IN REFERENCE TO THE ABOVE SYMBOLS:
"TV" INDICATES A THROTTLE VALVE.
"P.C.V." INDICATES A PRESSURE CONTROL VALVE.
"L.C.V." INDICATES A LEVEL CONTROL VALVE.

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NITROGEN REMOVAL FROM NATURAL GAS

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Filed Feb. 6, 1965, Ser. No. 87,242
10 Claims. (Cl. 62—21)

This invention relates to the removal from light hydrocarbon gases undergoing liquefaction of so-called "inert" gases such as hydrogen, helium, argon, oxygen, nitrogen, and any other gases having boiling temperatures lower than methane. Since most generally, nitrogen removal presents the major problem, the invention will be described with reference to the separation of nitrogen typically from natural gases where the primary object is to obtain liquid methane. The invention is not limited to natural gas but can apply to any light hydrocarbon gas containing inerts.

In any process for liquefying natural gas, the methane and heavier hydrocarbons can be condensed by cooling the compressed gas below its boiling point. The presence of gases having boiling temperatures below that of methane, necessitates cooling the gas to lower temperature than otherwise would be necessary for methane condensation. This requires additional expenditure of energy per unit of liquefied product obtained. Moreover, if the inerts were not removed, their concentration would increase in the refrigeration cycle to the point where liquefaction of the product would cease.

Heretofore it has been proposed to effect an initial methane condensation by cooling the compressed natural gas, and to vent or otherwise remove from the system the uncondensed inerts together with considerable quantities of methane upon which work has been done and energy expended in the compression and cooling. Alternatively the purge gas has been further processed in a distillation column. Such practices have been wasteful in terms of potential methane recovery in the case of the former and energy utilization in the case of the latter.

Our general object is to provide for greater efficiencies and economies in methane recovery from natural gas, by utilizing the energy potentials in a system involving nitrogen separation from refrigerated compressed natural gas at a rate equal to the nitrogen input rate, to controllably govern the methane content of the purged nitrogen stream in accordance with such methane content thereof as may be desired or needed for specific purposes, e.g. for fuel gas, while retaining in the system to final recovery, methane in excess of that desired quantity and which ordinarily would be lost from the system.

More specifically our objective is to utilize a combination of effects and conditions, including the temperature and pressure of the initially separated nitrogen stream, cooling by expansion thereof, and heat interchange, as required, with low temperature methane in the system, to condense and return to the system for final recovery, methane in the separated nitrogen which would be wasteful in the purged gas.

The invention, and the manner in which these and other objects are accomplished will be understood from the following description of an illustrative process embodying the invention, shown in flow sheet form by the accompanying drawing.

Referring to the drawing, the feed gas is delivered to the system through line 10 and compressor 11, where necessary, at a pressure typically in the range of 400 to 1500 p.s.i.g. The feed gas then is cooled by appropriate refrigeration, as by the conventional cascade cooling, at the refrigeration stage diagrammatically indicated at 12 and involving indirect heat interchange between the feed gas and a suitable refrigerant indicated to be passed through the refrigeration stage by way of line 13. Being precooled to a temperature that may be in the range of about —80 to —170° F. so that the feed gas is converted essentially to liquid phase, the refrigeration stage effluent is discharged through line 14 and pressure reduction valve 15 into a separating chamber 16 from which predominantly methane condensate is withdrawn through line 17.

For the purpose of ultimately producing the methane condensate at a temperature sufficiently low to permit storage at practically low pressures, we may employ a sequential flash and cascade system of adiabatic cooling, involving typically three flash stages beyond the separating zone 16. Thus the condensate in line 17 is flashed by pressure reduction at valve 18 into chamber 19, the vapor overhead from which passes through line 20 to be compressed and returned by compressor 21 into line 22, compressor 23 and line 24 into the feed gas stream entering the refrigeration stage 12 through line 10. Similarly, the condensate from chamber 19 is taken through line 25 to be flashed past valve 26 into chamber 27, the overhead from which passes through line 271 to compressor 28 for discharge through line 29 into line 20. Beyond chamber 27, the condensate is discharged through line 30 and flashed past valve 31 into storage tank 32, the vapor overhead from which is taken through line 33 to compressor 34 and returned to line 271.

