural gas field. The natural gas is typically extracted at a pressure in the range from about 2 bar to about 200 bar, preferably from about 10 bar to about 100 bar.

[0046] The composition of natural gas depends on the source. However, some embodiments of the present invention concern recovering valuable components of nitrogen-rich natural gas, *i.e.* natural gas having a low calorific value, *e.g.* a calorific value of no more than 300 BTU/scf ("British thermal units/standard cubic foot"), *i.e.* about 11.2 MJ/sm^3 ("mega Joules/standard metre cubed" at 15°C). The natural gas comprises at least about 70%, *e.g.* at least about 80% and preferably at least about 90%, nitrogen. The nitrogen content of the pressurized natural gas is usually no more than 99% and typically no more than 95%.

[0047] Other components of the natural gas suitable to be processed by the present invention include methane, helium and C_{2+} hydrocarbons, typically together with one or more impurities such as carbon dioxide, water and hydrogen sulfide.

[0048] Methane is typically present in the natural gas in an amount in the range from about 0.1% to about 30%, for example from about 0.1% to about 20% or from about 0.1% to about 10%.

[0049] Helium is typically present in an amount in the range from about 0.01% to about 10%, for example from about 0.01% to about 5%.

[0050] C_{2+} hydrocarbons typically comprise C_2 to C_4 hydrocarbons, often together with C_5 and C_6 hydrocarbons. Typical C_{2+} hydrocarbons include one or more hydrocarbons selected from the group consisting of ethane (C_2), propane (C_3), butanes (C_4), pentanes (C_5) and hexanes (C_6). The natural gas typically comprises at least ethane, propane and butane. The total amount of C_{2+} hydrocarbons in the natural gas is typically in the range of about 0.01% to about 5%.

[0051] The process may comprise extracting the pressurized natural gas from an underground source and pre-treating the pressurized nitrogen-rich natural gas to remove one or more impurities incompatible with the process and thereby produce pre-treated natural gas.

[0052] Purities that are incompatible with the process include carbon dioxide, water and hydrogen sulfide. These impurities are incompatible because at least a portion of the pressurized natural gas is cooled to a low temperature, typically below -100°C. At such cryogenic temperatures, these impurities freeze out of the gas causing blockages in pipework and channels within heat exchangers, *etc.* Therefore, such "freezable" components are removed before the natural gas is cooled.

[0053] The impurities may be removed using conventional techniques. In this regard, water may be removed in a selective adsorption process, *e.g.* using a zeolite adsorbent; and carbon dioxide and/or hydrogen sulfide may be removed in an absorption process, *e.g.* using an amine such as monoethanolamine.

[0054] The natural gas being pre-treated for impurity removal is typically at a pressure in the range from about 2 bar to about 100 bar, for example from about 40 bar to about 60 bar, *e.g.* about 50 bar. If the pressure of the natural gas after extraction is within this range, then the natural gas could be pre-treated without pressure adjustment. If the pressure of the natural gas is significantly more than 100 bar, then the pressure of the natural gas would be reduced prior to undergoing the pre-treatment.

[0055] The pre-treated natural gas is cooled to produce cooled pre-treated natural gas which is separated by distillation in the second distillation column system (*i.e.* an NGL recovery column system) to produce NGL and C_{2+} hydrocarbon-depleted natural gas comprising helium and methane.

[0056] The skilled person would appreciate that the temperature to which the pre-treated gas is cooled depends on the pressure and composition of the gas. With this data, it is possible to determine the temperature to which the gas is cooled prior to being fed to the NGL recovery column.

[0057] The second distillation column system may comprise more than one distillation column although, in preferred embodiments, the system comprises a single distillation column. The column may be trayed and/or packed as required or as desired.

[0058] The second distillation column system usually operates at a pressure from about 2 bar to about 35 bar, for example from about 25 bar to 35 bar, *e.g.* about 30 bar. In embodiments in which the pressure of the cooled pre-treated gas is within these ranges, the pre-treated gas could be fed to the second distillation column system without pressure adjustment. However, the pressure of the cooled pre-treated gas is typically substantially more than 35 bar. Therefore, the pressure of the cooled pre-treated gas is usually reduced prior to being fed to the second distillation column system.

Purification of helium product

[0059] The helium-enriched overhead vapor typically comprises at least 50%, for example at least 65%, preferably at least 80%, e.g. about 90%, helium. The remainder of the helium-enriched overhead vapor is usually predominantly nitrogen.

[0060] The helium-enriched overhead vapor may be purified. In such embodiments, the process may comprise warming the helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched gas; and purifying the helium-enriched gas to produce pure helium gas. The purified helium gas typically comprises at least 99% helium.

[0061] The helium-enriched gas is typically purified by a pressure swing adsorption (PSA) process. Tail gas from the PSA process may be recycled to the first distillation column system after suitable pressure and temperature adjustment.

[0062] If the feed gas contains hydrogen, the purification process may also include a catalytic oxidation step (*e.g.* a NIXOX unit). The catalytic oxidation step may be carried out upstream of the PSA, and the tail gas from the PSA recycled upstream of the feed pretreatment unit to remove resultant CO_2 and water, or to an intermediate point in the pretreatment unit, such as between the CO_2 and water removal steps if only water was produced in the NIXOX unit, or water and only small amounts of CO_2 that can be removed in the water removal step, or it may be treated separately in a TSA system.

[0063] In embodiments in which liquid nitrogen is produced as product, at least a portion of the liquid nitrogen may be used as a refrigerant in a process to liquefy the purified helium.

Recovery of methane from nitrogen-enriched bottoms liquid

[0064] Where the feed gas comprises methane, methane is typically recovered from nitrogen-enriched bottoms liquid as fuel gas and/or liquefied natural gas (LNG). In such embodiments, the methane is typically separated by distillation in a third distillation column system (*i.e.* a methane recovery column system) operating at elevated operating pressure(s) to produce nitrogen overhead vapor and methane-enriched bottoms liquid.

[0065] Methane-enriched bottoms liquid typically comprises at least 90%, for example about 95%, methane. The bottoms liquid may be removed from the process without vaporization to form an LNG product. Additionally or alternatively, a portion of the methane-enriched bottoms liquid may be vaporized to produce fuel gas.

[0066] Nitrogen-enriched overhead vapor typically comprises at least 99% nitrogen. The nitrogen overhead vapor may be warmed by indirect heat exchange to produce warmed nitrogen gas. Additionally or alternatively, at least a portion of the nitrogen in the nitrogen-enriched overhead vapor is condensed and removed as liquid nitrogen. The liquid nitrogen typically comprises at least 99% nitrogen.

[0067] A portion of the nitrogen gas may recycled to the third distillation column system after suitable pressure and temperature adjustment. The nitrogen gas may be recycled from any point downstream of the third distillation column system, *e.g.* after warming, compression, cooling and/or expansion. Such a recycle can increase the refrigeration available to the process, and therefore increase the quantity of liquid products that can be made.

[0068] Additionally or alternatively, a portion of the nitrogen gas may be expanded to produce expanded nitrogen gas, which is then warmed by indirect heat exchange to produce warmed expanded nitrogen gas. In such embodiments, the nitrogen gas is usually work expanded in an expander to provide refrigeration for the production of liquid from the process.

[0069] The third distillation column system may comprise a single distillation column, or more than one distillation column in which each column operates at the same or different elevated pressures. In some preferred embodiments, the third distillation column system comprises a higher pressure distillation column (HP column) and a lower pressure distillation column (LP column). The column(s) may be trayed and/or packed as required or as desired.

[0070] The third distillation column system may comprise a condenser for condensing overhead vapor. A portion of the condensed phase is typically returned to the top of the column system as reflux. The condenser may be a stand alone unit, or in preferred embodiments, is a section in the main heat exchanger.

[0071] The or each elevated operating pressure of the third distillation column system is typically less than the pressure of the bottoms liquid in the first distillation column system. Therefore, the process typically comprises expanding the bottoms liquid to the or one of the elevated pressures of the third distillation column system prior to separation.

[0072] The third distillation column system typically operates at one or more pressures in the range from more than 1 bar to about 35 bar. Where the third distillation column system comprises an HP column and an LP column, the HP column typically operates at a pressure from about 20 bar to about 35 bar, for example at about 25 bar, and the LP column typically operates at a pressure from more than 1 bar to about 10 bar, for example about 1.5 bar. The pressure of the nitrogen-enriched bottoms liquid is adjusted as required prior to being fed to the methane recovery column system.

[0073] In a first arrangement of the apparatus, there is comprised a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid; an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system; a first heat exchange system for cooling feed gas by indirect heat exchange with a first nitrogen-enriched bottoms liquid to produce cooled feed gas and vapor for the column system; a first pressure reduction device for reducing the pressure of a second nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid; a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against said reduced pressure bottoms liquid to produce at least partially condensed feed gas and at least partially vaporized bottoms liquid; and a second pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

[0074] The apparatus may comprise a separator to separate partially condensed overhead vapor into helium-enriched vapor and liquid reflux.

[0075] Preferably, this arrangement comprises a third pressure reduction device for reducing the pressure of a third nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

[0076] These preferred arrangements usually comprise an expander for expanding said nitrogen-enriched vapor to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

[0077] Preferably, this arrangement of the apparatus also comprises a fourth pressure reduction device for reducing the pressure of a fourth portion of said bottoms liquid to produce reduced pressure bottoms liquid for vaporization, optionally by said indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

[0078] An alternative embodiment of the first arrangement of the apparatus may comprise an expander for expanding said vaporized bottoms liquid to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

[0079] In these embodiments, the apparatus preferably comprises a fourth pressure reduction device for reducing the pressure of a fourth portion of said nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid; and a storage vessel for storing said reduced pressure bottoms liquid.

[0080] In a second arrangement of the apparatus, there is comprised a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid; an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system; a first pump for pumping a second nitrogen-enriched bottoms liquid to produce pumped bottoms liquid; a first heat exchange system for cooling feed gas by indirect heat exchange with said pumped bottoms liquid to produce cooled feed gas and nitrogen-enriched vapor; a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against a first nitrogen-enriched bottoms liquid to produce at least partially condensed feed gas and vapor for the column system; and a pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

[0081] Preferably, this arrangement comprises a third pressure reduction device for reducing the pressure of a third portion of said bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

[0082] These preferred arrangements usually comprise an expander for expanding said nitrogen-enriched vapor to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

[0083] Preferably, this arrangement of the apparatus also comprises a fourth pressure reduction device for reducing the pressure of a fourth portion of said bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

[0084] The or each heat exchange system may be an independent unit. In other embodiments, the two or more heat exchange systems may be different sections of a single heat exchange unit. In preferred embodiments, all of the heat exchange systems identified above are different sections of a primary (or main) heat exchanger.

[0085] The invention will now be further described with reference to the comparative process depicted in FIG. 1 and the embodiments of the present invention depicted in FIGs. 2 to 9, in which:

FIG. 1 is a flowsheet depicting a comparative process for recovering helium from nitrogen-rich natural gas in which the feed to the column system is predominantly gaseous (see Comparative Example 1);

FIG. 2 is a flowsheet depicting a helium recovery process according to the present invention in which the feed to the column is predominantly liquid (see Example 1);

FIG. 3 is a flowsheet depicting a modified process of FIG. 2 in which a further portion of the bottoms liquid is expanded to an intermediate pressure and used to provide refrigeration duty in the separation (see Example 2);

FIG. 4 is a flowsheet depicting a modified process of FIG. 3 where the feed is at higher pressure and part of the helium-free product is pumped and used to cool the feed upstream of the column reboiler (see Example 3);

FIG. 5 is a flowsheet depicting a preferred process in which most of the nitrogen product is expanded to provide refrigeration to provide some helium-free liquid nitrogen as product (see Example 4);

FIG. 6 is a flowsheet depicting a modified process of FIG. 5 in which liquid product is subcooled in the column overhead condenser before being reduced in pressure to the storage tank (see Example 5);

FIG. 7 is a flowsheet depicting a process according to the present invention in which the helium recovery process is integrated with an upstream an NGL recovery column (see Example 6);

FIG. 8 is a flowsheet depicting the helium recovery process integrated with a downstream nitrogen purification column system (see Example 7); and

FIG. 9 is a flowsheet depicting a fully integrated scheme for processing nitrogen-rich natural gas from an underground source involving NGL recovery, HP and LP columns for nitrogen production, liquid nitrogen production and helium purification by PSA (see Example 8).

[0086] The comparative process depicted in FIG. 1 is based on the process disclosed in US5167125 integrated with a main heat exchanger 92 and with a gaseous feed comprising 93% nitrogen, 5% methane and 2% helium. The feed is at a temperature of about 49°C and a pressure of 30 bar.

[0087] A stream 90 of feed gas is cooled by indirect heat exchange in the main heat exchanger 92 to form a stream 100 of cooled gas. The cooled gas is fed to reboiler 108 of distillation column 102 where it is further cooled by indirect heat exchange against bottoms liquid in the column to form a stream 110 of further cooled feed. A small amount (~11%) of the feed is condensed. Stream 110 is then expanded across valve 112 to about 25 bar and the expanded stream 113 fed to the distillation column where it is separated into nitrogen-enriched bottoms liquid and helium-enriched overhead vapor.

