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NITROGEN REMOVAL FROM NATURAL GAS

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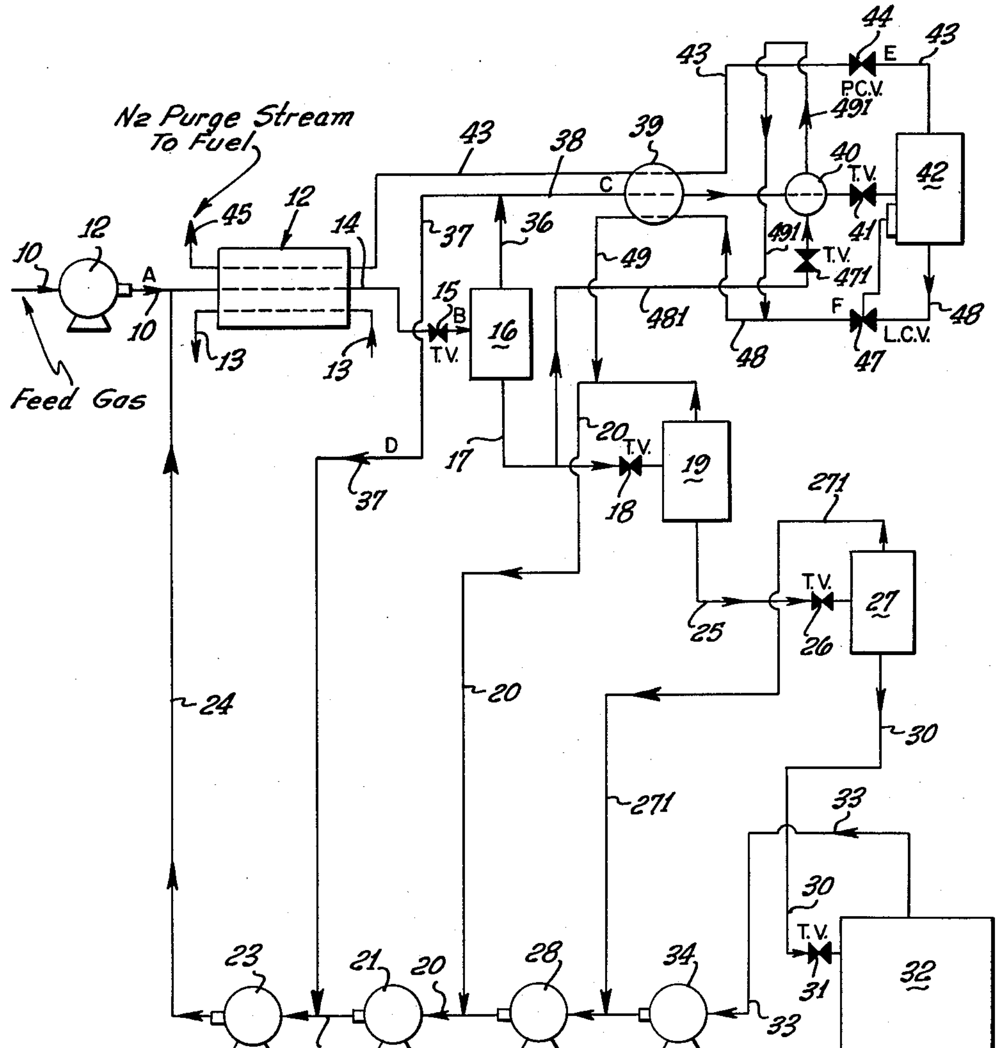

IN REFERENCE TO THE ABOVE SYMBOLS:
"T.V." INDICATES A THROTTLE VALVE.
"P.C.V." INDICATES A PRESSURE CONTROL VALVE.
"L.C.V." INDICATES A LEVEL CONTROL VALVE.