Merely as illustrative of pressure gradations in the system, with the refrigeration stage effluent at the high pressure side of valve 15 in the neighborhood of 700 p.s.i.a., the preceding temperature and pressure conditions in the methane flash stages may be about 280 p.s.i.a. and minus 173° F. in chamber 16, 110 p.s.i.a. and minus 206° F. in chamber 19, 50 p.s.i.a. and minus 233° F. in chamber 27, and 15 p.s.i.a. and minus 255° F. in the storage tank 32.

The invention is more particularly concerned with the treatment given the gaseous effluent from the separating zone 16 removed through line 36. Heretofore, the usual practice has been to purge all or part of such effluent from the system, and in so doing to accept an uneconomical loss because the purge necessarily involves a loss of product on which work has been done, and a loss of energy which otherwise could be available for further cooling and condensation of the feed gas. The present invention departs from such uneconomical practices in that we take to ultimately purge only that portion of the vapor in line 36 which contains the quantity of inerts entering with the feed gas, and limit the amount of combustible gas in the mixture going to purge, to an economical ratio which usually is that which can economically be used in the plant as fuel. Generally considered, these requirements are accomplished by further treating that portion of the gas stream going to purge from line 36, so as to adjust the concentration of inert gases relative to combustible gases.

One portion of the gas stream in line 36 is returned...
3. Through line 37, to line 22 to be recycled by compressor 15 through line 24 to the feed stream. A second portion of gas through heat exchanger 39 and indirect heat exchange with the feed stream in the refrigeration zone 12, to be discharged at 45 to fuel or other position. The gas in chamber 39 withdrawn through line 48 past liquid level-controlled valve 47 for heat interchange with the gas streams in exchanger 39, thence to be discharged through line 49 into chamber 19 overhead in line 20.

In so processing the gas stream in line 38, we maintain by back pressure control at valve 44, a pressure between that existing in chambers 16 and 19 such that the ratio of inerts to combustibles in the overhead from chamber 42 will be that required to remove from the system the inerts entering with the feed gas stream, while limiting the combustibles, essentially methane, in the gas stream to an economical quantity. The purged gas is used in expansion zone 50 where about one third of the refrigeration potential in the liquid and gaseous effluent from chamber 42. This supplies most of the refrigeration required to adequately condense the purged gas stream. The balance of the necessary refrigeration may be supplied by flashing at valve 471, a stream of zone 16 condensate taken therefrom through line 481. The flashed stream is discharged from exchanger 40 through line 491 into line 48. If the temperature level so reached in exchanger 40 is insufficient to produce the desired separation of nitrogen, part of the liquid from chamber 19 may be substituted for liquid taken from chamber 16.

The cold, liquefied purged gas is flashed at valve 41 to produce a vapor in line 83 with the required percent of inerts. The liquid withdrawn from chamber 42 undergoes expansion at valve 47, thus lowering the temperature of the stream passing through exchanger 39 in precooled condensation with the gas stream in line 38.

It may be mentioned generally that depending upon various factors such as the ratio of inerts to fuel in the feed stream, temperature and pressure and conditions in the earlier stages, the described purged gas processing system can be located between chambers 19 and 27, or between chamber 27 and the storage tank 32, as may be most advantageous.

In the following tabulation we show typical stream flows (based e.g. on 100 mols of feed at point A) and positions at locations designated at A to F in the drawing, where the feed gas contains around 85 percent methane. Natural gases, or other light hydrocarbon gases, treatable in accordance with the invention may contain, as illustrative around 50 to 98 percent methane with relatively small amounts of heavier hydrocarbons and roughly 2 to 30 percent inerts.)

The described system is economical both in the cost of equipment required and in operating expense, contrasted with other (e.g. distillation) systems, no heat is required or rebolting and which must be subsequently removed. The energy lost at the pressure control valve 44 can be partially recovered in a power recovery turbine when economically justified.