[0088] A stream 104 of bottoms liquid is removed from the column 102, expanded across valve 122 to about 1.5 bar and then used to partially condense overhead vapor from the column 102 by indirect heat exchange. In this regard, a stream 114 of overhead vapor is fed to condenser 116 where it is partially condensed by indirect heat exchange against vaporizing bottoms liquid to produce liquid reflux 120 for the column and a stream 118 of crude helium gas which is warmed by indirect heat exchange in the main heat exchanger 92, thereby producing a stream 119 of warmed helium gas (~90%) containing nitrogen (~10%).

[0089] A stream 126 of nitrogen-enriched bottoms liquid vaporized by the condensing overhead vapor is then used to cool the feed by indirect heat exchange in the main heat exchanger 92 to produce stream 128 of warmed nitrogen gas

(~95%) containing methane (~5%).

[0090] All of the refrigeration for the comparative process depicted in FIG. 1 is provided by Joule-Thomson expansion.

[0091] In this example, there is no liquid product 124 from the boiling side of the condenser 116. Heat balance means that, because all of the feed is in the gaseous phase and no significant refrigeration is provided, all of the products must also be in the gaseous phase.

[0092] FIG. 2 depicts an improved process over FIG. 1. Common features have been given the same reference numerals. The following is a discussion of the new features.

[0093] FIG. 2 depicts a process according to the invention where stream 100 is gaseous or two phase. The feed is fully, or almost fully, condensed in heat exchanger 136 which is cooled by boiling a stream 168 of helium-free bottoms liquid at elevated pressure. In this regard, a portion 164 of the bottoms liquid is expanded across valve 166 and fed as stream 168 to the heat exchanger 136 to form stream 170 of vaporized bottoms liquid. Additional refrigeration is provided by expanding stream 170 in expander 174 and using the expanded stream to help cool the feed 90 in the main heat exchanger 92. A stream 172 of warmed nitrogen gas is then removed from the heat exchanger and may be purified.

[0094] An advantage of the process of FIG. 2 over the comparative process depicted in FIG. 1 is that because of the additional condensation of the feed in heat exchanger 136, the vapor part of the feed and therefore the vapor flow in the column 102 above the feed location is reduced significantly leading to a reduction in the diameter of that section of the column.

[0095] FIG. 3 depicts an improved process over FIG. 2. Common features have been given the same reference numerals. The following is a discussion of the new features.

[0096] In FIG. 3, a further portion 132 of helium-free bottoms liquid is expanded across valve 133 and the expanded stream 134 is fed to the overhead condenser 116 where it is boiled and superheated at an intermediate pressure. Stream 138 of vaporized bottoms liquid is expanded in expander 140 and the expanded stream 142 and reheated in condenser 116 to produce a stream 144 of reheated nitrogen gas which is used to help cool the feed 90 in the main heat exchanger 92. Stream 146 of the resultant nitrogen gas is taken from the heat exchanger 92 and is available as a product or for further purification.

[0097] Stream 170 is used without expansion to cool the feed 90 in the main heat exchanger 92.

[0098] An advantage of the process of FIG. 3 over the process depicted in FIG. 2 is that refrigeration is integrated with the separation process, and the amount of product available at pressure is increased.

[0099] FIG. 4 depicts a modified process of FIG. 3 in which the feed pressure is greater. Common features have been given the same reference numerals. The following is a discussion of the new features.

[0100] Stream 164 of helium-free bottoms liquid is pumped in pump 165 to produce a stream 168 of pumped bottoms liquid which is used to cool the feed in heat exchanger 169 upstream of the column reboiler 108. The refrigeration provided by the expander 140 offsets the energy input to the process of the pump 168.

[0101] FIG. 5 depicts a preferred process in which most of the nitrogen product is boiled and expanded to provide refrigeration for production of some of the nitrogen product as liquid.

[0102] Feed 90 is cooled initially by indirect heat exchange in the main heat exchanger 92 to produce stream 100 and then subsequently further cooled and condensed by indirect heat exchange in the column reboiler 108 and heat exchanger 136. Stream 111 of condensed feed is expanded across valve 112 and fed to column 102 for distillation. The column 102 is reboiled by the feed in reboiler 108, and nitrogen in the overhead vapor is condensed in condenser 116 to provide reflux 120 for the column 102. A stream 118 of impure helium gas is removed from the condenser 116 and warmed against the feed 90 in the main heat exchanger 92 to produce a helium gas stream 119 suitable for purification by PSA or by some other means.

[0103] A first portion of the helium-free bottoms liquid 104 is boiled in the bottom of column 102 to provide vapor for the column.

[0104] A second portion 132 of helium-free bottoms liquid 104 is expanded across valve 133 and the expanded stream 134 is used to cool and condense the feed by indirect heat exchange in heat exchanger 136. A stream 138 of vaporized bottoms liquid is work expanded in expander 140 to produce expanded stream 142 which is then fed to the overhead condenser 116 to condense nitrogen in the overhead vapor for reflux 120. Stream 144 of nitrogen gas is then fed to the main heat exchanger 92 to help cool the feed 90, thereby producing a stream 146 of impure nitrogen gas suitable for further purification.

[0105] A third portion of helium-free bottoms liquid 104 is expanded across valve 122 to produce expanded stream 105 which is fed to the overhead condenser 116 to help condense nitrogen in the overhead vapor. Stream 126 of nitrogen gas is then fed to the main heat exchanger 92 to help cool the feed 90, thereby produce another stream 128 of impure nitrogen gas suitable for further purification.

[0106] A fourth portion 180 of helium-free bottoms liquid 104 is expanded across valve 182 to form a two phase stream 184 which is fed to a storage tank 185 where it is separated into a liquid stream 186 and a vapor stream 188. Liquid stream 186 could be vaporized to provide refrigeration, for example in a downstream helium liquefier, or exported as a product, for example for fracking. The vapor stream 188 is used to help cool the feed 90 in the main heat exchanger 92 to produce a further stream 190 of impure nitrogen gas suitable for further purification.

[0107] FIG. 6 depicts a modified process of FIG. 5 in which liquid product is subcooled in condenser 116. Common features have been given the same reference numerals. The following is a discussion of the new features.

[0108] The fourth portion 180 of helium-free bottoms liquid is fed without expansion to the condenser 116 where it is subcooled to form stream 181 of subcooled bottoms liquid. Stream 182 is expanded across valve 182 to produce expanded stream 184 which is two phase. Stream 184 is fed to the storage tank 185 where it is separated into the liquid stream 186 and the vapor stream 188.

[0109] If the feed contains C_{2+} hydrocarbons, a hydrocarbon (NGL) recovery column may be added upstream of the helium separation column 102, as illustrated in FIG. 7.

[0110] Feed 90 is cooled in the main heat exchanger 92 and divided into a first portion 191 and a second portion. The first portion 191 is work expanded in expander 192 and the expanded stream 193 is fed back to the main heat exchanger 92 where it is further cooled to produce stream 194 which is fed to an intermediate location in an NGL recovery column 96. The second portion is further cooled and condensed by indirect heat exchange in the main heat exchanger to form stream 196 of liquid feed which is expanded across valve 94 to produce expanded feed stream 198 which is fed to the top of the NGL recovery column 96.

[0111] The feeds to the column 96 are separated into C_{2+} hydrocarbon bottoms liquid, removed as stream 199, and C_{2+} hydrocarbon-depleted overhead vapor. Column 96 is reboiled in reboiler 98 using an external heat source such as steam, hot oil or cooling water.

[0112] A stream 100 of overhead vapor is removed from column 96 and used to reboil the helium recovery column 102 to produce a stream 110 of cooled and partially condensed overhead vapor. Stream 110 is further cooled and condensed in heat exchanger 136 by indirect heat exchange against helium-free bottoms liquid 134 from column 102. The further condensed stream 111 is then expanded across valve 112 and fed as stream 113 to column 102 where it is separated into nitrogen-enriched bottoms liquid and helium-enriched overhead vapor.

[0113] A stream 114 of helium-enriched overhead vapor is taken from column 102 and nitrogen in the vapor is condensed by indirect heat exchange in heat exchanger 116 to form a two phase stream 115 that is separated in phase separator 103. A stream 120 of nitrogen-enriched liquid is used to provide reflux to column 102. A stream 118 of impure helium gas is warmed by indirect heat exchange in heat exchanger 116 to form stream 121 of warmed helium gas which is then used to help cool the feed 90 by indirect heat exchange in the main heat exchanger 92. The stream 119 of impure helium gas from the main heat exchanger 92 is suitable for purification by PSA or by some other means.

[0114] A first portion of the helium-free bottoms liquid 104 is boiled in the bottom of column 102 to provide vapor for the column.

[0115] A second portion of nitrogen-enriched bottoms liquid 104 is expanded across valve 122 and the expanded stream 105 is used to provide refrigeration duty in heat exchanger 116. The resultant stream 126 of vaporized liquid is then used to help cool the feed 90 by indirect heat exchange in the main heat exchanger 92 to produce a stream 128 of warmed impure nitrogen gas suitable for further purification.

[0116] A third portion 132 of the helium-free bottoms liquid 104 is expanded across valve 133 and then used to provide refrigeration duty in heat exchanger 136. The stream 137 of impure nitrogen gas is then removed from heat exchanger 136 and fed to the main heat exchanger 92 where is helps cool the feed 90. A stream 138 of warmed impure nitrogen gas is then work expanded in expander 140 and the expanded stream 142 is used to provide refrigeration duty in heat exchanger 116. The resultant stream 144 of impure nitrogen gas is then used to help cool the feed in the main heat exchanger 92.

[0117] A fourth portion 180 of the helium-free bottoms liquid is subcooled in heat exchanger 116 and the resultant stream 181 is expanded across valve 182 to form a two phase stream 184 which is fed to a storage tank 185 from which a stream 186 of liquid nitrogen may be removed. A stream 188 of impure nitrogen gas is taken from the storage tank 185 and used to help cool the feed 90 by indirect heat exchange in the main heat exchanger 92. Stream 190 of warmed impure nitrogen gas is suitable for further purification.

[0118] If pure nitrogen and/or a fuel stream are required, the helium-depleted bottoms liquid from the helium recovery column may be separated before and/or after work expansion, as illustrated in FIG. 8.

[0119] The feed 90 is cooled initially by indirect heat exchange in the main heat exchanger 92 and then further cooled and condensed by indirect heat exchange in the reboiler 108 of the helium recovery column 102 and heat exchanger 136. The condensed stream 111 is expanded across valve 112 and then fed as stream 113 to the column 102 where it is separated into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid.

[0120] Overhead vapor is removed as stream 114 and nitrogen in the stream is condensed by indirect heat exchange in heat exchanger 116 to form a two-phase stream 115 which is phase separated in phase separator 103. The liquid portion 120 is fed back to the top of the column 102 as reflux. The vapor portion 118 is used to help cool the overhead vapor in heat exchanger 116 and is then further warmed in the main exchanger 92 against the cooling feed 90. The resultant stream 119 of helium gas is suitable for further purification.

[0121] A portion 132 of the bottoms liquid 104 is expanded across valve 133 and the expanded stream 134 is warmed by indirect heat exchange in heat exchanger 136 before being fed as stream 200 to a first nitrogen purification column 208. The feed 200 is separated into methane-enriched bottoms liquid and nitrogen-enriched overhead vapor.

[0122] Overhead vapor 230 is condensed by indirect heat exchanger against expanded bottoms liquid 214 in overhead condenser 232 to produce reflux 234 for the column 208, and a stream 130 of liquid nitrogen. Stream 130 is cooled by indirect heat exchange in heat exchanger 136 and the cooled stream 180 is subcooled in heat exchanger 116. Subcooled stream 181 is expanded across valve 182 and the expanded stream 184 is fed to storage tank 185. A stream 186 of pure nitrogen liquid can be removed from tank 185. Vapor 188 from the tank is used to help cool the feed 90 in the main heat exchanger 92 to produce stream 190 of nitrogen gas.

[0123] A stream 210 of bottoms liquid is expanded across valve 212 and the expanded stream 214 is fed to the overhead condenser for refrigeration duty. Vaporized bottoms liquid is removed from the overhead condenser 232 as stream 216. Unvaporized bottoms liquid is removed as stream 218, vaporized by indirect heat exchange in heat exchanger 136 and the vaporized stream 220 is combined with stream 216 to form combined stream 222 which is used to help cool the feed 90 in the main heat exchanger 92 and then work expanded in expander 140. The expanded stream 142 is then fed to a second nitrogen purification column 258 operating at a lower pressure than the first nitrogen purification column 208.

[0124] A second portion 250 of bottoms liquid 104 from the helium recovery column 102 is subcooled by indirect heat exchange in heat exchanger 116 and the subcooled liquid 252 is expanded across valve 254 and the expanded stream 256 is fed to the top of the second nitrogen purification column.

[0125] The feeds to the second nitrogen purification column 258 are separated into methane-enriched bottoms liquid and nitrogen-enriched overhead vapor. A first portion 262 of the methane-enriched bottoms liquid is reboiled in heat exchanger 116 and fed back to the column 258 to provide vapor for the distillation. A second portion 270 of the bottoms liquid is pumped in pump 272 and the pumped stream 274 is used to help cool the feed 90 in the main heat exchanger 92 to produce a stream 276 of fuel gas.