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NITROGEN REMOVAL FROM NATURAL GAS

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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

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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 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 predominently 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 practicably 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** through 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., succeeding 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 **37**, 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 hrough line **37**, to line **22** to be recycled by compressor **!3** through line **24** to the feed stream. A second portion ›f the separating zone effluent is discharged through line **!8**, and subjected to cooling by suitable means, typically ›y passage through heat exchangers **39** and **40** to be lashed past valve **41** into chamber **42**. The purged gas ›verhead therefrom is discharged through line **43** conaining back pressure valve **44**, through exchanger **39** and n indirect heat exchange with the feed stream in the efrigeration zone **12**, to be discharged at **45** to fuel or ›ther suitable disposition. The condensate in chamber **42** s withdrawn through line **48** past liquid level controlled 'alve **47** for heat interchange with the gas streams in ex:hanger **39**, thence to be discharged through line **49** into he chamber **19** overhead in line **20**.

In so processing the gas stream in line **38**, we mainain by back pressure control at valve **44**, a pressure ›etween that existing in chambers **16** and **19** such that he ratio of inerts to combustibles in the overhead from :hamber **42** will be that required to remove from the ystem the inerts entering with the feed gas stream, while imiting the combustibles, esssentially methane, in the ›urged gas to an economical quantity. The purged gas is :ooled in exchanger **39** where advantage is taken of the efrigeration potential in the liquid and gaseous effluent rom chamber **42**. This supplies most of the refrigeration equired to adequately condense the purged gas stream. The balance of the necessary refrigeration may be sup›lied by flashing at valve **471**, a stream of zone **16** conlensate taken therefrom through line **481**. The flashed tream is discharged from exchanger **40** through line **491** nto line **48**. If the temperature level so reached in ex:hanger **40** is insufficient to produce the desired separation ›f nitrogen, part of the liquid from chamber **19** may be ubstituted for liquid taken from chamber **16**.

The cold, liquefied purge gas is flashed at valve **41** to ›roduce a vapor in line **43** with the required percent of nerts. The liquid withdrawn from chamber **42** undergoes xpansion at valve **47**, thus lowering the temperature of he stream passing through exchanger **39** in precooling elation with the gas stream in line **38**.

It may be mentioned generally that depending upon 'arious factors such as the ratio of inerts to fuel in the eed stream, temperature and pressure and conditions in he earlier stages, the described purged gas processing ystem can be located between chambers **19** and **27**, or ›etween chamber **27** and the storage tank **32**, as may be nost economical.

In the following tabulation we show typical stream lows (based e.g. on 100 mols of feed at point A) and posiions at locations designated at A to F in the drawing, vhere the feed gas contains around 83 percent methane. Natural gases, or other light hydrocarbon gases, treatable n accordance with the invention may contain, as illustraive around 50 to 98 percent methane with relatively small .mounts of heavier hydrocarbons, and roughly 2 to 30 ›ercent inerts.)

	A	B	C	D	E	F
$_2$	4.9	24.5	5.9	10.4	4.9	1.0
H_4	83.3	171.8	15.7	27.6	4.2	11.5
$_2H_6$	6.8	7.0	.1	.1		.1
$_3H_8$	2.2	2.2				
$_4H_{10}$	1.3	1.3				
$_4H_{12}$	1.5	1.5				
	100.0	208.3	21.7	38.1	9.1	12.6

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

The system is flexible and simple to operate since there are no distillation columns whose proper operation is sensitive to liquid or vapor overloadings, 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 expending the cooled effluent from said zone into a separating zone, withdrawing liquid methane from said separating zone and further cooling the withdrawn methane, withdrawing 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 passage means 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 wherein the components of said gas stream 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, separately withdrawing methane condensate and 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 resulting methane vapor to the 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 wherein the components of said gas stream exist as a nitrogen-methane gas component and an essentially methane condensate component, separately removing from said

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.

References Cited by the Examiner

UNITED STATES PATENTS

2,162,963	6/39	McKittrick	62—23
2,557,171	6/51	Bodle	62—23
2,601,599	6/52	Denning	62—23
2,771,149	11/56	Miller	62—37
2,880,592	4/59	Davison	62—23
2,940,271	6/60	Jackson	62—23
2,959,020	11/60	Knapp	62—9
2,973,834	3/61	Cicalese	62—23
3,062,015	11/62	Cost	62—23

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