

(12) UK Patent Application (19) GB (11) 2 297 825 (13) A

(43) Date of A Publication 14.08.1996

(21) Application No 9502126.7

(22) Date of Filing 03.02.1995

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(51) INT CL⁶

F25J 3/02

(52) UK CL (Edition O)

F4P PFB P931 P941 P960

(56) Documents Cited

EP 0233609 A2 EP 0132984 A1 EP 0131128 A1

(58) Field of Search

**UK CL (Edition N) B1B BE , F4P PFB PFC PFE
INT CL⁶ F25J 3/02**

(54) Process to remove nitrogen from natural gas

(57) Nitrogen is removed from a natural gas feed stream by a cryogenic distillation process in which the feed stream (101) is separated in a distillation column (11); a methane-rich bottoms liquid (103) is recovered as a methane-rich product (104); a nitrogen-rich overhead vapor (105) is warmed in heat exchange (4) with intermediate vapor stream(s) (112, 114) to at least partially condense said stream(s) for return (113, 115) to the distillation column (11) to provide reflux; and a portion (108) of the warmed nitrogen-rich overhead vapor (105) is utilized as a recycle nitrogen-rich heat pump stream (109, 110, 111) above the critical pressure of nitrogen to provide at least part of the reboil (2) to the distillation column and to produce a mixed vapor-liquid stream, which is returned to the distillation column (11) to provide reflux. Preferably the methane rich product (104) is pumped (21) to increase its pressure.