[0126] A stream 226 of nitrogen vapor is warmed in heat exchangers 116 and 92 to provide a vent gas stream 146.

[0127] FIG. 9 depicts a fully integrated scheme with NGL recovery, HP and LP columns and liquid nitrogen production from an underground gas source, and helium purification by PSA to produce a stream 302 pure helium that can be fed directly to a helium liquefier.

[0128] Feed gas 70 from an underground source is pre-treated 72 to removed water and carbon dioxide to produce stream 90 of dry, CO_2-free feed gas which is cooled by indirect heat exchange in the main heat exchanger 92. A first portion 191 of the cooled feed is expanded in expander 192 to produce a two phase stream 193 which is phase separated in separator 95. The liquid phase 197 is fed directly to an NGL recovery column 96. The vapor phase 195 is cooled in the main heat exchanger 92 and the cooled stream 194 is also fed to the NGL recovery column 96. A second portion of the cooled feed is further cooled in the main heat exchanger 92, expanded in valve 94 and fed to the column 96 as reflux stream 198.

[0129] The feeds to the NGL column 96 are separated into a C_{2+}-enriched bottoms liquid and C_{2+}-depleted overhead vapor. The bottoms liquid is reboiled with external heat in reboiler 98 to provide vapor for the separation, and an NGL stream 199 is removed. Further vapor (stream 402) for the column 96 is provided by reboiling a stream 400 of liquid taken from an intermediate location of the column 96 in the main heat exchanger 92.

[0130] A stream 100 of overhead vapor is cooled and condensed in the main heat exchanger 92 by indirect heat exchange against reboiling helium-free bottoms liquid 410 and expanded bottoms liquid 204 from the helium recovery column 102. The condensed feed 111 is then expanded across valve 112 and the expanded stream 113 fed to the helium recovery column 102 where it is separated into the helium-free bottoms liquid and helium-enriched overhead vapor.

[0131] A stream 114 of overhead vapor is fed to the main heat exchanger 92 where nitrogen in the stream in condensed to form a two phase stream 115 which is phase separated in separator 103. The liquid phase 120 is fed as reflux to the helium recovery column 102. The vapor phase 118 is used to help cool the feed 90 in the main heat exchanger 92 and the resultant warmed stream 119 is fed to a helium PSA unit 300 which produces a stream 302 of pure helium. A stream 304 of tail gas from the PSA unit 300 is compressed in compressor 306 and the compressed stream 308 is cooled by indirect heat exchange in aftercooler 310 and the main heat exchanger 92 before being recycled as stream 314 to the helium recovery column 102.

[0132] After cooling the feed to the helium recovery column 102, a portion of the expanded helium-free bottoms liquid is fed as stream 200 from the main heat exchanger to a first nitrogen purification column 208 where it is separated into methane-enriched bottoms liquid and nitrogen-enriched overhead vapor.

[0133] A stream 230 of nitrogen-enriched overhead vapor is condensed by indirect heat exchange in the main heat exchanger. A portion 234 of the condensed stream is fed to the first nitrogen purification column as reflux. The remaining portion is cooled by indirect heat exchange in the main heat exchanger 92 and the cooled stream 181 expanded across valve 182 to form two phase stream 184. Stream 184 is fed to a storage tank 185 from which a stream 186 of liquid nitrogen may be taken. Vapor stream 188 is warmed in the main heat exchanger 92 to produce nitrogen gas stream 190.

[0134] A stream 210 of methane-enriched bottoms liquid is expanded across valve 212 and expanded stream 214 is warmed and vaporized by indirect heat exchange in the main heat exchanger 92. Gaseous stream 138 is expanded in expander 140 and the expanded stream is fed to a second nitrogen purification column 258. Reflux to the second nitrogen purification column 258 is provided by a portion 252 of the expanded bottoms liquid 204 from the helium recovery column 102. Stream 252 is expanded across valve 254 and fed as reflux stream 256 to the column 258.

[0135] The feeds to the second nitrogen purification column are separated into methane-enriched bottoms liquid and nitrogen-enriched overhead vapor. The column is reboiled by vaporizing a stream 260 of bottoms liquid in the main heat exchanger 92. A stream 270 of bottoms liquid is pumped in pump 272 and pumped stream 274 is used to help cool the feed 90 in the main heat exchanger 92 to produce fuel gas stream 276.

[0136] A stream 226 of overhead vapor is warmed by indirect heat exchange in the main heat exchanger 92 and divided into two portions, streams 147 and 280. Stream 147 may be a product stream but it is usually vented. Stream 280 is compressed in compressor 282 and the compressed stream 284 is cooled in aftercooler 286. The cooled stream 288 is cooled in the main heat exchanger 92 before being combined with stream 214 after it has been vaporized to form combined stream 138 from the first nitrogen purification column 208 to the second nitrogen purification column 258.

[0137] Aspects of the present invention include:

#1. A process for recovering helium from a nitrogen-rich feed gas comprising helium, said process comprising:

cooling a pressurized nitrogen-rich feed gas comprising helium to produce cooled feed; and
separating said feed in a first distillation column system operating at an elevated operating pressure to produce helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);

wherein said feed to the first distillation column system is at least partially condensed; and
wherein said cooling of said feed gas is achieved by indirect heat exchange against at least a first nitrogen-enriched bottoms liquid at a first elevated pressure and a second nitrogen-enriched bottoms liquid at a second elevated pressure that is different from said first elevated pressure.

#2. A process according to aspect #1, wherein said first elevated pressure is equal to the elevated operating pressure of said first distillation column system.

#3. A process according to aspect #1 or aspect #2, wherein said first elevated pressure is from about 2 bar to about 35 bar.

#4. A process according to any of aspects #1 to #3, wherein the second elevated pressure is less than the elevated operating pressure of said first distillation column system.

#5. A process according to aspect #4, wherein the second elevated pressure is from about 1 bar to about 30 bar.

#6. A process according to aspect #4 or aspect #5 comprising expanding bottoms liquid to produce said second bottoms liquid.

#7. A process according to any of aspects #1 to #6, wherein said feed gas is at subcritical pressure.

#8. A process according to aspect #7, wherein said feed gas is cooled by indirect heat exchange against said first bottoms liquid and then at least partially condensed by indirect heat exchange against said second bottoms liquid.

#9. A process according to any of aspects #1 to #3, wherein the second elevated pressure is greater than the elevated operating pressure of said first distillation column system.

#10. A process according to aspect #9, wherein the second elevated pressure is from about 3 bar to about 150 bar.

#11. A process according to #9 or #10 comprising pumping bottoms liquid to produce said second bottoms liquid.

#12. A process according to any of aspects #1 to #3 and #9 to #11, wherein said feed gas is at supercritical pressure.

#13. A process according to aspect #12, wherein said feed gas is cooled by indirect heat exchange against said first bottoms liquid and then further cooled by indirect heat exchange against said second bottoms liquid.

#14. A process according to aspect #12, comprising expanding said cooled feed prior to feeding said cooled feed to said first distillation column system.

#15. A process according to any of aspects #1 to #14, wherein the feed to the first distillation column system has a vapor fraction of no more than 0.5.

#16. A process according to any of aspects #1 to #15, wherein the feed to the first distillation column system is at least substantially fully condensed.

#17. A process according to any of aspects #1 to #16, wherein said pressurized nitrogen-rich feed gas is at a pressure greater than said elevated operating pressure of said first distillation column, said process comprising expanding said at least partially condensed feed prior to separation.

#18. A process according to any of aspects #1 to #17, wherein the pressure of said pressurized nitrogen-rich feed gas is from about 2 bar to about 200 bar.

#19. A process according to any of aspects #1 to #18, wherein said elevated operating pressure of the distillation column system is from about 2 bar to about 35 bar.

#20. A process according to any of aspects #1 to #19 comprising expanding vaporized bottoms liquid, or a fluid derived therefrom, to produce expanded nitrogen-enriched gas and using said expanded gas to provide a part of the refrigeration duty of the process.

#21. A process according to any of aspects #1 to #20, wherein said second bottoms liquid is at least partially vaporized as a result of said indirect heat exchange against said feed gas, said process comprising:

warming said vaporized bottoms liquid by indirect heat exchange to produce warmed nitrogen-enriched gas; and

expanding said warmed nitrogen-enriched gas to produce expanded nitrogen-enriched gas; and

cooling said feed gas by indirect heat exchange with said expanded nitrogen-enriched gas to produce cooled feed gas.

#22. A process according to any of aspects #1 to #21 comprising:

expanding a third bottoms liquid to produce expanded nitrogen-enriched fluid;

vaporizing said expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce nitrogen-enriched gas;

expanding said nitrogen-enriched gas to produce expanded nitrogen-enriched gas; and

condensing nitrogen gas in said first distillation column system by indirect heat exchange against said expanded nitrogen-enriched gas to produce liquid reflux for said first distillation column system.

#23. A process according to aspect #22 comprising:

expanding a fourth bottoms liquid to produce further expanded nitrogen-enriched fluid; and

vaporizing said further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce further nitrogen-enriched gas.

#24. A process according to any of aspects #1 to #20, wherein said second bottoms liquid is vaporized as a result of said indirect heat exchange against said feed gas, said process comprising:

expanding said vaporized bottoms liquid to produce expanded nitrogen-enriched gas; and

condensing nitrogen gas in said first distillation column system by indirect heat exchange with said expanded nitrogen-enriched gas to produce liquid reflux for said first distillation column system and warmed nitrogen-enriched gas.

#25. A process according to #24 comprising:

expanding a third bottoms liquid to produce further expanded nitrogen-enriched fluid; and

vaporizing said further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce further nitrogen-enriched gas.

#26. A process according to aspect #24 or aspect #25 comprising expanding a fourth bottoms liquid to form an expanded fluid; and separating said expanded fluid into a vapor phase and a liquid phase.

#27. A process according to aspect #26, wherein said vapor phase is warmed by indirect heat exchange to produce a gaseous nitrogen product.

#28. A process according to aspect #26 of #27, comprising sub-cooling said fourth bottoms liquid prior to expansion to form said expanded two phase fluid.

#29. A process according to aspect #28, wherein said fourth bottoms liquid is sub-cooled by indirect heat exchange against expanded nitrogen-enriched gas

#30. A process according to any of aspects #1 to #29, wherein the process is autorefrigerated.

#31. A process according to any of aspects #1 to #30, wherein said feed gas is taken from a natural gas liquids (NGL) recovery column.

#32. A process according to any of aspects #1 to #31 comprising:

cooling and at least partially condensing pressurized dry, carbon dioxide-free natural gas to produce at least partially condensed natural gas; and

separating said at least partially condensed natural gas in a second distillation column system operating at an elevated operating pressure to produce C_{2+}-depleted overhead vapor and C_{2+}-enriched bottoms liquid,

wherein said C_{2+}-depleted overhead vapor is said nitrogen-rich feed gas.

#33. A process according to aspect #32, wherein said natural gas comprises at least 70% nitrogen (N2).

#34. A process according to aspect #32 or aspect #33, wherein said pressurized dry, carbon dioxide-free natural gas is at a pressure of from about 2 bar to about 200 bar.

#35. A process according to any of aspects #32 to #34, wherein said elevated pressure of said second distillation column system is less than the pressure of said pressurized dry, carbon dioxide-free natural gas, said process comprising expanding said at least partially condensed natural gas to said elevated pressure of said second distillation column system prior to separation.

#36. A process according to any of aspects #32 to #34, wherein said elevated operating pressure of said second distillation column system is from about 2 bar to about 35 bar.

#37. A process according to any of aspects #1 to #36 comprising:

warming said helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched gas; and

purifying said helium-enriched gas to produce pure helium gas.

#38. A process according to aspect #37, wherein said helium-enriched gas is purified by a pressure swing adsorption (PSA) process.

#39. A process according to aspect #38, wherein tail gas from said PSA process is recycled to said first distillation column system after suitable pressure and temperature adjustment.

#40. A process according to any of aspects #1 to #39, wherein said feed gas comprises methane, said process comprising recovering methane from said bottoms liquid to produce fuel gas or LNG.

#41. A process according to any of aspects #1 to #40 comprising separating bottoms liquid in a third distillation column system operating at elevated operating pressure(s) to produce nitrogen overhead vapor and methane-enriched bottoms liquid.

#42. A process according to aspect #41 comprising warming said nitrogen overhead vapor by indirect heat exchange to produce warmed nitrogen gas.

#43. A process according to aspect #41 or aspect #42 comprising recycling a portion of said nitrogen gas to said third distillation column system after suitable pressure and temperature adjustment.

#44. A process according to aspect #42 comprising expanding a portion of said nitrogen gas to produce expanded nitrogen gas, and warming said expanded nitrogen gas by indirect heat exchange to produce warmed expanded nitrogen gas.

#45. A process according to any of aspects #41 to #44 comprising warming and evaporating said methane-enriched bottoms liquid by indirect heat exchange to produce fuel gas.

#46. A process according to any of aspects #41 to #45, wherein said third distillation column system additionally produces a nitrogen-enriched overhead vapor, said process comprising condensing said nitrogen-enriched overhead vapor to produce condensed vapor and removing a portion of said condensed vapor as a liquid nitrogen product.

