LOW TEMPERATURE FRACTIONATION OF NATURAL GAS COMPONENTS

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Fig. 1

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This invention relates generally to low temperature fractionation or separation of natural gas into at least some of its components, and it is directed particularly to an improved process for the separation of nitrogen, or both nitrogen and helium, by methods characterized by their greater practicability and economy than those customarily employed or previously proposed.

Generally contemplated for fractionation in accordance with the invention, are natural gases containing C₃ (ethane) and heavier fractions recoverable as L.P.G., as well as natural gasoline components, together with methane, nitrogen and helium. One overall primary object of the invention is to provide for the efficient removal of nitrogen from the other components, regardless of for what purpose they may be used, as where the major consideration may be the separation from fuel or synthesis gas components of inert and useless nitrogen; or where helium recovery is desired and is justified by the helium content of the natural gas, the major object may be helium recovery together with nitrogen separation.

The methane content of the gas is employed in novel and significant relation to accomplishment of the invention, not merely because of the contemplation of separation and use of methane as a product component, but also by toleration and utilization of methane in the fractionation and final separation stages in a manner contributing both uniqueness and practical economy in the nitrogen separation. In this respect and as will later appear, the invention achieves the end result of nitrogen separation to the desired completeness, but without the expensive undertaking of precise or complete single stage methane and nitrogen separation, by so conducting and controlling the fractionation process as to recover methane usable for the plant fuel requirements from a deliberately incomplete separation of methane and nitrogen, while employing separated methane as a refrigerant in the course of achieving the low temperature nitrogen fractionation.

In terms of the general process contemplated, the feed gas passes successively through a series of refrigeration zones which may comprise heat exchangers of appropriate construction through which components or fractions being separated at successive stages in the process, are passed in cumulative and generally countercurrent relation to the feed gas as it becomes progressively depleted of separated components. Thus an initial separation may be the removal of L.P.G. components to deplete the gas of C₄ and heavier hydrocarbons, and thus render the gas properly composed for a second stage separation of nitrogen from methane and C₂ hydrocarbons present. The invention is concerned primarily with this second stage nitrogen separation and related influences, and is characterized by so controlling the fractionation as to provide in the separated nitrogen stream a substantial amount of methane. The remaining methane condensate is employed in the manner later described as refrigerant in the nitrogen fractionation stage, and is recoverable as a separated methane plus C₃ fraction. Thus the nitrogen separation is permitted to occur without excessive refrigeration or expensive physical equipment otherwise, which would be required for more precise methane-nitrogen separation, all to the advantages of lower initial and operating costs of the process.

The methane-containing nitrogen mixture then is subject to further cooling and straight forward separation of all the methane, by simple equipment and procedures consistent with the overall economy of the process. The condensate derived from the final nitrogen-hydrocarbon separation has sufficient methane content as fuel in the plant boilers, fired heaters, gas turbines and the like.

The invention is particularly concerned with the fractionation of helium-containing natural gas, with the end objective of separating out the bulk of the nitrogen as waste in the fuel gas and as waste, and recovering a helium rich product which may be predominately helium and capable of separation as by known low temperature methods, into substantially pure helium and waste residual nitrogen. As employed primarily for helium recovery, the invention possesses all the operational and low-cost advantages mentioned in the foregoing, since throughout the fractionation and separation stages, up to the final nitrogen and helium-rich residue separation, the helium remains uncondensed along with the nitrogen content of the gas.

All the features and objects of the invention, as well as the details of a typical and preferred embodiment, will be understood from the following detailed description of the accompanying drawing which illustrates the process in flow sheet form.

The system will be described with reference to the fractionation of natural gas of the general type and composition produced in regions of Southwestern United States, typically the Texas Panhandle area. Such gas, fed to the system through line 10, may contain from about 0.4 to 3% helium, 5 to 30% nitrogen, 3 to 15% C₂ and heavier hydrocarbons, and the balance methane. It will be understood that the feed gas may have been pre-treated for such purposes as moisture and acid gas removal, to satisfactorily condition it for component separation or fractionation in accordance with the invention. Typically, the gas may be fed to the system at a pressure in the range of about 250 to 1000 p.s.i.g. and at a temperature that may range from around —40°F. up to higher temperatures in the neighborhood of atmospheric temperature, particularly where the gas contains no consequential L.P.G. components.

The column or shell 11 may be regarded as illustrative of any suitable chamber through which the feed gas is passed to be cooled by indirect heat exchange with the later described low temperature streams passing through coils contained in the chamber. The feed gas undergoes partial condensation in the chamber 11 and the gas and condensate mixture passes through line 12 to separator 13 from which the condensate composed essentially of C₃ and heavier hydrocarbons going to make up L.P.G. and natural gasoline, are withdrawn through line 14 to be passed through coils 15 in chamber 11, to L.P.G.—natural gasoline separation, purification and storage. The condensate may be recovered at a pressure from 20 to 250 p.s.i.g. and at a temperature in the range of about —80 to —140°F. The uncondensed components leave separator 13 through line 16 to pass through a second chamber 17 for further cooling by indirect heat exchange with the later described component streams.