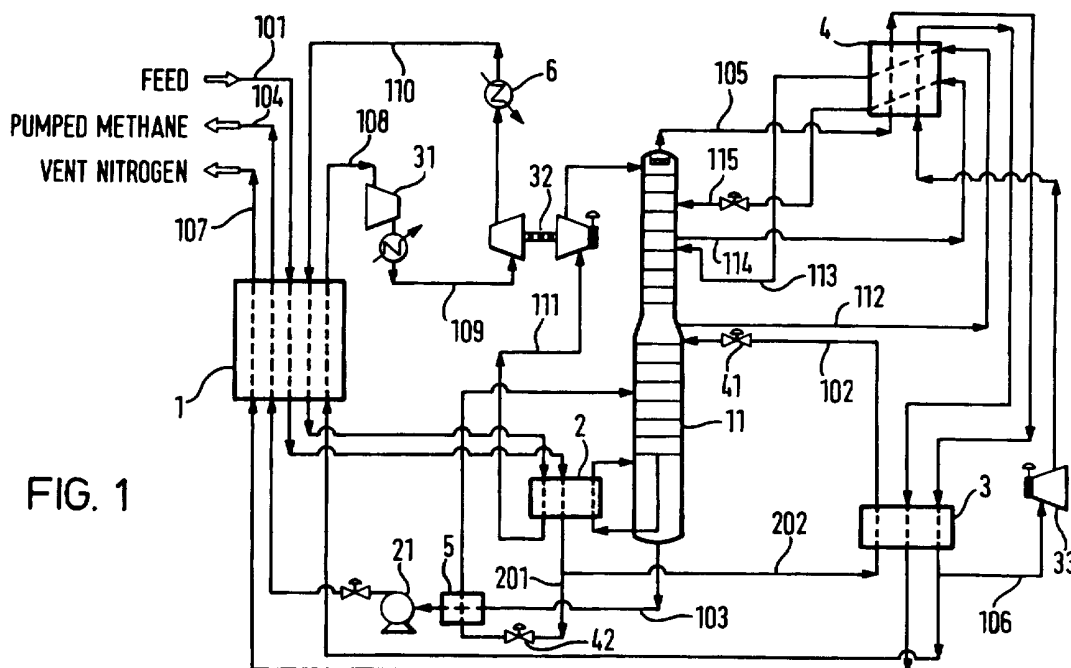


FIG. 1

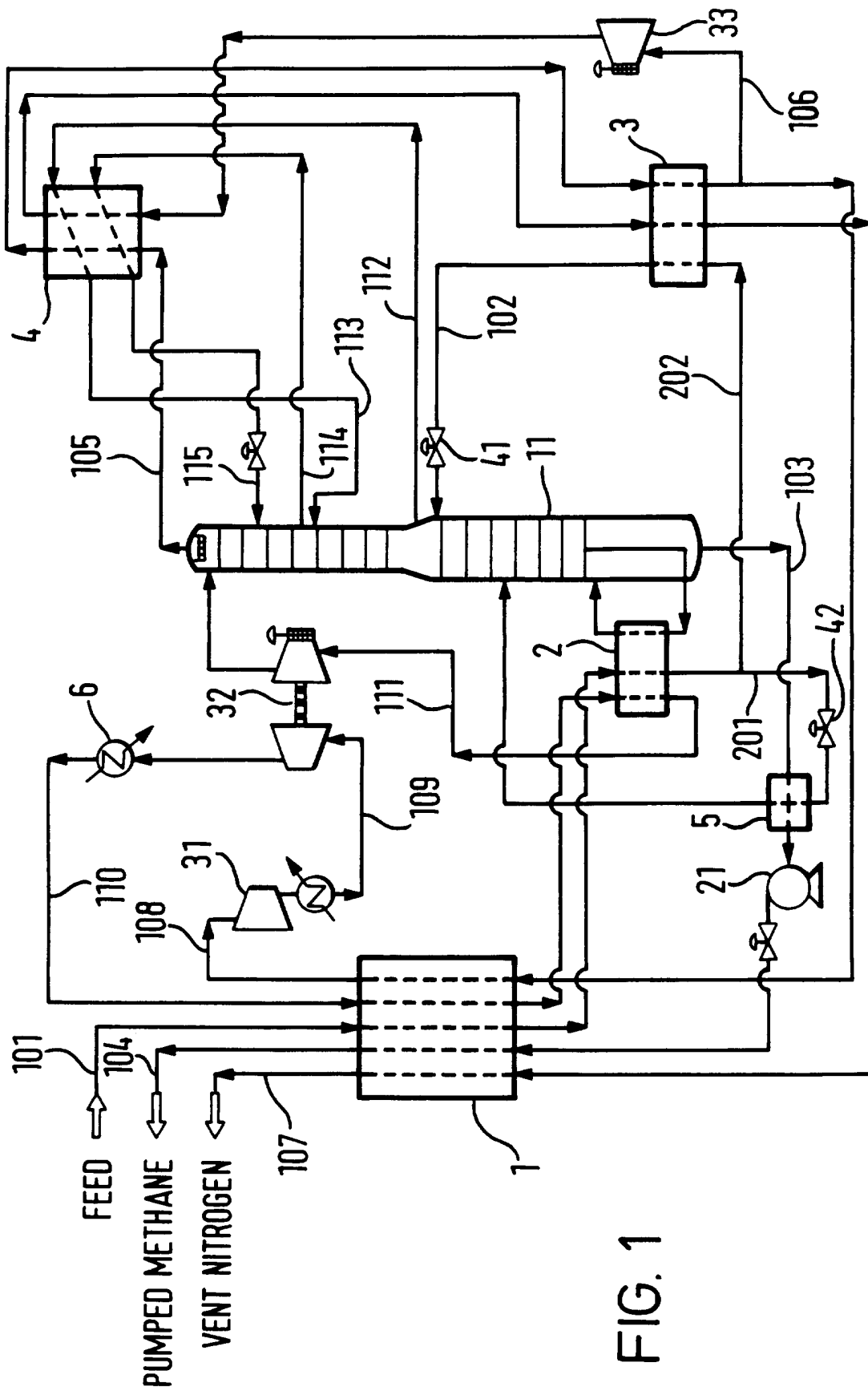


FIG. 1

PROCESS TO REMOVE NITROGEN FROM NATURAL GAS

5 The present invention relates to a cryogenic process
for the removal of nitrogen from feed gas comprising
nitrogen and hydrocarbons.

10 The increasing use of natural gas as a fuel has
resulted in a need to remove nitrogen from some natural gas
sources, in order to meet Wobbe Index and calorific value
specifications, particularly where the gas is delivered
into a country's gas transmission system. The nitrogen may
either be naturally occurring or resulting from nitrogen
injection into oil fields for enhanced recovery.

15 A particular problem is to design a process for
efficient removal of nitrogen from natural gas feed at high
pressure (75 to 130 bar absolute; 7.5 to 13 MPa), with
relatively small concentrations of nitrogen (5 to 15 mol%),
20 and to produce sales gas at a pressure similar to the feed
gas pressure.

A further problem is that, as gas reservoir pressure
decays to below the required sales gas pressure (e.g.,
25 about 75 bar absolute; 7.5 MPa in the case of the United
Kingdom's National Transmission System), feed gas
compression needs to be added. This is a relatively
expensive investment because it is not utilized fully
throughout the life of the nitrogen removal unit (NRU).

30 Therefore, an object of the present invention is to
provide an improved process to remove nitrogen from natural
gas feed with low nitrogen content (about 5 to 15 mol%) and
at high pressure (75 to 130 bar absolute; 7.5 to 13 MPa).
35 It is a further object of this invention to provide a
process for removal of nitrogen from natural gas feed,

which is sufficiently flexible to operate at lower feed pressure (25 to 75 bar absolute; 2.5 to 7.5 MPa) while still producing sales gas at higher pressure (about 75 bar absolute; 7.5 MPa), without the need for feed gas
5 compression.