The system is flexible and simple to operate since there are no distillation columns whose proper operation is sensitive to liquid or vapor overloading, and there are no moving parts to maintain. The adaptability of the system to varying quantities of inerts in the feed gas is particularly noteworthy. The quantity of inerts in the purge gas can be varied simply by adjusting the back pressure at valve 44. For even greater flexibility, duplicate piping can be arranged between expansion zones 19 and 27 so that the purge stream may be extracted from line 20 if desired.

We claim:

1. The process that includes cooling a feed stream of compressed natural or other gas containing methane and nitrogen in a refrigeration zone below the boiling temperature of methane, passing and expanding the cooled effluent from said zone into a separating zone, withdrawing liquid methane from said separating zone and further cooling the withdrawn methane, discharging from the last mentioned zone a separated nitrogen and methane gas stream, compressing and returning a portion of said separated gas stream to the feed stream in said refrigeration zone through passages leading from the separating zone to the refrigeration zone, passing the remainder of said gas stream at reduced pressure into a cooling and expansion zone, permitting the gas stream to exist as nitrogen-methane gas and essentially methane condensate, cooling said separated gas stream going to said cooling and expansion zone by passage through an exchange zone, separating the nitrogen-methane gas from said cooling and expansion zone, removing a stream of the withdrawn nitrogen-methane gas as purge gas from the system, and variably restricting the last mentioned stream to impose back pressure in said cooling and expansion zone varying in accordance with variations in said restriction of the last mentioned stream to thereby vary methane vaporization in the cooling and expansion zone and therefore the methane content in the purge gas stream.

2. The process according to claim 1, in which the pressure of said cooled separated gas stream is reduced after passage through said exchange zone.

3. The process according to claim 1, in which said separated gas stream going to the cooling and expansion zone is indirectly heat-interchanged with a stream of said essentially methane condensate withdrawn from said cooling and expansion zone.

4. The process according to claim 1, in which said separated gas stream going to the cooling and expansion zone is heat-interchanged in said exchange zone with said purge gas from the cooling and expansion zone.

5. The process according to claim 4, in which the pressure of the purge gas is controllably reduced between the cooling and expansion zone and said exchange zone.

6. The process according to claim 5, in which the purge gas is heat-interchanged with the feed gas in said refrigeration zone.

7. The process that includes cooling compressed natural gas containing methane and nitrogen in a refrigeration zone below the boiling temperature of methane, passing and expanding the cooled effluent from said zone into a separating zone, withdrawing liquid methane from said separating zone and further cooling the withdrawn methane by flashing the methane at reducing pressures within a series of flash zones with formation of methane vapor as a result of the reducing pressures, compressing and recycling the remaining gas from said feed gas stream and said refrigeration zone, withdrawing from the separating zone a separated nitrogen and methane gas stream, compressing and returning a portion of said separated gas stream to said refrigeration zone, passing the remainder of said gas stream at reduced pressure into a cooling and expansion zone where in the components of said gas stream exist as a nitrogen-methane gas component and an essentially methane condensate component, separately removing from said.
5 cooling and expansion zone said components as purge gas and condensate streams, thermally interchanging one of said components with said separated gas stream going to said cooling and expansion zone, combining the interchanged methane condensate component with methane withdrawn from said separating zone, and variably restricting said purge gas stream to impose back pressure in said cooling and expansion zone varying in accordance with variations in said restriction of the purge gas stream to thereby vary methane vaporization in the cooling and expansion zone and therefore the methane content in the purge gas stream.

8. The process according to claim 7, in which both of said components are heat-interchanged with said separated gas stream going to the cooling and expansion zone.

9. The process according to claim 7, in which the said combining of interchanged methane from the cooling and expansion zone is by addition of such methane to methane vapors from one of said flash zones.

10. The process according to claim 7, in which said separated gas stream going to the cooling and expansion zone is heat interchanged with cooled liquid methane derived from said separating zone and the interchanged methane is combined with the vapor from one of said flash zones.

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