#47. A process according to any of aspects #41 to #46, wherein the or each elevated pressure of said third distillation column system is less than the pressure of the bottoms liquid, said process comprising expanding said bottoms liquid to the or one of the elevated pressures of said third distillation column system prior to separation.

#48. A process according to any of aspects #41 to #47, wherein said elevated pressure of said third distillation column system is from more than 1 bar to about 35 bar.

#49. A process substantially as described herein with reference to the examples and/or drawings.

#50. Apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);

an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;

a first heat exchange system for cooling feed gas by indirect heat exchange with a first nitrogen-enriched bottoms liquid to produce cooled feed gas and vapor for the column system;

a first pressure reduction device for reducing the pressure of a second nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid;

a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against said reduced pressure bottoms liquid to produce at least partially condensed feed gas and vaporized bottoms liquid; and

a second pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

#51. Apparatus according to aspect #50 comprising a third pressure reduction device for reducing the pressure of a third nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

#52. Apparatus according to aspect #51 comprising an expander for expanding said nitrogen-enriched vapor to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

#53. Apparatus according to aspect #51 or aspect #52 comprising a fourth pressure reduction device for reducing the pressure of a fourth nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

#54. Apparatus according to #50 comprising an expander for expanding said vaporized bottoms liquid to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

#55. Apparatus according to #54 comprising:

a fourth pressure reduction device for reducing the pressure of a fourth nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid; and

a storage vessel for storing said reduced pressure bottoms liquid.

#56. Apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);

an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;

a first pump for pumping a second nitrogen-enriched bottoms liquid to produce pumped bottoms liquid;

a first heat exchange system for cooling feed gas by indirect heat exchange with said pumped bottoms liquid to produce cooled feed gas and nitrogen-enriched vapor;

a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against a first nitrogen-enriched bottoms liquid to produce at least partially condensed feed gas and vapor for the column system; and

a pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

#57. Apparatus according to aspect #56 comprising a second pressure reduction device for reducing the pressure of a third nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

#58. Apparatus according to aspect #57 comprising an expander for expanding said nitrogen-enriched vapor to produce expanded nitrogen-enriched vapor for warming by indirect heat exchange in said overhead condenser to produce warmed nitrogen-enriched vapor.

#59. Apparatus according to aspect #57 or aspect #58 comprising a third pressure reduction device for reducing the pressure of a fourth nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid for vaporization by indirect heat exchange in said overhead condenser to produce nitrogen-enriched vapor.

#60. Apparatus substantially as described herein with reference to the accompanying examples and/or drawings.

COMPARATIVE EXAMPLE 1

[0138] A computer simulation of the process depicted in FIG. 1 has been carried out using Aspen Plus (version 7.2, ©Aspen Technology Inc.). The resultant heat and mass balance data for the key streams is presented in Table 1.

TABLE 1

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-144.5	-151.8	-191.4		-147.3		-154.5	-189.3	45.2	
Pressure	bar	30.0	30.0	25.0	1.5		30.0		25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.272	0.000	0.278	0.000	0.169	0.006	0.006	
Vapor Fraction		1.00	1.00	0.00	0.49		0.89		1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300		0.9590	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500		0.0020	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200		0.0390	0.9000	0.9000	

		126	128
Temperature	C	-184.1	45.2
Pressure	bar	1.5	1.5
Molar Flow	kmol/s	0.272	0.272
Vapor Fraction		1.00	1.00
Mole fraction Nitrogen		0.9489	0.9489
Mole fraction Methane		0.0511	0.0511
Mole fraction Helium		0.0000	0.0000

Product recompression	2899	kW
Total	2899	kW

[0139] The power to recompress the product 128 to the feed pressure of 30 bar is 2899 kW.

EXAMPLE 1

[0140] A computer simulation of the process depicted in FIG. 2 has been carried out using Aspen Plus. The heat and mass balance data for the key streams is presented in Table 2.

TABLE 2

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-142.8	-151.8	-191.4		-147.3	-155.5	-158.8	-189.3	46.9	
Pressure	bar	30.0	30.0	25.0	1.5		30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.036		0.278	0.278	0.031	0.006	0.006	0.000
Vapour Fraction		1.00	1.00	0.00	0.49		0.90	0.04	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300	0.9300	0.8150	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500	0.0500	0.0009	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200	0.0200	0.1841	0.9000	0.9000	
		126	128	134	138	142	144	164	168	170	172	
Temperature	C	-160.8	46.9					-151.8	-157.5	-154.4	46.9	
Pressure	bar	1.5	1.5					25.0	18.6	18.6	18.2	
Molar Flow	kmol/s	0.036	0.036					0.236	0.236	0.236	0.236	
Vapour Fraction		1.00	1.00					0.00	0.14	1.00	1.00	
Mole fraction Nitrogen		0.9489	0.9489					0.9489	0.9489	0.9489	0.9489	
Mole fraction Methane		0.0511	0.0511					0.0511	0.0511	0.0511	0.0511	
Mole fraction Helium		0.0000	0.0000					0.0000	0.0000	0.0000	0.0000	
Product recompression	806	kW										
Expander power	-10	kW										
Total	796	kW										

[0141] In this example, only 13% of the helium-free product stream is boiled at low pressure in the column condenser. Product stream 170 is boiled at 18.6 bar and expanded to 18.2 bar in expander 174. The total power (mostly product recompression power for streams 128 and 172) is reduced by 73% from 2899 kW to 796 kW. In addition, because the vapour fraction of the feed is reduced, the vapor flow in the column above the feed location is reduced significantly leading to a reduction in the column diameter.

EXAMPLE 2

[0142] A computer simulation of the process depicted in FIG. 3 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 3.

TABLE 3

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-140.7	-151.8	-191.4		-147.2	-158.9	-161.9	-189.3	46.8	
Pressure	bar	30.0	30.0	25.0	1.5		30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.007		0.278	0.278	0.019	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00	0.49		0.92	0.02	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300	0.9300	0.7038	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500	0.0500	0.0005	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200	0.0200	0.2957	0.9000	0.9000	
		126	128	134	138	142	144	146	164	168	170	172
Temperature	C	-163.3	46.8	-174.8	-163.3	-187.1	-163.3	46.8	-151.8	-160.9	-155.5	46.8
Pressure	bar	1.5	1.5	6.4	6.4	1.5	1.5	1.5	25.0	15.4	15.4	15.4
Molar Flow	kmol/s	0.007	0.007	0.010	0.010	0.010	0.010	0.010	0.254	0.254	0.254	0.254
Vapour Fraction		1.00	1.00	0.36	1.00	0.96	1.00	1.00	0.00	0.20	1.00	1.00
Mole fraction Nitrogen		0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489
Mole fraction Methane		0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Product recompression	788	kW										
Expander power	-8	kW										
Total	779	kW										

[0143] The advantage in this example is that the refrigeration is integrated with the separation process, and the amount of product available at pressure is increased - only 6.3% of the product is at low pressure. The total power consumption is also slightly reduced (by 2.1 %) to 779 kW.

EXAMPLE 3

[0144] A computer simulation of the process depicted in FIG. 4 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 4.

TABLE 4

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-134.1	-151.8	-191.4		-144.0	-149.9	-157.2	-189.3	46.9	
Pressure	bar	50.0	50.0	25.0	1.5		50.0	50.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.008		0.278	0.278	0.045	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00	0.49		1.00	0.00	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300	0.9300	0.8711	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500	0.0500	0.0013	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200	0.0200	0.1276	0.9000	0.9000	

		126	128	134	138	142	144	146	164	168	170	172
Temperature	C	-159.3	46.9	-172.2	-159.3	-187.3	-159.3	46.9	-151.8	-149.2	-140.0	46.9
Pressure	bar	1.5	1.5	7.7	7.7	1.5	1.5	1.5	25.0	39.5	39.5	39.5
Molar Flow	kmol/s	0.008	0.008	0.038	0.038	0.038	0.038	0.038	0.226	0.226	0.226	0.226
Vapour Fraction		1.00	1.00	0.34	1.00	0.95	1.00	1.00	0.00	0.00	1.00	1.00
Mole fraction Nitrogen		0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489
Mole fraction Methane		0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Product recompression	765	kW
Expander power	-35	kW
Pump power	28	kW
Total	757	kW

[0145] In this case, part of the helium-free product 164 is increased in pressure in a pump 165 and used to cool the feed in heat exchanger 169 upstream of the column reboiler 108. The refrigeration provided by the expander offsets the energy input to the process in the pump. In the example, the total power including recompression of the products back to the feed pressure (50 bar in this case) is 757 kW.

EXAMPLE 4

[0146] A computer simulation of the process depicted in FIG. 5 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 5.

TABLE 5

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-122.4	-151.8	-191.4		-134.2	-150.9	-156.1	-189.3	46.9	
Pressure	bar	30.0	30.0	25.0	1.5		30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.005		0.278	0.278	0.066	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00	0.49		1.00	0.18	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300	0.9300	0.9084	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500	0.0500	0.0017	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200	0.0200	0.0899	0.9000	0.9000	
		126	128	134	138	142	144	146	164	168	170	172
Temperature	C	-158.1	46.9	-156.3	-136.2	-188.3	-158.1	46.9				
Pressure	bar	1.5	1.5	19.8	19.8	1.5	1.5	1.5				
Molar Flow	kmol/s	0.005	0.005	0.215	0.215	0.215	0.215	0.215				
Vapour Fraction		1.00	1.00	0.12	1.00	0.93	1.00	1.00				
Mole fraction Nitrogen		0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489				
Mole fraction Methane		0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511				
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000				
		180	181	186	188	190						
Temperature	C	-151.8		-194.2	-194.2	46.9						
Pressure	bar	25.0		1.1	1.1	1.1						
Molar Flow	kmol/s	0.052		0.025	0.027	0.027						
Vapour Fraction		0.00		0.00	1.00	1.00						
Mole fraction Nitrogen		0.9489		0.8994	0.9957	0.9957						
Mole fraction Methane		0.0511		0.1006	0.0043	0.0043						
Mole fraction Helium		0.0000		0.0000	0.0000	0.0000						
Product recompression	2957	kW										
Expander power	-316	kW										
Total	2640	kW										
Liquid production (1.1 bara)	59	TPD										
Power with no liquid (Example 3)	779	kW										

(continued)

		180	181	186	188	190
Additional power for Liquid	1861	kW				
Liquid specific power	762	kWh/t				

[0147] In the example, 9.2% of the product is produced as saturated liquid at 1.1 bar. The total power including product recompression to 30 bar and net of the power generation from the expander of 316 kW is 2342 kW. 59 tonnes (t) per day of liquid is produced. Compared to Example 2, the total power is 1861 kW higher for the production of 59 tonnes per day liquid, meaning that the specific power for the liquid production is 762 kWh/t.

EXAMPLE 5

[0148] A computer simulation of the process depicted in FIG. 6 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 6.

TABLE 6

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-117.1	-151.8	-191.4		-130.0	-151.5	-156.4	-189.3	46.9	
Pressure	bar	30.0	30.0	25.0	1.5		30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272	0.005		0.278	0.278	0.059	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00	0.49		1.00	0.14	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489	0.9489		0.9300	0.9300	0.8983	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511	0.0511		0.0500	0.0500	0.0016	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000	0.0000		0.0200	0.0200	0.1001	0.9000	0.9000	
		126	128	134	138	142	144	146	164	168	170	172
Temperature	C	-156.5	46.9	-154.0	-132.0	-188.3	-156.5	46.9				
Pressure	bar	1.5	1.5	22.4	22.4	1.5	1.5	1.5				
Molar Flow	kmol/s	0.005	0.005	0.235	0.235	0.235	0.235	0.235				
Vapour Fraction		1.00	1.00	0.07	1.00	0.93	1.00	1.00				
Mole fraction Nitrogen		0.9489	0.9489	0.9489	0.9489	0.9489	0.9489	0.9489				
Mole fraction Methane		0.0511	0.0511	0.0511	0.0511	0.0511	0.0511	0.0511				
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000				
		180	181	186	188	190						
Temperature	C	-151.8	-186.8	-194.6	-194.6	46.9						
Pressure	bar	25.0	25.0	1.1	1.1	1.1						
Molar Flow	kmol/s	0.032	0.032	0.029	0.003	0.003						
Vapour Fraction		0.00	0.00	0.00	1.00	1.00						
Mole fraction Nitrogen		0.9489	0.9489	0.9442	0.9976	0.9976						
Mole fraction Methane		0.0511	0.0511	0.0558	0.0024	0.0024						
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000						
Product recompression	2935	kW										
Expander power	-364	kW										
Pump power		kW										
Total	2570	kW										
Liquid production (1.1 bara)	69	TPD										
Power with no liquid (Example 3)	779	kW										

(continued)

		180	181	186	188	190
Additional power for Liquid	1791	kW				
Liquid specific power	619	kWh/t				

[0149] In this case, 10.7% of the product is produced as saturated liquid at 1.1 bar. The total power including product recompression to 30 bar and net of the power generation from the expander of 364 kW is 2570 kW. 69 tonnes per day of liquid is produced. Compared to Example 2, the total power is 1791 kW higher for the production of 69 tonnes per day liquid, meaning that the specific power for the liquid production is 619 kWh/t.