Nitrogen removal from natural gas is usually most economically effected by cryogenic distillation. Numerous cycles have been developed, many based on the concept of
10 double distillation columns as used in air separation. One problem associated with double column cycles is that, at feed nitrogen concentrations less than 25 mol%, the quantity of reflux liquid that can be generated is insufficient to achieve an economic recovery of methane.
15 Another problem is that relatively low concentrations of carbon dioxide and hydrocarbons, such as benzene, hexane and heavier components, would freeze at the cryogenic temperatures associated with the lower pressure column.

20 GB-B-2,208,699 describes an improved process that is less energy intensive at low levels of feed nitrogen concentration, in which the separation is effected in two columns with integrated condensation of overhead first column vapor and second column reboil. While this process
25 overcomes the problems mentioned above, it is relatively complicated and expensive.

US-A-4,415,345 describes a process employing a nitrogen heat pump cycle to generate liquid reflux, which
30 is compatible with both single column and double column arrangements. However, this cycle produces methane product at a lower pressure than the natural gas feed and will generally require product gas compression in addition to the nitrogen heat pump compressor. It also limits the
35 column to a low operating pressure of from 15 to 125 psia; (100 to 865 kPa), with heat pump nitrogen at a pressure of

50 to 470 psia (345 to 3,250 kPa), i.e. below the critical pressure. The column pressure is, therefore, dictated by the nitrogen pressure to allow sufficient temperature difference to condense the nitrogen against boiling methane in the column reboiler. This low column operating pressure increases power consumption for product compression.

The present invention provides a cryogenic process for the removal of nitrogen from a natural gas feed stream comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:

(i) feeding the feed stream to a distillation column providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapor, and at least one intermediate vapor stream;

(ii) recovering the methane-rich bottoms as a methane-rich product, preferably after pumping to increase its pressure;

(iii) warming the nitrogen-rich overhead vapor in heat exchange with the at least one intermediate vapor stream to at least partially condense said at least one intermediate vapor stream;

(iv) returning the at least partially condensed intermediate vapor stream to the distillation column, preferably at an intermediate location, to provide reflux;

(v) utilizing a portion of the warmed nitrogen-rich overhead vapor as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapor-liquid stream; and

(vi) returning the mixed vapor-liquid stream to the distillation column, preferably at the top thereof, to provide reflux.

The invention also provides an apparatus for the cryogenic removal of nitrogen from a natural gas feed stream by the process of the invention, the apparatus

comprising:

a distillation column for providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapor, and at least one intermediate vapor stream;

5 means for feeding the feed stream to the distillation column;

means for recovering the methane-rich bottoms as a methane-rich product;

10 heat exchange means for warming the nitrogen-rich overhead vapor in heat exchange with the at least one intermediate vapor stream to at least partially condense said at least one intermediate vapor stream;

means for returning the at least partially condensed intermediate vapor stream to the distillation column to provide reflux;

15 means for utilizing a portion of the warmed nitrogen-rich overhead vapor as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapor-liquid stream; and

20 means for returning the mixed vapor-liquid stream to the distillation column to provide reflux.

One or more intermediate vapor streams can be partially condensed in step (iii) of the process of the invention but it is preferred that a lower intermediate vapor stream is withdrawn at or above the location of the feed point of the natural gas feed stream and returned as reflux above said withdrawal point, and an upper intermediate vapor stream is withdrawn at or above said lower reflux feed point and returned as reflux above the withdrawal point of the upper intermediate vapor stream.

Suitably, the methane-rich bottoms liquid is pumped and then vaporized to increase its pressure before recovery as the methane-rich product.

Usually, the distillation column is reboiled by heat exchange with both the natural gas feed stream and the recycle nitrogen-rich heat pump stream.

5 In a presently preferred embodiment, the present invention relates to a cryogenic process for the removal of nitrogen from a natural gas feed stream comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:

10 (a) cooling and at least partially condensing the natural gas feed stream;

(b) further cooling and reducing the pressure of at least part of the natural gas feed stream and feeding it to an intermediate location of a single distillation column;

15 (c) removing a methane-rich bottoms liquid from the distillation column, pumping the removed, methane-rich bottoms liquid to increase its pressure, vaporizing the pumped, removed, methane-rich bottoms liquid, and recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product;

20 (d) removing a nitrogen-rich overhead stream from the distillation column, warming the removed, nitrogen-rich overhead stream to recover refrigeration, and dividing the warmed, removed, nitrogen-rich overhead stream into first and second substreams;

25 (e) expanding and warming said first substream to recover refrigeration;

30 (f) warming said second substream, compressing the warmed, second substream, cooling the compressed, second substream, and expanding the cooled, compressed, second substream thereby producing a mixed vapor-liquid stream;

(g) feeding the mixed vapor-liquid stream to the top of the distillation column; and

35 (h) using at least a part of the refrigeration recovered from warming the nitrogen-rich overhead stream of step (d) to condense at least one intermediate vapor stream

from the distillation column to provide intermediate reflux to the distillation column.

