United States Patent [19]

Swearingen

[11] **3,747,358**

[45] **July 24, 1973**

[54] **CRYOGENIC PROCESS**

[76] Inventor: **Judson S. Swearingen,** 2235 Carmelina Avenue, Los Angeles, Calif. 90064

[22] Filed: **May 1, 1968**

[21] Appl. No.: **725,776**

[52] **U.S. Cl.** **62/12,** 62/27, 62/23, 62/39, 62/17

[51] **Int. Cl.** **F25j 1/00,** F25j 3/02, F25j 3/04

[58] **Field of Search** 62/12, 13, 2o, 27, 62/28, 38, 39, 20, 14

[56] **References Cited**

UNITED STATES PATENTS

2,031,118	2/1936	Merrill	62/24
3,283,521	11/1966	Harmens	62/23
3,306,057	2/1967	Harmens	62/28
2,824,428	2/1958	Yendall	62/14
2,846,853	8/1958	Matsch	62/14
2,873,583	2/1959	Potts	62/14
R25,193	7/1962	Schilling	62/27
3,187,064	6/1965	Wang	62/20
3,224,208	12/1965	Schlumberger	62/12
3,290,890	12/1966	Bray	62/12

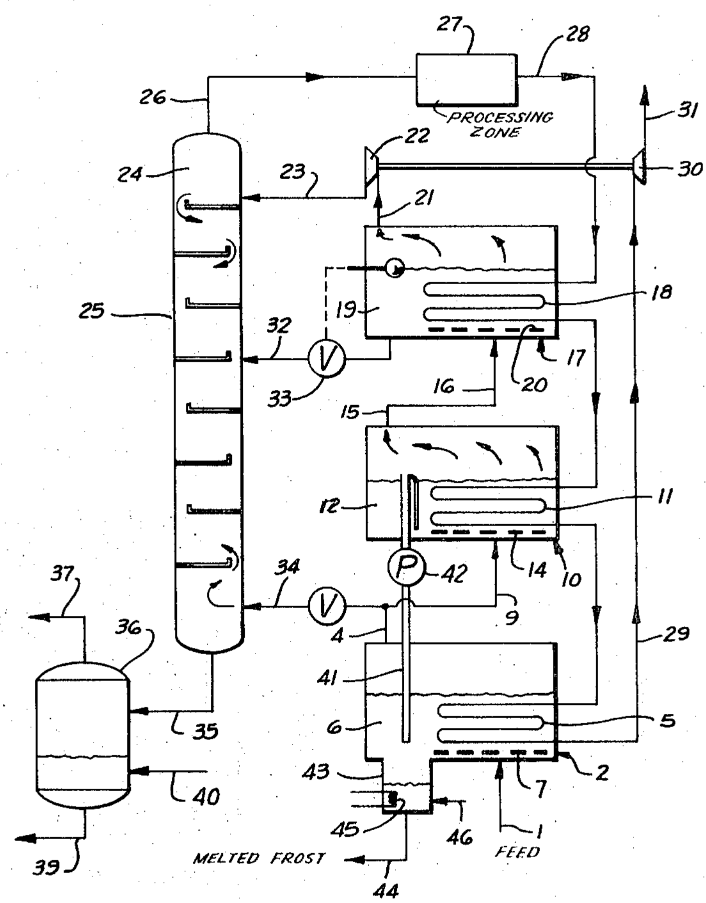
Primary Examiner—Norman Yudkoff
Assistant Examiner—Arthur F. Purcell
Attorney—Ralph R. Browning