The gas stream from line 16, containing helium, nitrogen, methane and C₂ hydrocarbons, is cooled in passing through the exchanger chamber 1y to temperature in the range of say —140 to —200°F., and is discharged through line 18 to an intermediate location in the frac-
tionating column 19. Depending upon the gas feed pressure to the system, the stream pressure may be reduced at valve 20 prior to introduction to the fractionating column. The latter is operated and controlled under particularly contemplated conditions providing for substantial methane content in the overhead gas stream leaving the column by line 21. Temperature and pressure conditions in the column are controlled so that the mixture going to line 21 will contain the helium and nitrogen content of the feed, possible minor quantities of C₂, hydrocarbons, and from 10 to 50 percent methane, the upper methane limit in this range being dependent upon the quantity of methane usable as fuel in the plant, i.e., for operation of the later-mentioned compressor 22, and possibly also for heating purposes in equipment serving to pre-treat the gas before its feed to exchanger 11. The column 19 may contain suitable packing or trays 23 as required for the fractionation, and also an upper exchanger section 24 (which may be inside the outside the column) comprising tubes through which the overhead flows to the outlet line 21. Depending upon such considerations as the gas composition and the percentage of methane to be retained in the overhead, the column 19 is operated in the pressure range of about 240 to 450 p.s.i.g. and at a top temperature, above the exchanger section 24, of about -170 to -240° F.

A portion of the condensate bottoms in column 19, and consisting for the most part of pure methane, minor percentages of C₂ fractions and possibly a small concentration (e.g. 2 to 5 percent) of nitrogen, is withdrawn from the base of the column at a temperature in the range of say -130 to -180° F., through line 24 and throttled at valve 241 to pass through exchanger tubes 25 and in heat exchange with the fractionating column feed stream being cooled in chamber 17, thence through line 26 and exchange tubes 27 in chamber 11, and finally to suitable disposition through line 28. More generally, throttling will be accomplished by valve 241 instead of at valve 20. A second portion of the fractionating column bottoms is withdrawn through line 29 to flow through tubes 30 in exchanger chamber 31, and thence to cooler 32 past valve 33 into the exchanger section 34 of column 19. The pressure reduction at valve 33 will be controlled to externally cool the tubes 24 and maintain the proper column top temperature. The refrigerant stream, consisting mainly of methane, leaves the exchanger section through line 321 to flow through chamber 31 to one or more appropriate compressors, diagrammatically indicated at 22, which operates to compress and discharge the prediately methane stream through line 34 to cooler 35. From the cooler the methane stream flows through line 36 and exchanger tubes 37 in chamber 31, and thence is introduced through line 38 to the lower portion of column 19 to serve as stripping gas. Thus, that portion of the fractionating column bottoms withdrawn through line 29 serves the dual purposes of a refrigerant for cooling the exchanger section in the column, and as stripping gas in the column, all to the end that the column operation will produce the desired concentration of methane in the column overhead.

The helium, nitrogen and methane mixture in line 21 is passed through exchanger chamber 40, wherein the methane is condensed and withdrawn through line 41, with or without pressure reduction at valve 42, to separator 43 containing typically a pressure in the range of about 190 to 2400 p.s.i.g. at a temperature of about -265 to -290° F. The uncondensed gases consisting essentially of helium and nitrogen, flow from the separator through line 44 to pass through the low temperature exchanger chamber 45 for further cooling and collection in the fractionated bottom section 46 in the chamber of condensate consisting essentially of nitrogen. The liquid nitrogen is withdrawn through line 47 and passed successively through exchanger coil 48 in chamber 45, line 49, coil 50 in chamber 40, line 51, exchanger coil 52 in chamber 17, line 53, coil 54 in exchanger 11, and thence to disposal through line 55.

The gas remaining uncondensed in exchanger 45 and consisting of helium together with a lesser quantity of nitrogen, say within the range of 10 to 40 percent nitrogen, is withdrawn through section 46 through line 56 and passed successively through line 57, coil 58, coil 59 in exchanger 49, line 60, coil 61 in exchanger 17, line 62, coil 63 in exchanger 11, and finally to storage or further disposition through line 64. As will be understood, this product helium-nitrogen gas may be subjected to further purification by any of the known methods for recovery of helium at the purity required.

Referring back to the separator 43, the methane-nitrogen condensate withdrawn therefrom through line 65, passes thence through coil 66 in exchanger 45, line 67, coil 68 in exchanger 59, line 69, coil 70 in exchanger 17, line 71, coil 72 in exchanger 11 and then through line 73 for use as fuel gas in the plant.

As previously observed, the compressor (22) operation and consequent refrigeration cost, are governed primarily by the methane purity desired in the fractionator column bottoms withdrawn through lines 24 and 29, and the methane composition of the fractionating column overhead stream in line 21. The top temperature, above the condenser section 24, is a temperature at about -30° F. to be cooled in the separator 13 at around -120° F. The stream leaving exchanger 17 enters column 19 at a temperature of about -190° F. and the conditions of fractionation under pressure of about 400 p.s.i.g. are such that the overhead gas leaves the column at about -225° F. with the liquid bottoms at about -150° F. Exchanger 40 operates to further cool the gas to a temperature in separator 43 of about -280° F. at 200 p.s.i.g. In chamber 45 the liquid nitrogen and gaseous impure helium separate at about -500° F. and at a pressure in the neighborhood of 190 p.s.i.g. The product helium-nitrogen stream recovered through line 64 will contain about 80 to 90 percent helium.