5 Preferably, the natural gas feed stream is divided into first and second portions; said first portion is reduced in pressure and fed to an intermediate location of the distillation column; and said second portion is reduced in pressure, partially vaporized and then fed to the distillation column at a location below the feed for said
10 first portion.

15 A portion of the warmed nitrogen-rich overhead vapor suitably is expanded and then further warmed to recover refrigeration.

20 The methane-rich bottoms liquid usually will be subcooled prior to pumping and suitably this subcooling is by heat exchange with a reduced pressure portion of the feed stream.

25 The recycle nitrogen-rich heat pump stream conveniently is both compressed and, after cooling, subsequently expanded in a compander.

30 Preferably, the pressure of at least part of the natural gas feed stream is reduced, prior to feeding to the distillation column, with a dense fluid expander.

35 The distillation column can have an intermediate reboiler/condenser located below the feed point of the natural gas feed stream.

 The following is a description by way of example only and with reference to the accompanying drawing of a
35 presently preferred embodiment of the invention.

Figure 1 is a schematic diagram of the process of the present invention.

Referring to the process flow diagram shown in Figure 1, a natural gas feed in line 101, which has been treated to reduce to acceptable concentrations freezing components, such as water and carbon dioxide, is cooled and at least partially condensed in main heat exchanger 1. The natural gas feed will generally contain 5 to 15 mol% nitrogen with the balance being natural gas and impurities and will be at a pressure of 25 to 130 bar absolute (2.5 to 13 MPa), preferably 60 to 80 bar absolute (6 to 8 MPa). The cooled and at least partially condensed natural gas feed is then further cooled and condensed (if not totally condensed in main heat exchanger 1) in reboiler 2, and split into two portions in lines 201 and 202. The major portion in line 202 is subcooled in subcooler 3 before being introduced via line 102 to distillation column 11 through valve 41. The smaller portion in line 201 is reduced in pressure across valve 42 and partially vaporized in subcooler 5 before also being introduced to distillation column 11.

Distillation column 11 operates at a pressure from 10 to 30 bar absolute (1,000 to 3,000 kPa), preferably between 15 and 22 bar absolute (1 and 2.2 MPa), and separates the natural gas feeds into a methane-rich bottoms liquid stream 103 and a nitrogen-rich overhead vapor stream 105. The nitrogen-rich overhead stream 105 typically contains 2 mol% methane, and the methane-rich bottoms stream 103 has a typical nitrogen concentration of 0.5 mol%, which is generally lower than the required nitrogen content of natural gas that is delivered, for example, to the United Kingdom's National Transmission System (NTS), where concentrations of 4 to 5 mol% are acceptable in gas with parts per million concentrations of carbon dioxide. By reducing the nitrogen content to this low level, which is

perfectly feasible in a cryogenic NRU, the quantity of natural gas feed that must be processed is reduced, the final sales gas product being blended from feed gas bypass and NRU product. The UK's NTS specification allows up to 2
5 mol% CO₂, and with increasing CO₂ content, nitrogen would need to be removed to a lower concentration in the sales gas by processing more gas in the NRU.

Part of the reboil duty for column 11 is provided by
10 heat exchange with the natural gas feed cooling in reboiler 2. The remainder is provided by heat exchange with a recycle, nitrogen-rich heat pump stream also cooling in reboiler 2. The recycle, nitrogen-rich heat pump stream in line 110 is cooled in main heat exchanger 1 and reboiler 2
15 at a pressure of 35 to 130 bar absolute (3.5 to 13 MPa), preferably between 60 and 80 bar absolute (6 and 8 MPa). The cooled, recycle, nitrogen-rich heat pump stream in line 111 is then expanded in the expansion wheel of compander 32 and introduced into the top of distillation column 11. The
20 expander outlet stream contains 4 to 5% by mass of liquid, and this is used to provide reflux for the top section of distillation column 11.

The nitrogen-rich overhead vapor in line 105 from the
25 top of column 11, containing about 2 mol% methane, is warmed in condenser 4 and subcooler 3. Condenser 4 provides the bulk of the reflux liquid for distillation column 11 by condensing vapor side streams withdrawn from the column. The lower side stream in line 112 is withdrawn
30 at or above the feed entry location (line 102) and returned as reflux liquid in line 113 which is several equilibrium stages above the withdrawal point (line 112). The upper side stream in line 114 is withdrawn at or above the lower reflux feed point (line 113) and returned in line 115
35 several equilibrium stages above this withdrawal point. This reflux philosophy is more efficient than a process

that provides all of the column reflux liquid at the top of the column, because the majority of the refrigeration required to condense the reflux is provided at the warmer condensing temperatures of the side streams.