EXAMPLE 6

[0150] A computer simulation of the process depicted in FIG. 7 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 7.

TABLE 7

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-144.7	-151.7	-191.4		-147.3	-154.9	-158.5	-189.3	47.0	
Pressure	bar	50.0	30.0	25.0	1.5		30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.275	0.269	0.025		0.275	0.275	0.033	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00	0.49		0.85	0.05	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9200	0.9283	0.9473	0.9473		0.9283	0.9283	0.8271	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0504	0.0515	0.0515		0.0504	0.0504	0.0010	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0202	0.0000	0.0000		0.0202	0.0202	0.1719	0.9000	0.9000	
Mole fraction Ethane		0.0080	0.0011	0.0012	0.0012		0.0011	0.0011	0.0000	0.0000	0.0000	
Mole fraction Propane		0.0020	0.0000	0.0000	0.0000		0.0000	0.0000	0.0000	0.0000	0.0000	
		126	128	134	138	142	144	146	164	168	170	172
Temperature	C	-157.0	47.0	-156.9	-109.5	-181.0	-157.0	47.0				
Pressure	bar	1.5	1.5	19.2	19.2	1.5	1.5	1.5				
Molar Flow	kmol/s	0.025	0.025	0.200	0.200	0.200	0.200	0.200				
Vapour Fraction		1.00	1.00	0.13	1.00	1.00	1.00	1.00				
Mole fraction Nitrogen		0.9473	0.9473	0.9473	0.9473	0.9473	0.9473	0.9473				
Mole fraction Methane		0.0515	0.0515	0.0515	0.0515	0.0515	0.0515	0.0515				
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000				
Mole fraction Ethane		0.0012	0.0012	0.0012	0.0012	0.0012	0.0012	0.0012				
Mole fraction Propane		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000				
		180	181	186	188	190	191	193	194	196	199	
Temperature	C	-151.7	-187.3	-194.6	-194.6	47.0	-37.7	-65.7	-142.9	-150.7	18.5	
Pressure	bar	25.0	25.0	1.1	1.1	1.1	50.0	30.0	30.0	50.0	30.0	
Molar Flow	kmol/s	0.044	0.044	0.040	0.004	0.004	0.262	0.262	0.262	0.015	0.002	
Vapour Fraction		0.00	0.00	0.00	1.00	1.00	1.00	1.00	0.99	0.00	0.00	
Mole fraction Nitrogen		0.9473	0.9473	0.9428	0.9976	0.9976	0.9200	0.9200	0.9200	0.9200	0.0000	
Mole fraction Methane		0.0515	0.0515	0.0559	0.0024	0.0024	0.0500	0.0500	0.0500	0.0500	0.0100	
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0200	0.0200	0.0200	0.0200	0.0000	
Mole fraction Ethane		0.0012	0.0012	0.0013	0.0000	0.0000	0.0080	0.0080	0.0080	0.0080	0.7685	
Mole fraction Propane		0.0000	0.0000	0.0000	0.0000	0.0000	0.0020	0.0020	0.0020	0.0020	0.2215	
Product recompression	3411	kW										

(continued)

		180	181	186	188	190	191	193	194	196	199
Expander power	-363	kW									
Warm expander power	-183	kW									
Total	2864	kW									
Liquid production (1.1 bara)	94	TPD									
Power with no liquid (Example 4)	757	kW									
Additional power for Liquid	2107	kW									
Liquid specific power	537	kWh/T									

[0151] In this case, the feed is at 50 bar as in Example 3, but is expanded in warm expander 192 prior to separation in NGL recovery column 96. A small part of the high pressure feed is condensed and fed to the top of the NGL recovery column as reflux. This column is reboiled with an external heat source such as steam, hot oil or cooling water. The liquid production is higher than in Example 5 because of the higher feed pressure and additional refrigeration provided by the warm expander. The specific power for liquid production is also lower as the warm and cold expander system provides refrigeration more efficiently than a single expander. The total power including product recompression to 50 bar and net of the power generation from the expanders of 546 kW is 2864 kW. 94 tonnes per day of liquid is produced. Compared to Example 3 (which also has a feed pressure of 50 bar), the total power is 2107 kW higher for the production of 94 tonnes per day liquid, meaning that the specific power for the liquid production is 537 kWh/t.

EXAMPLE 7

[0152] A computer simulation of the process depicted in FIG. 8 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 8.

TABLE 8

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-144.6	-151.8			-147.5	-155.7	-159.0	-189.3	46.9	
Pressure	bar	30.0	30.0	25.0			30.0	30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.278	0.272			0.278	0.278	0.030	0.006	0.006	
Vapour Fraction		1.00	1.00	0.00			0.85	0.04	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9300	0.9300	0.9489			0.9300	0.9300	0.8090	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0500	0.0511			0.0500	0.0500	0.0009	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0200	0.0000			0.0200	0.0200	0.1902	0.9000	0.9000	

		126	128	134	138	142	144	146	164	168	170	172
Temperature	C			-151.8	-116.3	-183.7	-155.6	46.9				
Pressure	bar			25.0	18.2	1.5	1.5	1.5				
Molar Flow	kmol/s			0.239	0.208	0.208	0.227	0.227				
Vapour Fraction				0.00	1.00	0.99	1.00	1.00				
Mole fraction Nitrogen				0.9489	0.9413	0.9413	0.9947	0.9947				
Mole fraction Methane				0.0511	0.0587	0.0587	0.0053	0.0053				
Mole fraction Helium				0.0000	0.0000	0.0000	0.0000	0.0000				

		180	181	186	188	190	191	193	194	196	199	200
Temperature	C	-153.2	-188.2	-195.1	-195.1	46.9						-151.6
Pressure	bar	25.0	25.0	1.1	1.1	1.1						25.0
Molar Flow	kmol/s	0.031	0.031	0.029	0.002	0.002						0.239
Vapour Fraction		0.00	0.00	0.00	1.00	1.00						0.22
Mole fraction Nitrogen		0.9990	0.9990	0.9989	0.9999	0.9999						0.9489
Mole fraction Methane		0.0010	0.0010	0.0011	0.0000	0.0000						0.0511
Mole fraction Helium		0.0000	0.0000	0.0000	0.0001	0.0001						0.0000

		210	222	226	250	252	270	274	276
Temperature	C	-151.6	-150.3	-191.3	-151.8	-188.5	-167.1	-166.3	46.9
Pressure	bar	25.0	18.2	1.5	25.0	25.0	1.5	6.0	6.0
Molar Flow	kmol/s	0.208	0.208	0.227	0.033	0.033	0.013	0.013	0.013
Vapour Fraction		0.00	1.00	1.00	0.00	0.00	0.00	0.00	1.00
Mole fraction Nitrogen		0.9413	0.9413	0.9947	0.9489	0.9489	0.0500	0.0500	0.0500
Mole fraction Methane		0.0587	0.0587	0.0053	0.0511	0.0511	0.9500	0.9500	0.9500

(continued)

		210	222	226	250	252	270	274	276
Mole fraction Helium		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Product recompression	2868	kW
Expander power	-354	kW
Pump power	1	kW
Total	2515	kW
Liquid production (1.1 bara)	70	TPD
Power with no liquid (Example 3)	779	kW
Additional power for Liquid	1736	kW
Liquid specific power	596	kWh/T

[0153] In this case, the total power including product recompression to 30 bar and net of the power generation from the expander of 354 kW is 2515 kW. 70 tonnes per day of liquid is produced. Compared to Example 2, the total power is 1736 kW higher for the production of 70 tonnes per day liquid, meaning that the specific power for the liquid production is 596 kWh/t.

EXAMPLE 8

[0154] A computer simulation of the process depicted in FIG. 9 has been carried out using Aspen Plus. The resultant heat and mass balance data for the key streams is presented in Table 9.

TABLE 9

		90	100	104	105	106	110	111	114	118	119	124
Temperature	C	48.9	-145.4	-151.8				-155.6	-159.9	-189.3	46.9	
Pressure	bar	50.0	30.0	25.0				30.0	25.0	25.0	25.0	
Molar Flow	kmol/s	0.278	0.275	0.270				0.275	0.032	0.008	0.008	
Vapour Fraction		1.00	1.00	0.00				0.04	1.00	1.00	1.00	
Mole fraction Nitrogen		0.9200	0.9286	0.9477				0.9286	0.7784	0.1000	0.1000	
Mole fraction Methane		0.0500	0.0504	0.0514				0.0504	0.0008	0.0000	0.0000	
Mole fraction Helium		0.0200	0.0202	0.0000				0.0202	0.2208	0.9000	0.9000	
Mole fraction Ethane		0.0080	0.0008	0.0008				0.0008	0.0000	0.0000	0.0000	
Mole fraction Propane		0.0020	0.0000	0.0000				0.0000	0.0000	0.0000	0.0000	

		126	128	134	138	142	144	146	164	168	170	172
Temperature	C				-123.0	-184.9		46.9				
Pressure	bar				18.2	1.5		1.5				
Molar Flow	kmol/s				0.227	0.227		0.243				
Vapour Fraction					1.00	0.98		1.00				
Mole fraction Nitrogen					0.9442	0.9442		0.9947				
Mole fraction Methane					0.0549	0.0549		0.0053				
Mole fraction Helium					0.0000	0.0000		0.0000				
Mole fraction Ethane					0.0009	0.0009		0.0000				
Mole fraction Propane					0.0000	0.0000		0.0000				

		180	181	186	188	190	191	193	194	196	199	200
Temperature	C		-189.2	-195.1	-195.1	46.9	-38.6	-66.6	-143.6	-160.0	18.2	-151.4
Pressure	bar		25.0	1.1	1.1	1.1	50.0	30.0	30.0	50.0	30.0	25.0
Molar Flow	kmol/s		0.044	0.041	0.003	0.003	0.264	0.264	0.264	0.014	0.003	0.241
Vapour Fraction			0.00	0.00	1.00	1.00	1.00	1.00	0.98	0.00	0.00	0.30
Mole fraction Nitrogen			0.9990	0.9989	0.9999	0.9999	0.9200	0.9200	0.9200	0.9200	0.0000	0.9477
Mole fraction Methane			0.0010	0.0011	0.0000	0.0000	0.0500	0.0500	0.0500	0.0500	0.0100	0.0514
Mole fraction Helium			0.0000	0.0000	0.0001	0.0001	0.0200	0.0200	0.0200	0.0200	0.0000	0.0000
Mole fraction Ethane			0.0000	0.0000	0.0000	0.0000	0.0080	0.0080	0.0080	0.0080	0.7759	0.0008
Mole fraction Propane			0.0000	0.0000	0.0000	0.0000	0.0020	0.0020	0.0020	0.0020	0.2141	0.0000

(continued)

		210	222	226	250	252	270	274	276	280	288	302
Temperature	C	-151.4		-191.3		-189.0	-164.1	-163.2	46.9	46.9	40.0	46.9
Pressure	bar	25.0		1.5		25.0	1.5	6.0	6.0	1.5	18.2	25.0
Molar Flow	kmol/s	0.196		0.243		0.029	0.013	0.013	0.013	0.031	0.031	0.006
Vapour Fraction		0.00		1.00		0.00	0.00	0.00	1.00	1.00	1.00	1.00
Mole fraction Nitrogen		0.9362		0.9947		0.9477	0.0330	0.0330	0.0330	0.9947	0.9947	0.0000
Mole fraction Methane		0.0628		0.0053		0.0514	0.9500	0.9500	0.9500	0.0053	0.0053	0.0000
Mole fraction Helium		0.0000		0.0000		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000
Mole fraction Ethane		0.0010		0.0000		0.0008	0.0167	0.0167	0.0167	0.0000	0.0000	0.0000
Mole fraction Propane		0.0000		0.0000		0.0000	0.0003	0.0003	0.0003	0.0000	0.0000	0.0000
		304	312	314								
Temperature	C	46.9	40.0	-150.0								
Pressure	bar	1.3	25.0	25.0								
Molar Flow	kmol/s	0.002	0.002	0.002								
Vapour Fraction		1.00	1.00	1.00								
Mole fraction Nitrogen		0.3571	0.3571	0.3571								
Mole fraction Methane		0.0000	0.0000	0.0000								
Mole fraction Helium		0.6429	0.6429	0.6429								
Mole fraction Ethane		0.0000	0.0000	0.0000								
Mole fraction Propane		0.0000	0.0000	0.0000								
Product recompression					3741						kW	
Expander power					-369						kW	
Warm expander power					-183						kW	
Tail gas compression					29						kW	
Recycle compression					332						kW	
Pump power					1						kW	
Total					3550						kW	
Liquid production (1.1 bara)					100						TPD	
Power with no liquid (Example 4)					757						kW	
Additional power for					2793						kW	

(continued)

	304	312	314		
Liquid					
Liquid specific power				670	kWh/T

[0155] The total power including recycle and tail gas compression as well as product recompression to 50 bar and net of the power generation from the expanders of 552 kW is 3550 kW. 100 tonnes per day of liquid is produced. Compared to Example 3 (which also has a feed pressure of 50 bar), the total power is 2793 kW higher for the production of 100 tonnes per day liquid, meaning that the specific power for the liquid production is 670 kWh/t.