Generally considered, the ratio of the condensate returned for feed exchange from the fractionator 19 through line 24 to the second stream taken for refrigeration through line 29 will depend upon the nitrogen content of the feed gas. Under the usual operating conditions just cited, the quantity ratio of the condensate streams in lines 24 and 29 may be about 2 to 1. At lower nitrogen contents, say in the neighborhood of 13 percent, the corresponding ratio may be about 4 to 1, and in the fractionation of residue or refinery gases having still lower nitrogen content, the same ratio may range upwardly of 6 to 1.

I claim:

1. The process for low temperature fractionation of natural gas containing methane and nitrogen, that comprises cooling the gas to a low sub-zero temperature, feeding the cooled gas with its contained methane and nitrogen into a vertically extended fractionating zone partially condensing the methane content of the mixture in a condensing zone at the top of said zone and removing therefrom an overhead nitrogen-methane stream containing between 10-50% methane, passing an essentially methane stream from the bottom of said fractionating zone through one passage in a heat exchange zone, expanding the methane stream and passing it as coolant through said condensing zone and thence through a second passage in said heat exchange zone in heat transfer relation with the methane in said first passage, thereby precooling the methane stream in advance of its passage through said condensing zone then compressing the methane from said
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second passage, and cooling the compressed methane and returning it to said fractionating zone.

2. The process according to claim 1, in which the compressed and cooled methane is passed through said heat exchange zone in indirect heat exchange relation with methane in said second passage.

3. The process according to claim 1, in which methane is withdrawn from the base of said fractionating zone and is passed in indirect heat exchange relation with the methane-nitrogen feed to said zone.

4. The process according to claim 1, in which the methane-nitrogen mixture is fed to said fractionating zone at a temperature between about —140 and —200° F.

5. The process according to claim 4, in which said overhead stream is removed from the fractionating and condensing zones at a temperature between about —170 to —240° F.

6. The process according to claim 5, in which said overhead stream is passed through a condensing zone maintained at a temperature sufficiently low to condense out substantially all of the methane.

7. The process according to claim 1, in which the natural gas contains also L.P.G. hydrocarbons, and in which said hydrocarbons are condensed out of the gas in advance of the feed of residual methane and nitrogen to the fractionating zone.

8. The process according to claim 1, in which the natural gas contains also L.P.G. hydrocarbons, and in which the gas is cooled by indirect heat exchange with methane from said fractionating zone to condense said hydrocarbons out of the gas in advance of the feed of residual methane and nitrogen.

9. The process according to claim 1, in which the gas contains helium which is carried through the fractionating zone into said overhead stream, the process including the further step of condensing and separating methane and nitrogen from the overhead stream to leave a helium rich residual gas.

10. The process according to claim 9, in which said overhead stream is cooled to condense said methane and nitrogen therefrom by adiabatic pressure reduction and expansion of components of said stream and heat exchange between resultant liquid and vapor phases.

11. The process according to claim 9, in which methane separated from said overhead stream is passed in indirect heat exchange with said overhead stream to produce cooling from the methane condensation.

12. The process according to claim 9, in which the methane and nitrogen are sequentially condensed from said overhead stream, condensed nitrogen and said residual gas are individually passed in indirect heat exchange with said overhead stream to produce cooling for the methane condensation.

13. The process according to claim 12, in which streams of said condensed methane, nitrogen and said residual gases after said heat exchange with the overhead stream are heat exchanged with the nitrogen-methane feed to said fractionating zone.

14. The process according to claim 13 in which the natural gas fed to the system contains L.P.G. hydrocarbons, and said streams of methane, nitrogen and residual gases after heat exchange with the fractionating zone feed are heat exchanged with gas fed to the system containing said L.P.G. hydrocarbons to produce L.P.G. condensate.

15. The process for low temperature fractionation of natural gas containing methane, nitrogen, and helium, that comprises cooling the gas to a low sub-zero temperature, feeding the cooled gas with its contained methane, nitrogen, and helium into a vertically extended fractionating zone, partially condensing the methane content of the mixture in a condensing zone at the upper interior of said zone and removing therefrom an overhead nitrogen-methane-helium stream containing between 10–50% methane, circulating a normally liquid coolant fluid stream through said condensing zone, cooling said overhead stream to condensate and separate methane and nitrogen therefrom to leave a helium rich residual gas by expanding said coolant fluid at least partially to vapor into said condensing zone, and thereafter compressing and cooling the expanded fluid to convert it to liquid phase.

16. The process according to claim 15, in which the methane-nitrogen-helium mixture is condensed to said fractionating zone at a temperature between about —140 and —200° F., and said overhead stream is removed from the fractionating and condensing zones at a temperature between about —170 to —240° F.

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