5

After warming in subcooler 3, a portion of the nitrogen-rich overhead vapor in line 106 is expanded in expander 33, providing additional refrigeration to condenser 4 and subcooler 3. This expanded portion is then warmed in main heat exchanger 1 and vented via line 107 to atmosphere. Environmental constraints will generally limit the methane content in vented nitrogen to 2 mol% maximum. The process is capable of achieving much lower methane content, if required, by increasing the recycle nitrogen flow in line 110. Some of the nitrogen-rich vent stream may be used as utility nitrogen for purposes such as cold box purge and adsorber regeneration. The remaining nitrogen-rich overhead vapor is warmed in main heat exchanger 1, compressed in compressor 31, passed via line 109 to the compressor wheel of compander 32 and cooled in cooler 6 to form the recycle, nitrogen-rich stream to main heat exchanger 1 in line 110.

Methane-rich bottoms liquid from column 11 is subcooled in subcooler 5 before being increased in pressure by pump 21. Subcooler 5 minimizes the elevation which distillation column 11 is required to be above pump 21 in order to provide the necessary net positive suction head (NPSH) at the pump suction, particularly, if there is a large turndown requirement where heat leak into the pump suction piping could cause cavitation at turndown. The pumped liquid is then vaporized in main heat exchanger 1 to be delivered as sales gas product at a pressure of 25 to 130 bar absolute (2.5 to 13 MPa), preferably 60 to 80 bar absolute (6 to 8 MPa).

The process achieves a very high methane recovery, typically about 99.8 mol%, because the methane content in the vent nitrogen can be reduced to less than 2 mol%.

- 5 Table 1 summarizes a mass balance for a typical application of this invention.

[illegible]

Several modifications of the above-described process are possible within the scope of the invention, including:

5 The number of side streams that are condensed in condenser 4 could be increased to three or more if the resulting reduction in power consumption warranted the additional complexity. Alternatively, the system could be simplified by condensing only one side stream.

10 All or part of the vent nitrogen can be produced at a higher pressure and used as a by-product by increasing expander 33 outlet pressure or by the removal of a nitrogen containing stream prior to the inlet to compressor 31, prior to the inlet to the compressor wheel of compander 32,
15 or from downstream of cooler 6. This may result in the elimination of expander 33 from the process. It is possible to eliminate expander 33 in any event by increasing the refrigeration produced by compander 32, although this is less efficient.

20 Expander 33 could be moved to provide refrigeration at a warmer part of the process, e.g., around exchanger 1. This could be beneficial where the feed pressure was much lower than the required sales gas product pressure.

25 In addition to the vent nitrogen, all or some of the recycle heat pump nitrogen could be expanded in expander 33. This would reduce the flowrate of the recycle heat pump nitrogen, but would also reduce the inlet pressure to
30 compressor 31 and, thus, possibly require two (2) differing pressure feeds to the compressor.

35 It is possible to improve the process efficiency by expanding the feed to the column in a dense fluid expander, rather than valve 41. The expansion work could be recovered in a suitable device, such as an electricity

generator, and the refrigeration produced would reduce the refrigeration required from compander 32.

5 Subcooler 5 could be eliminated and the required pump NPSH developed by increasing the elevation difference between the column sump and the pump suction.

10 Part of the feed could be subcooled to a lower temperature in subcooler 3, rather than subcooling all of the feed, that is expanded across valve 41, to a warmer temperature. This colder feed could then be introduced to column 11 a few stages higher than the remaining feed from upstream of subcooler 3.

15 The column system could be modified to include an intermediate reboiler between the bottom of the column and the main feed stage. This may be appropriate for higher feed nitrogen concentrations.

20 The position of compander 32 could be changed so that recycle nitrogen was compressed in the compressor wheel of the compander before being compressed in compressor 31. This would be determined by the optimum machinery configuration.

25 Liquid methane from the bottom of column 11 could be further processed to recover a natural gas liquids product.

30 The process could be modified to recover a helium-rich stream from the overhead vapor from column 11, where there was sufficient helium in the natural gas feed to make this economically attractive.

35 The process could be operated with a much higher methane content in the vent nitrogen for possible use as a fuel stream with a consequent reduction in power

consumption.

5 The exemplified embodiment of the invention provides a process cycle with only slightly higher power consumption than the efficient cycle described in GB-B-2,208,699, but which is much simpler and has a significantly lower capital cost.

10 The refrigeration provided by the expansion wheel of compander 32 and expander 33 is sufficient to compensate for pump work, heat leak and temperature difference at the warm end of main heat exchanger 1 and enables the sales gas product to be delivered at a similar pressure to the feed with no need for any product compression. The expansion
15 work of the recycle nitrogen is efficiently recovered in the compressor wheel of compander 32.