[0156] While the invention has been described with reference to the preferred embodiments depicted in the figures, it will be appreciated that various modifications are possible within the spirit or scope of the invention.

[0157] In this specification, unless expressly otherwise indicated, the word 'or' is used in the sense of an operator that returns a true value when either or both of the stated conditions are met, as opposed to the operator 'exclusive or' which requires only that one of the conditions is met. The word 'comprising' is used in the sense of 'including' rather than to mean 'consisting of'. All prior teachings above are hereby incorporated herein by reference. No acknowledgement of any prior published document herein should be taken to be an admission or representation that the teaching thereof was common general knowledge in Australia or elsewhere at the date thereof.

Claims

1. A process for recovering helium from a nitrogen-rich feed gas comprising helium, said process comprising:

cooling a pressurized nitrogen-rich feed gas comprising helium to produce cooled feed; and
separating said feed in a first distillation column system operating at an elevated operating pressure to produce helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);

wherein said feed to the first distillation column system is at least partially condensed; and
wherein said cooling of said feed gas is achieved by indirect heat exchange against at least a first nitrogen-enriched bottoms liquid at a first elevated pressure and a second nitrogen-enriched bottoms liquid at a second elevated pressure that is different from said first elevated pressure.

2. A process according to Claim 1, wherein said first elevated pressure is equal to the elevated operating pressure of said first distillation column system.

3. A process according to Claim 1 or Claim 2, wherein said first elevated pressure is from 2 bar to 35 bar.

4. A process according to any of Claims 1 to 3, wherein the second elevated pressure is less than the elevated operating pressure of said first distillation column system.

5. A process according to any of Claims 1 to 3, wherein the second elevated pressure is greater than the elevated operating pressure of said first distillation column system.

6. A process according to any of the preceding claims, wherein the feed to the first distillation column system has a vapor fraction of no more than 0.5.

7. A process according to any of the preceding claims, wherein the feed to the first distillation column system is at least substantially fully condensed.

8. A process according to any of the preceding claims, wherein said elevated operating pressure of the distillation column system is from 2 bar to 35 bar.

9. A process according to any of of the preceding claims comprising expanding vaporized bottoms liquid, or a fluid derived therefrom, to produce expanded nitrogen-enriched gas and using said expanded gas to provide a part of the refrigeration duty of the process.

10. A process according to any of the preceding claims, wherein said second bottoms liquid is at least partially vaporized as a result of said indirect heat exchange against said feed gas, said process comprising:

warming said vaporized bottoms liquid by indirect heat exchange to produce warmed nitrogen-enriched gas; and
expanding said warmed nitrogen-enriched gas to produce expanded nitrogen-enriched gas; and
cooling said feed gas by indirect heat exchange with said expanded nitrogen-enriched gas to produce cooled feed gas.

11. A process according to any of of the preceding claims comprising:

expanding a third bottoms liquid to produce expanded nitrogen-enriched fluid;
vaporizing said expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce nitrogen-enriched gas;
expanding said nitrogen-enriched gas to produce expanded nitrogen-enriched gas;
and
condensing nitrogen gas in said first distillation column system by indirect heat exchange against said expanded nitrogen-enriched gas to produce liquid reflux for said first distillation column system.

12. A process according to Claim 11 comprising:

expanding a fourth bottoms liquid to produce further expanded nitrogen-enriched fluid; and
vaporizing said further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce further nitrogen-enriched gas.

13. A process according to any of Claims 1 to 9, wherein said second bottoms liquid is vaporized as a result of said indirect heat exchange against said feed gas, said process comprising:

expanding said vaporized bottoms liquid to produce expanded nitrogen-enriched gas; and
condensing nitrogen gas in said first distillation column system by indirect heat exchange with said expanded nitrogen-enriched gas to produce liquid reflux for said first distillation column system and warmed nitrogen-enriched gas.

14. A process according to Claim 13 comprising:

expanding a third bottoms liquid to produce further expanded nitrogen-enriched fluid; and
vaporizing said further expanded nitrogen-enriched fluid by indirect heat exchange against condensing nitrogen in said first distillation column system to produce further nitrogen-enriched gas.

15. A process according to any of the preceding claims, wherein said feed gas is taken from a natural gas liquids (NGL) recovery column.

16. A process according to any of the preceding claims comprising:

cooling and at least partially condensing pressurized dry, carbon dioxide-free natural gas to produce at least partially condensed natural gas; and
separating said at least partially condensed natural gas in a second distillation column system operating at an elevated operating pressure to produce C_{2+}-depleted overhead vapor and C_{2+}-enriched bottoms liquid,

wherein said C_{2+}-depleted overhead vapor is said nitrogen-rich feed gas.

17. A process according to Claim 16, wherein said natural gas comprises at least 70% nitrogen (N_2).

18. A process according to any of the preceding claims comprising:

warming said helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched gas; and
purifying said helium-enriched gas to produce pure helium gas.

19. A process according to Claim 18, wherein said helium-enriched gas is purified by a pressure swing adsorption (PSA) process.

20. A process according to Claim 19, wherein tail gas from said PSA process is recycled to said first distillation column system after suitable pressure and temperature adjustment.

21. A process according to any of the preceding claims, wherein said feed gas comprises methane, said process comprising recovering methane from said bottoms liquid to produce fuel gas or LNG.

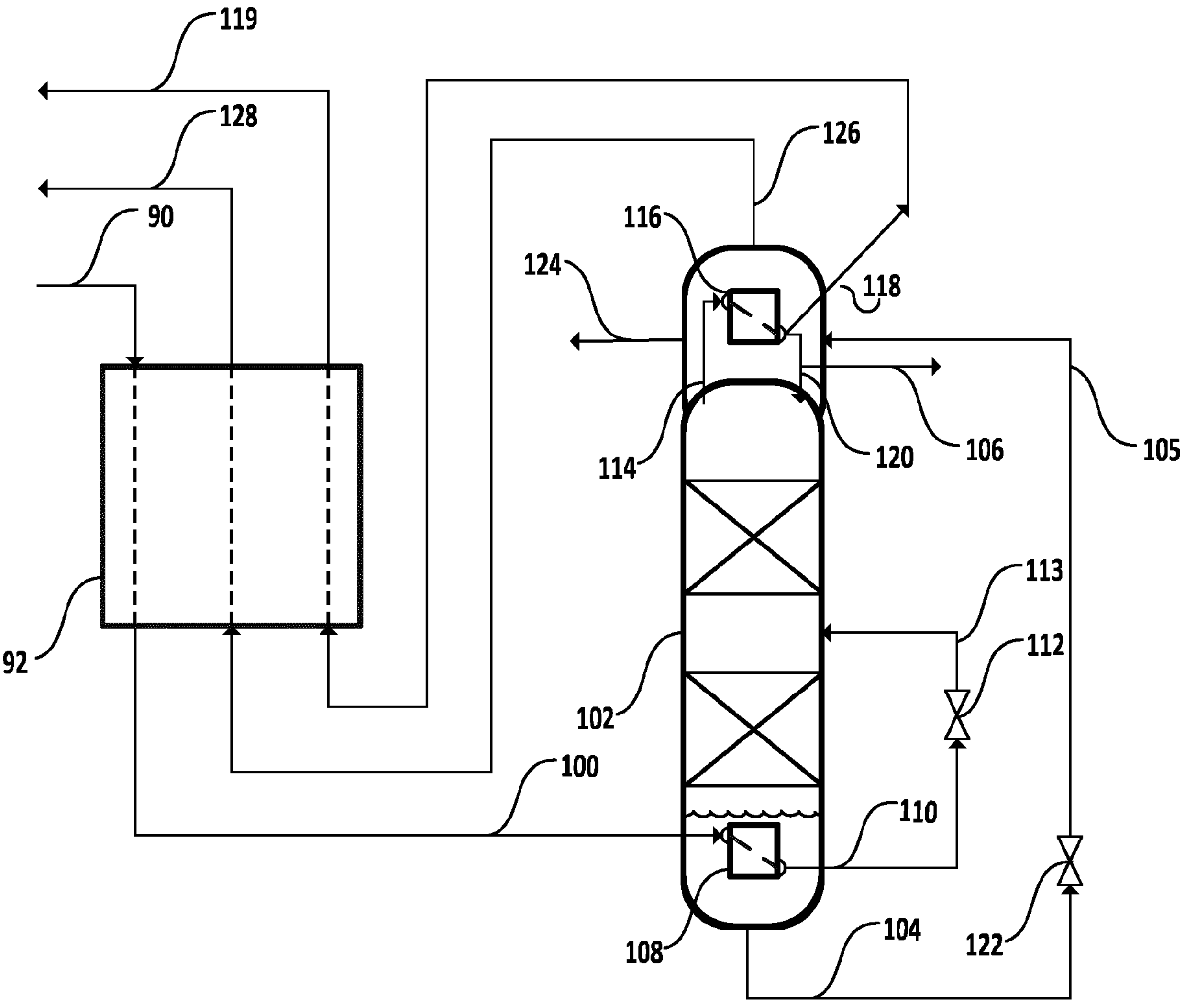
22. A process according to any of the preceding claims comprising separating bottoms liquid in a third distillation column system operating at elevated operating pressure(s) to produce nitrogen overhead vapor and methane-enriched bottoms liquid.

23. Apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);
an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;
a first heat exchange system for cooling feed gas by indirect heat exchange with a first nitrogen-enriched bottoms liquid to produce cooled feed gas and vapor for the column system;
a first pressure reduction device for reducing the pressure of a second nitrogen-enriched bottoms liquid to produce reduced pressure bottoms liquid;
a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against said reduced pressure bottoms liquid to produce at least partially condensed feed gas and vaporized bottoms liquid; and
a second pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.

24. Apparatus for recovering helium from a nitrogen-rich feed gas comprising helium, said apparatus comprising:

a distillation column system for operation at an elevated operating pressure to separate at least partially condensed feed gas into helium-enriched overhead vapor and nitrogen-enriched bottoms liquid(s);
an overhead condenser for partially condensing helium-enriched overhead vapor by indirect heat exchange to produce helium-enriched vapor as product and liquid for reflux in the column system;
a first pump for pumping a second nitrogen-enriched bottoms liquid to produce pumped bottoms liquid;
a first heat exchange system for cooling feed gas by indirect heat exchange with said pumped bottoms liquid to produce cooled feed gas and nitrogen-enriched vapor;
a second heat exchange system for cooling said cooled feed gas by indirect heat exchange against a first nitrogen-enriched bottoms liquid to produce at least partially condensed feed gas and vapor for the column system; and
a pressure reduction device for reducing the pressure of said at least partially condensed feed gas to produce at least partially condensed feed gas at reduced pressure for use as said feed to the distillation column system.