20 If the feed pressure reduces over a period of time, for example, due to decay of gas reservoir pressure, the sales gas can still be produced at the required pressure simply by increasing the refrigeration provided by the expansion wheel of compander 32 beyond what is required for the column reflux liquid. This compensates for the reduced Joule-Thomson refrigeration that is available from the
25 lower pressure feed. By this method, sales gas can be produced at, for example, 79 bar absolute (7.9 MPa) with the feed gas pressure as low as 25 bar absolute (2.5 MPa). Operation of the NRU is less efficient at feed gas pressures much below the required sales gas pressure, and
30 capacity will be reduced because all of the feed gas will need to be processed in the NRU because there can be no bypass. Also, the size of compander 32 will limit production. However, this gives the plant operator the choice of whether or not to invest in feed gas compression
35 and certainly postpones the date at which it becomes economically viable to purchase or lease this compression

system.

5 The problem of freezing carbon dioxide and heavy hydrocarbons is mitigated by the single column process, operating at high pressure, because the freezing components are recovered in the bottom section of the column where the temperature is higher. The closest approach to freezing generally occurs at the feed inlet to the column, downstream of valve 41. The process is tolerant to
10 significantly higher concentrations of carbon dioxide and heavy hydrocarbons than a double column process.

The exemplified embodiment of the present invention differs from prior art by, inter alia,:

15 (i) Separating nitrogen from methane in a single column where refrigeration for column reflux is provided by a nitrogen-rich heat pump system operating above the critical pressure of nitrogen, i.e. at a pressure of 35 to 130 bar absolute (3.5 to 13 MPa) preferably between 60 and 80 bar absolute (6 and 8 MPa), thereby permitting a higher column
20 operating pressure of 10 to 30 bar absolute (1 to 3 MPa), preferably between 15 and 22 bar absolute (1 and 2.2 MPa). The column is reboiled by indirect heat exchange with both
25 the nitrogen-rich heat pump stream and the feed stream.

(ii) Expanding the nitrogen-rich heat pump stream in a compander to provide an outlet stream with up to 10% liquid, preferably 4 to 5% liquid, thereby directly
30 providing reflux for the top section of the column and efficiently recovering the expansion work.

(iii) Condensing side streams of column vapor to provide the majority of the reflux liquid at warmer temperatures by
35 indirect heat exchange with the overhead vapor from the column, which comprises the nitrogen-rich heat pump stream

and the nitrogen-rich vent stream.

(iv) Providing sufficient refrigeration with the compander 32 and, where included, the vent nitrogen expander 33 to produce, by pumping, all of the sales gas product at a pressure similar to, or higher than, the natural gas feed pressure and avoiding the need for a product compressor.

(v) Subcooling the feed against recycle heat pump nitrogen in subcooler 3 prior to introduction to the distillation column.

(vi) Subcooling the column bottoms liquid prior to pumping in order to provide sufficient NPSH without elevating the column relative to the pump suction. This is particularly beneficial where height limitations are imposed for environmental reasons.

It will be appreciated that the invention is not restricted to the specific details of the embodiment described above and that numerous modifications and variations can be made without departing from the scope of the invention as defined in the following claims.

CLAIMS:

1. A cryogenic process for the removal of nitrogen from a natural gas feed stream comprising nitrogen and hydrocarbons primarily having a carbon content between 1 and 8 carbon atoms comprising:
 - (i) feeding the feed stream to a distillation column providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapor, and at least one intermediate vapor stream;
 - (ii) recovering the methane-rich bottoms as a methane-rich product;
 - (iii) warming the nitrogen-rich overhead vapor in heat exchange with the at least one intermediate vapor stream to at least partially condense said at least one intermediate vapor stream;
 - (iv) returning the at least partially condensed intermediate vapor stream to the distillation column to provide reflux;
 - (v) utilizing a portion of the warmed nitrogen-rich overhead vapor as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapor-liquid stream; and
 - (vi) returning the mixed vapor-liquid stream to the distillation column to provide reflux.
2. A process as claimed in Claim 1, wherein the at least partially condensed intermediate vapor stream is returned at an intermediate location of the distillation column and the mixed vapor-liquid stream is returned at the top of the distillation column.
3. A process as claimed in Claim 1 or Claim 2, wherein only one intermediate vapor stream is partially condensed in step (iii).

4. A process as claimed in Claim 1 or Claim 2, wherein, in steps (iii) and (iv), a lower intermediate vapor stream is withdrawn at or above the location of the feed point of the natural gas feed stream and, after at least partial
5 condensation, returned above said withdrawal point to provide reflux, and an upper intermediate vapor stream is withdrawn at or above said lower reflux feed point and, after at least partial condensation, returned above the withdrawal point of the upper intermediate vapor stream to
10 provide reflux.

5. A process as claimed in any one of the preceding claims, wherein the methane-rich bottoms liquid is pumped to increase its pressure prior to recovery as methane-rich
15 product.

6. A process as claimed in Claim 5, wherein the increased pressure, methane-rich bottoms liquid is vaporized before recovery as the methane-rich product.

20

7. A process as claimed in any one of the preceding claims, wherein the distillation column is reboiled by heat exchange with both the natural gas feed stream and the recycle nitrogen-rich heat pump stream.

25

8. A cryogenic process for the removal of nitrogen from a natural gas feed stream as claimed in Claim 1 comprising:

- (a) cooling and at least partially condensing the natural gas feed stream;
- 30 (b) further cooling and reducing the pressure of at least part of the natural gas feed stream and feeding it to an intermediate location of a single distillation column;
- (c) removing a methane-rich bottoms liquid from the distillation column, pumping the removed, methane-rich
35 bottoms liquid to increase its pressure, vaporizing the pumped, removed, methane-rich bottoms liquid, and

recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product;

(d) removing a nitrogen-rich overhead stream from the distillation column, warming the removed, nitrogen-rich overhead stream to recover refrigeration, and dividing the warmed, removed, nitrogen-rich overhead stream into first and second substreams;

(e) expanding and warming said first substream to recover refrigeration;

(f) warming said second substream, compressing the warmed, second substream, cooling the compressed, second substream, and expanding the cooled, compressed, second substream thereby producing a mixed vapor-liquid stream;

(g) feeding the mixed vapor-liquid stream to the top of the distillation column; and

(h) using at least a part of the refrigeration recovered from warming the nitrogen-rich overhead stream of step (d) to condense at least one intermediate vapor stream from the distillation column to provide intermediate reflux to the distillation column.

9. A process as claimed in any one of the preceding claims, wherein the natural gas feed stream is divided into first and second portions; said first portion is reduced in pressure and fed to an intermediate location of the distillation column; and said second portion is reduced in pressure, partially vaporized and then fed to the distillation column at a location below the feed for the said first portion.

10. A process as claimed in any one of the preceding claims, wherein a portion of the warmed nitrogen-rich overhead vapor is expanded and then further warmed.

11. A process as claimed in any one of the preceding claims, wherein the recycle nitrogen-rich heat pump stream

is both compressed and, after cooling, subsequently expanded in a compander.

5 12. A process as claimed in any one of the preceding claims, wherein at least part of the natural gas feed stream is expanded in a dense fluid expander prior to feeding to the distillation column.

10 13. A process as claimed in any one of the preceding claims, wherein the distillation column has an intermediate reboiler/condenser located below the feed point of the natural gas feed stream.

15 14. A process as claimed in any one of the preceding claims, wherein the methane-rich bottoms liquid is subcooled and then pumped to increase its pressure prior to recovery as methane-rich product.

20 15. A process as claimed in Claim 14, wherein the subcooling of the methane-rich bottoms liquid is by heat exchange with a reduced pressure portion of the feed stream.

25 16. A process as claimed in any one of the preceding claims, wherein the distillation column operates at 1 to 3 MPa (10 to 30 bar absolute).

30 17. A process as claimed in Claim 16, wherein the distillation column operates at 1.5 to 2.2 MPa (15 to 22 bar absolute).

35 18. A process as claimed in any one of the preceding claims, wherein the nitrogen-rich overhead vapor contains about 2 mol% methane and the methane-rich bottoms liquid contains about 0.5 mol% nitrogen.

19. A process as claimed in Claim 1 and substantially as hereinbefore described with reference to the accompanying drawing.

5 20. An apparatus for the cryogenic removal of nitrogen from a natural gas feed stream by a process as claimed in Claim 1, the apparatus comprising:

a distillation column for providing a methane-rich bottoms liquid, a nitrogen-rich overhead vapor, and at least one intermediate vapor stream;

10 means for feeding the feed stream to the distillation column;

means for recovering the methane-rich bottoms as a methane-rich product;

15 heat exchange means for warming the nitrogen-rich overhead vapor in heat exchange with the at least one intermediate vapor stream to at least partially condense said at least one intermediate vapor stream;

20 means for returning the at least partially condensed intermediate vapor stream to the distillation column to provide reflux;

means for utilizing a portion of the warmed nitrogen-rich overhead vapor as a recycle nitrogen-rich heat pump stream above the critical pressure of nitrogen to provide at least part of the reboil to the distillation column and to produce a mixed vapor-liquid stream; and

25 means for returning the mixed vapor-liquid stream to the distillation column to provide reflux.

30 21. A apparatus as claimed in Claim 20, wherein the at least partially condensed intermediate vapor stream is returned at an intermediate location of the distillation column and the mixed vapor-liquid stream is returned at the top of the distillation column.

22. An apparatus as claimed in Claim 20 or Claim 21, wherein the heat exchange means at least partially condenses only one intermediate vapor stream for return to the distillation column to provide reflux.