Prior Art

FIG. 1

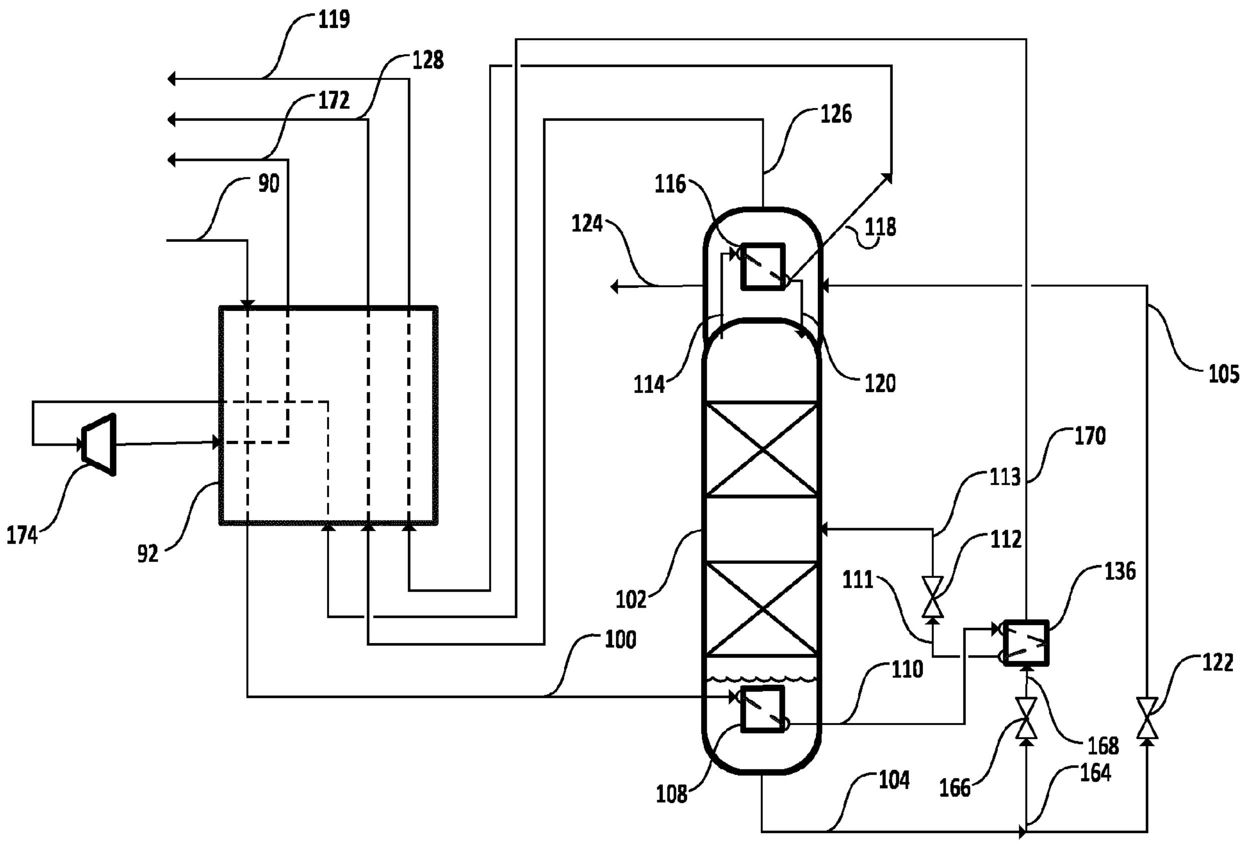

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FIG. 2

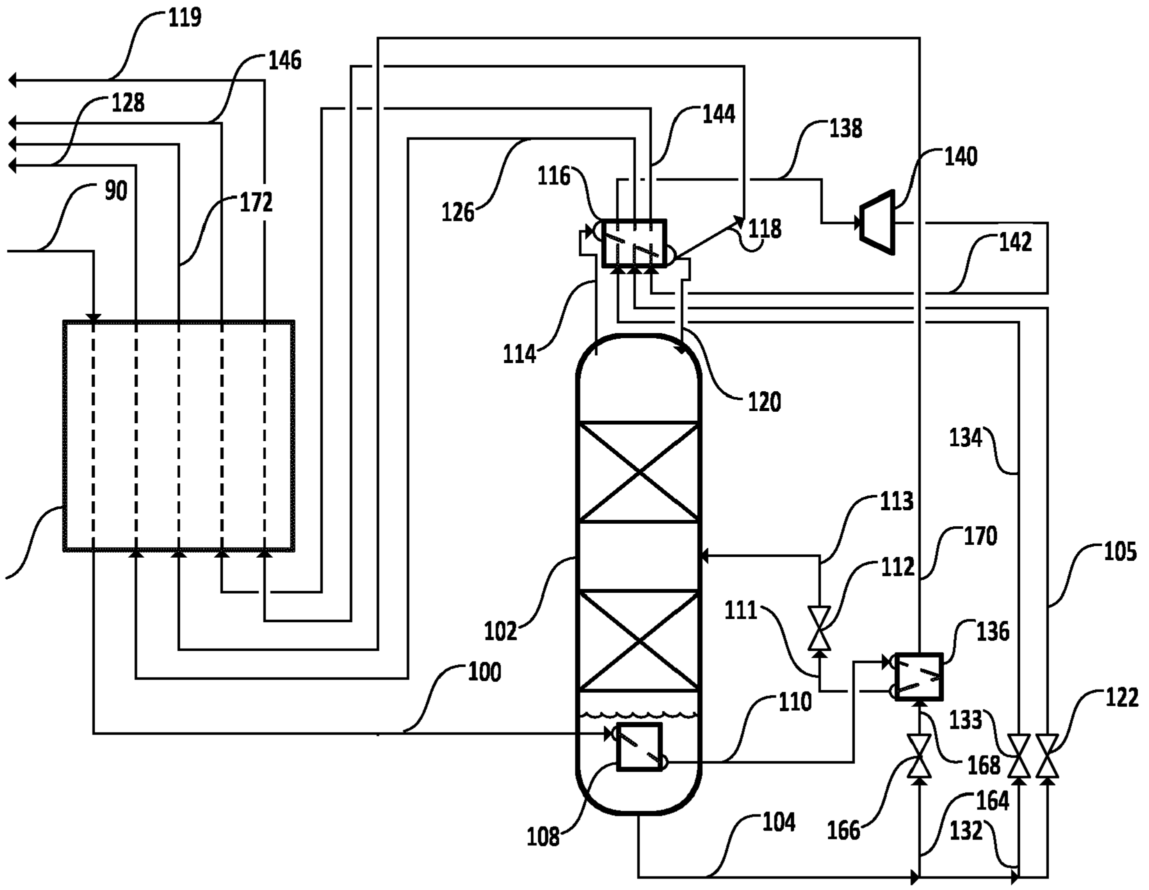
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FIG. 3

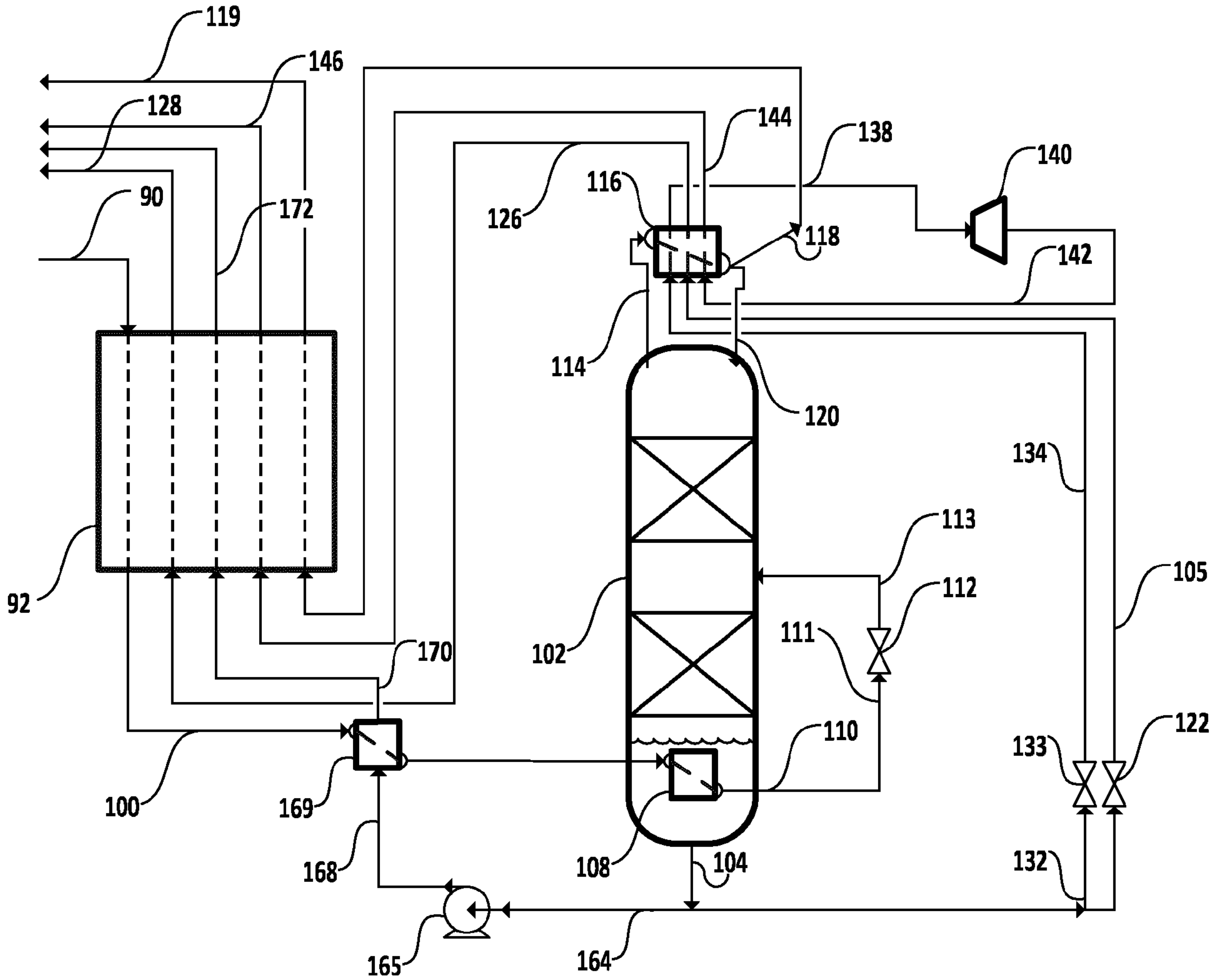

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FIG. 4

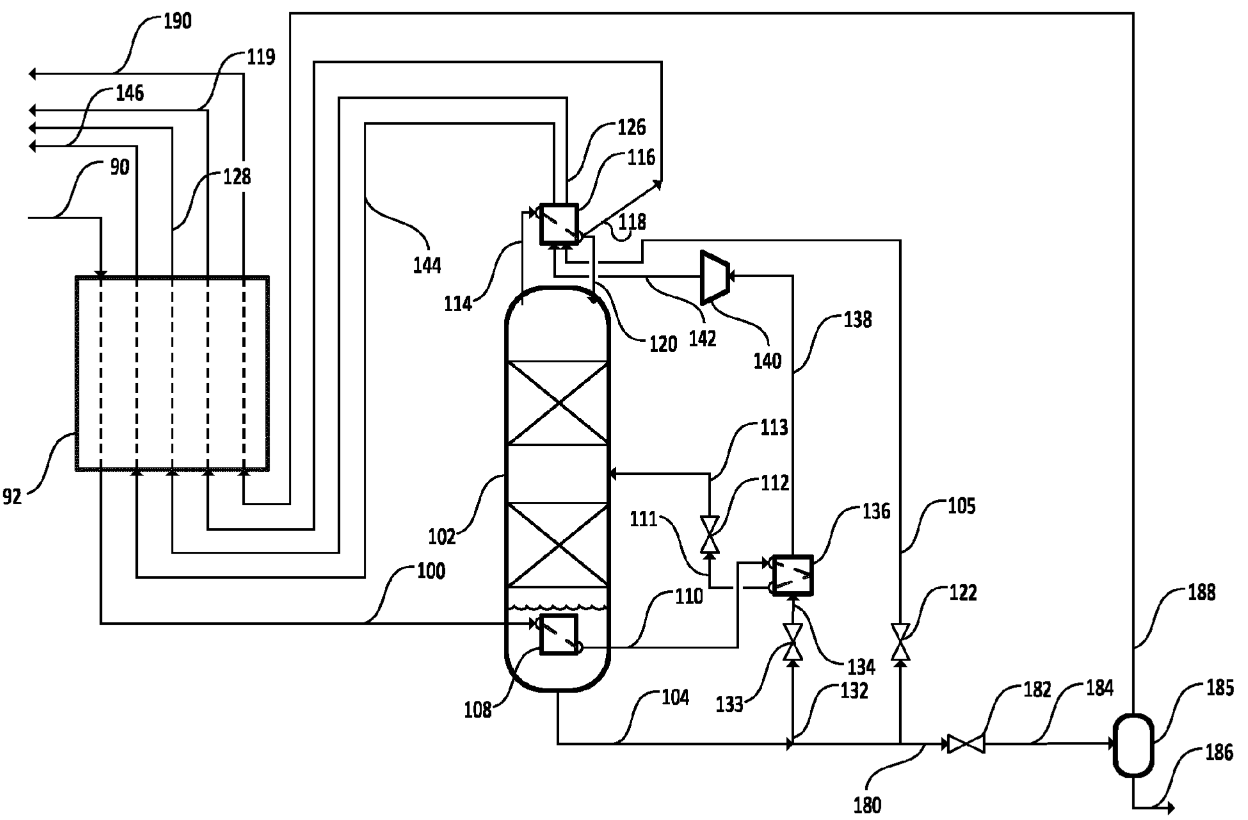
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FIG. 5

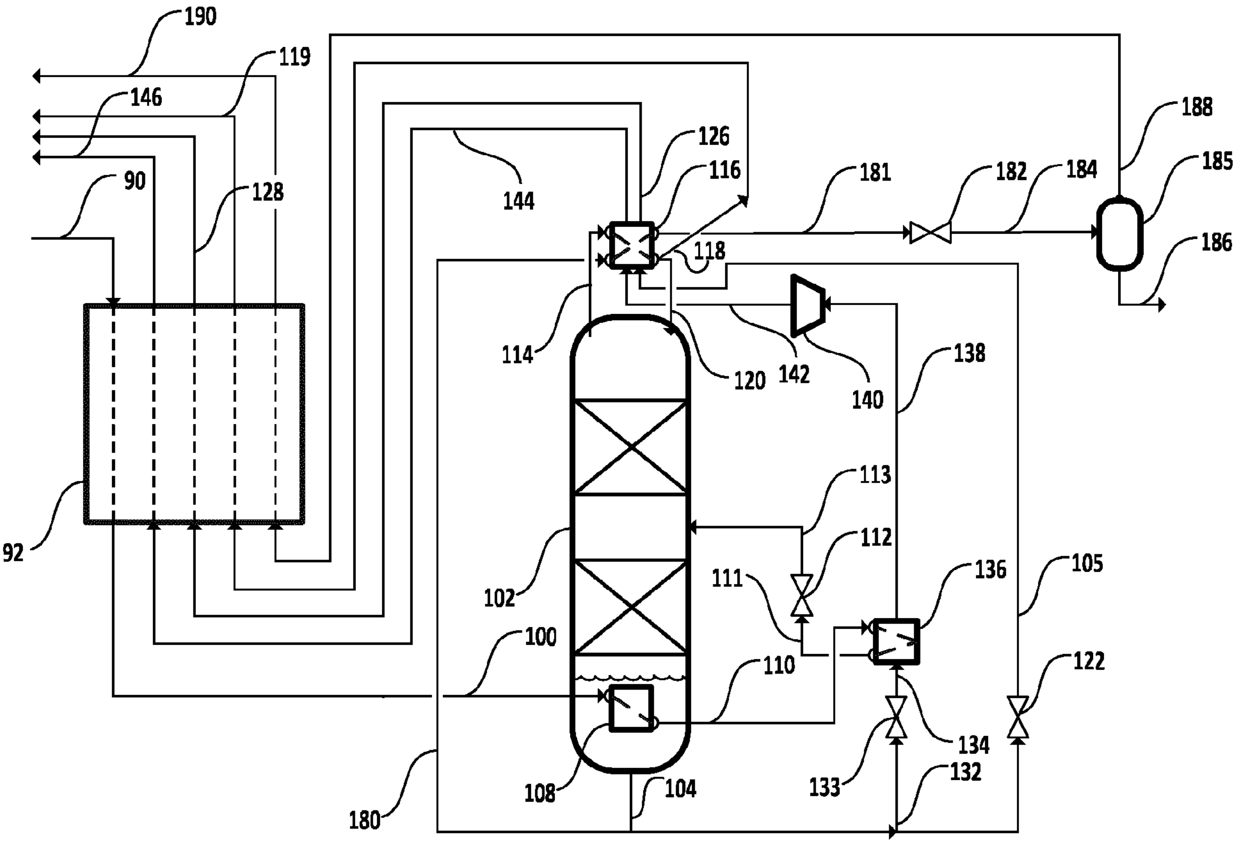
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FIG. 6

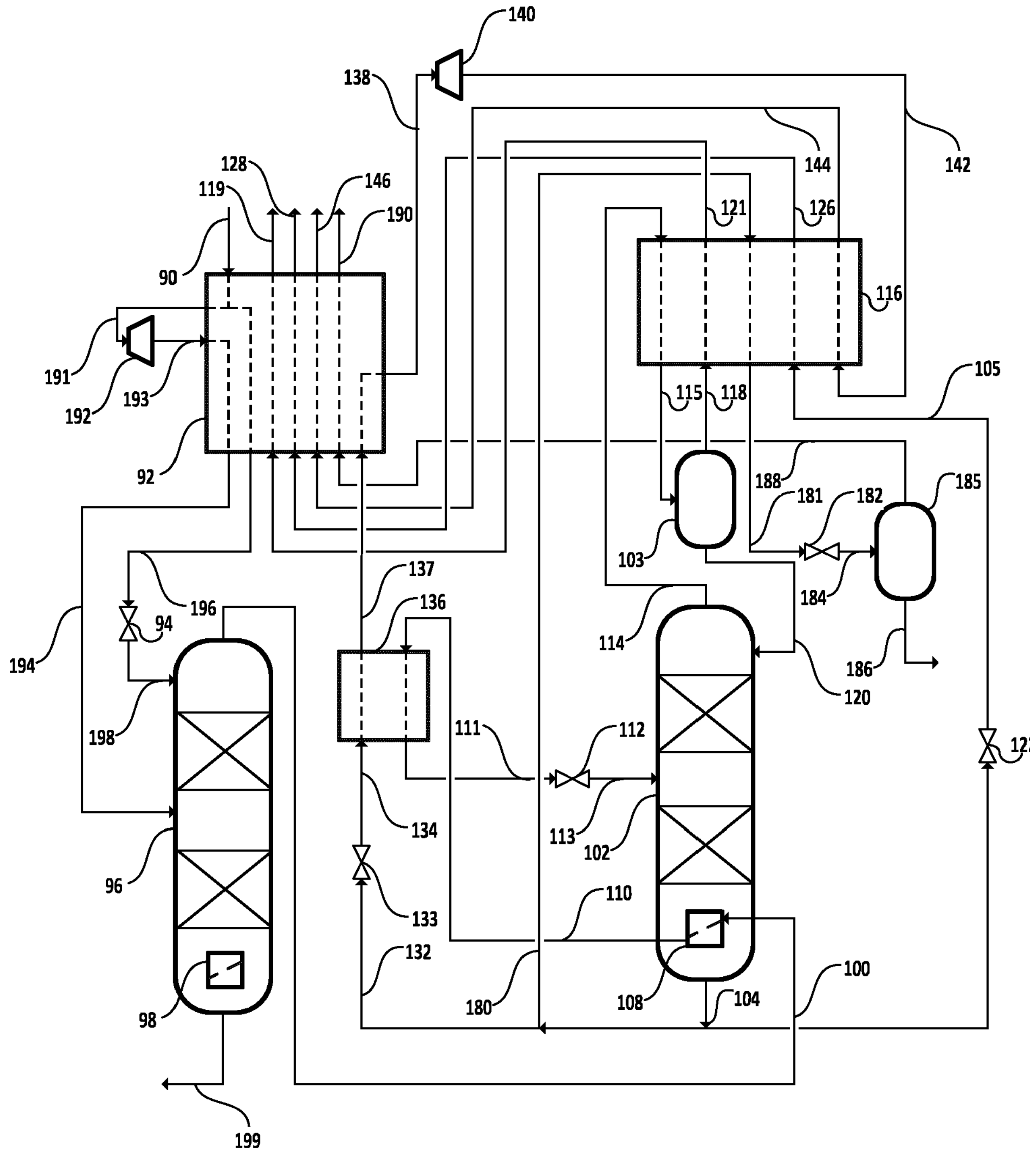
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FIG. 7

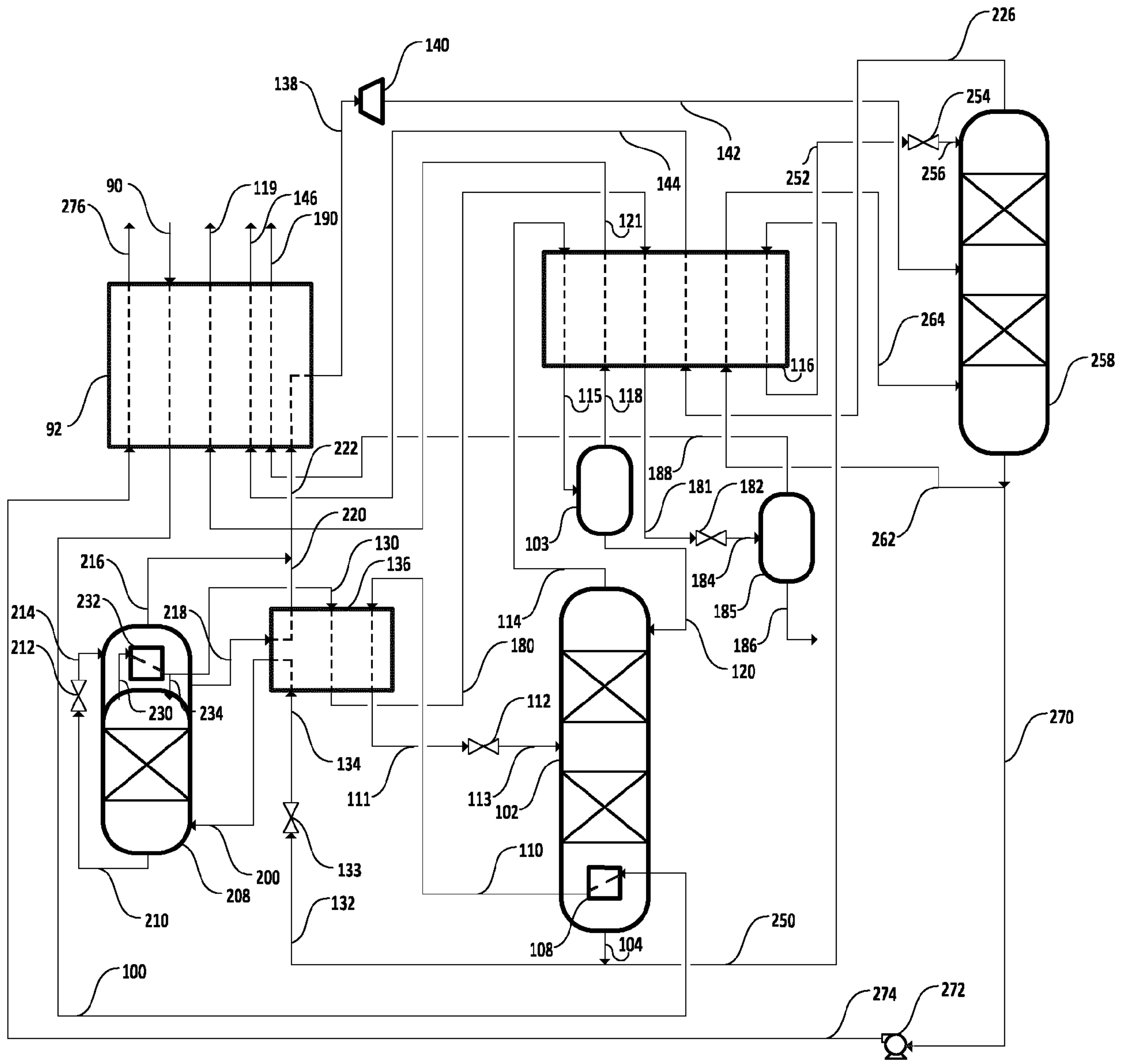
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FIG. 8

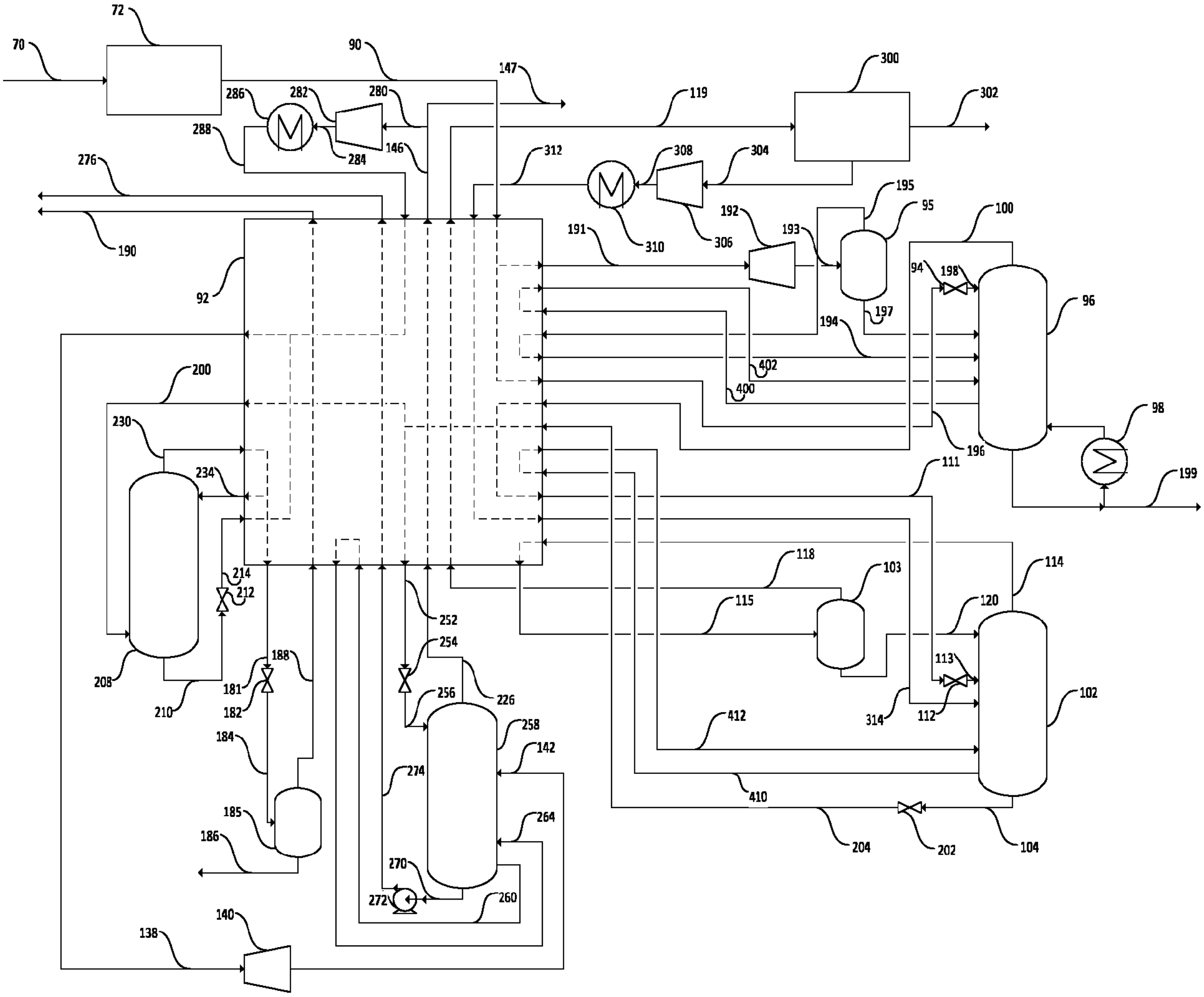
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FIG. 9

REFERENCES CITED IN THE DESCRIPTION

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