5

23. An apparatus as claimed in Claim 20 or Claim 21, wherein the heat exchange means at least partially condenses a lower intermediate vapor stream withdrawn from the distillation column at or above the location of the feed point of the natural gas feed stream and an upper intermediate vapor stream withdrawn at or above the feed point at which at least partially condensed lower intermediate vapor stream is returned to the distillation column and the means for returning at least partially condensed intermediate vapor to the distillation column to provide reflux returns both said at least partially condensed intermediate streams to the distillation column at locations above their respective withdrawal points.

20 24. An apparatus as claimed in any one of Claims 20 to 23, comprising means for increasing the pressure of the methane-rich bottoms liquid prior to recovery as methane-rich product.

25 25. An apparatus as claimed in Claim 24, comprising means for vaporizing the increased pressure, methane-rich bottoms liquid before recovery as the methane-rich product.

30 26. An apparatus as claimed in any one of Claims 20 to 25, comprising means for reboiling the distillation column by heat exchange with both the natural gas feed stream and the recycle nitrogen-rich heat pump stream.

35 27. An apparatus as claimed in Claim 20 comprising:
(a) means for cooling and at least partially condensing the natural gas feed stream;

(b) means for further cooling and reducing the pressure of at least part of the natural gas feed stream and feeding it to an intermediate location of a single distillation column;

5 (c) means for removing a methane-rich bottoms liquid from the distillation column,

(d) means for pumping the removed, methane-rich bottoms liquid to increase its pressure, vaporizing the pumped, removed, methane-rich bottoms liquid, and
10 recovering the vaporized, increased pressure, methane-rich liquid as a sales gas product;

(e) means for removing a nitrogen-rich overhead stream from the distillation column, warming the removed, nitrogen-rich overhead stream to recover refrigeration, and
15 dividing the warmed, removed, nitrogen-rich overhead stream into first and second substreams;

(f) means for expanding and warming said first substream to recover refrigeration;

(g) means for warming said second substream,
20 compressing the warmed, second substream, cooling the compressed, second substream, and expanding the cooled, compressed, second substream thereby producing a mixed vapor-liquid stream;

(h) means for feeding the mixed vapor-liquid stream
25 to the top of the distillation column; and

(j) means for using at least a part of the refrigeration recovered from warming the nitrogen-rich overhead stream in means (e) to condense at least one intermediate vapor stream to provide intermediate reflux to
30 the distillation column.

28. An apparatus as claimed in any one of Claims 20 to 27, comprising means for dividing the natural gas feed stream into first and second portions; means for reducing the
35 pressure of said first portion; means for feeding said reduced pressure first portion to an intermediate location

of the distillation column; means for reducing the pressure of said second portion; means for partially vaporizing said reduced pressure second portion; and means for feeding said partially vaporized second portion to the distillation
5 column at a location below the location of the feed for the said first portion.

29. An apparatus as claimed in any one of Claims 20 to 28, comprising means for expanding a portion of the warmed
10 nitrogen-rich overhead vapor and means for further warming said expanded portion.

30. An apparatus as claimed in any one of Claims 20 to 29, comprising a compander for both compressing the recycle
15 nitrogen-rich heat pump stream and, after cooling, subsequently expanding said compressed portion.

31. An apparatus as claimed in any one of Claims 20 to 30, comprising a dense fluid expander for expanding at least a
20 portion of the natural gas feed stream prior to feeding to the distillation column.

32. An apparatus as claimed in any one of Claims 20 to 31, wherein an intermediate reboiler/condenser is located in
25 the distillation column below the feed point of the reduced pressure, natural gas feed stream.

33. An apparatus as claimed in any one of Claims 20 to 32, comprising means for subcooling the methane-rich bottoms
30 liquid and means for increasing the pressure of the subcooled methane-rich bottoms liquid prior to recovery as methane-rich product.

34. An apparatus as claimed in Claim 33, wherein the means
35 for subcooling of the methane-rich bottoms liquid heat exchanges said bottoms liquid with a reduced pressure

portion of the feed stream.

35. An apparatus as claimed in Claim 20 and substantially
as hereinbefore described with reference to the

5 accompanying drawing.



Application No: GB 9502126.7
Claims searched: 1-35

Examiner: Michael Richardson
Date of search: 4 July 1995

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.N): F4P (PFB, PFC, PFE); B1B (BE)

Int Cl (Ed.6): F25J 3/02

Other:

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	EP 0 233 609 A2 (AIR PRODUCTS AND CHEMICALS) See Figure 1	1, 20
A	EP 0132984 A2 (UNION CARBIDE) See Figure 1	1,20
A	EP 0131128 A2 (AIR PRODUCTS AND CHEMICALS) See Figure 1	1,20

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.