[57] **ABSTRACT**

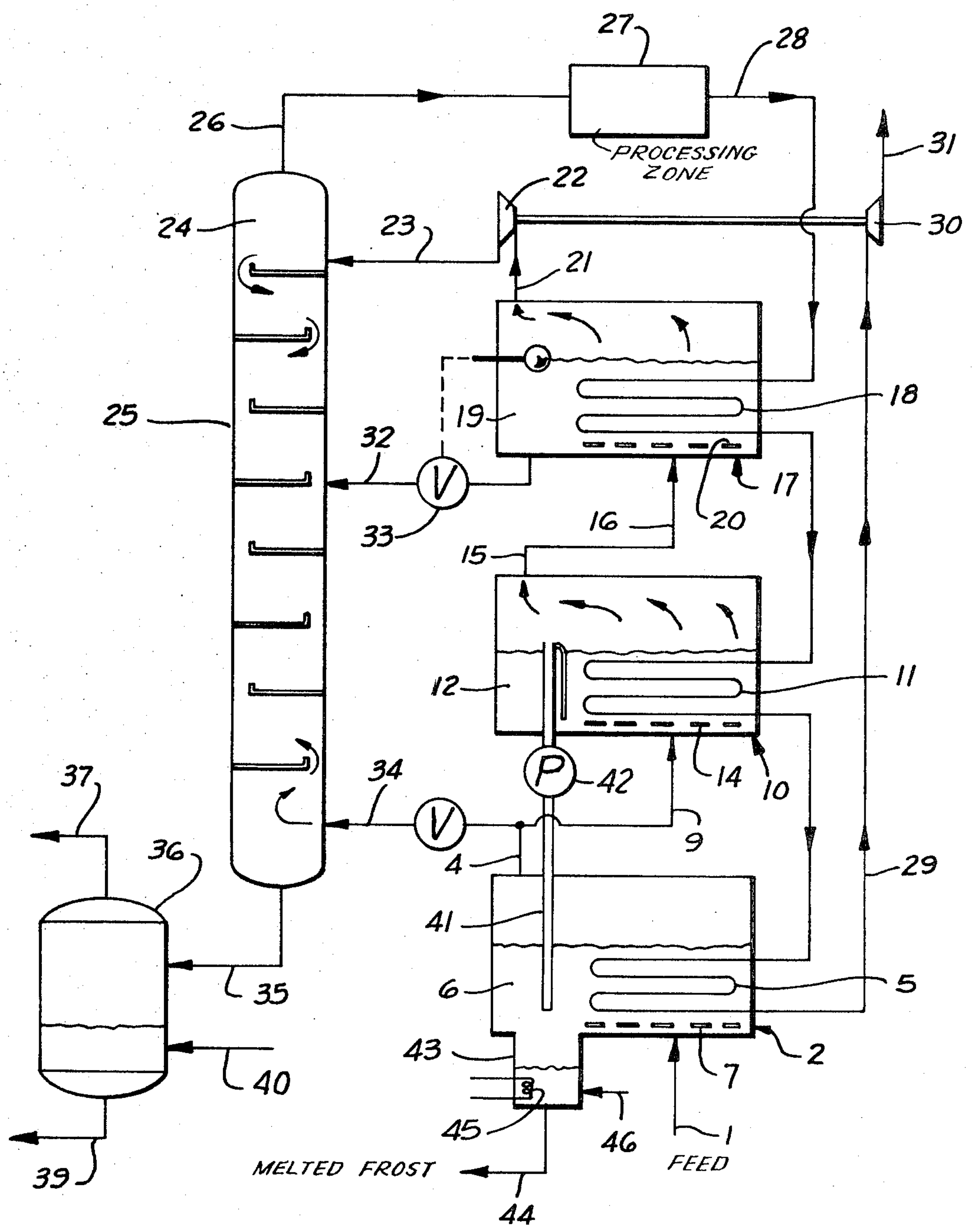
A process for the low temperature separation of pressurized raw gas into a condensed portion and a residue gas. Pressurized feed gas which may contain a frost forming constituent is chilled by passing it through a liquid medium in a series of chill chambers. The chambers are maintained at successively colder temperatures by refrigerant coils which are in series and are cooled by the returning chilled stream of residue gas. Any frost forming will be at the interfaces between the gas and liquid medium thereby preventing accumulation of frost on the heat exchange equipment.

The uncondensed gas from the coldest chill chamber is fed into a turboexpander which expands the uncondensed gas further chilling it and thereby condensing some of it. The turboexpander discharge is fed into a liquid disengaging zone at the top of a fractionating column where the condensed liquid is disengaged. The residue gas is discharged into the refrigerant coil system where its refrigeration content is recovered. The disengaged liquid acts as the top tray feed of the fractionating column. Condensed liquid from the chill chambers is fed into the fractionating column as a side feed. In addition, a warm stream of feed gas is fed into the bottom. This gas stream acts as a stripping vapor warming the downflowing product liquid to strip it of its more volatile constituents while at the same time being cooled by the colder liquid. Accordingly, the refrigeration in the product liquid is recovered. Again any frost formed will be on the surface of the liquid and carried down with the liquid. To assure that there will be a liquid medium in the warmer chill chamber, overflows are provided from colder chambers. Means are provided to collect and reject the dispersed frost.

12 Claims, 1 Drawing Figure


PROCESSING ZONE
MELTED FROST
FEED

PATENTED JUL 24 1973 3.747.358



JUDSON S. SWEARINGEN
INVENTOR.

BY
Browning, Hyer, Eickenroht & Thompson
ATTORNEYS

CRYOGENIC PROCESS

This invention relates in general to a cryogenic process for removing condensables from a gas containing a frost forming constituent. More particularly it relates to low temperature chilling of a raw feed gas and separation of a pressurized feed gas into a condensed portion and a residue gas.

The conventional method of low temperature processing of gas involves passing the feed gas through a heat exchanger where it is cooled by refrigeration from a cold countercurrent stream of the product. From the heat exchanger, the chilled gas passes into a separator (if liquid has been formed in the heat exchanger) and then through an expansion engine which further cools the chilled gas for recovery of additional liquefiable constituents. These liquids are recovered and the residue gas is cycled back through the heat exchanger for recovery of its refrigeration content.

If the feed gas contains water vapor, carbon dioxide or the like which forms frost on being chilled, such component must be removed in some way otherwise the frost and ice deposits will foul up the heat exchanger and impede the operation of the liquefication system. Further, unless the discharge temperature of the heat exchanger is extremely low, sufficient water vapor or carbon dioxide may be present in the chilled gas to foul the passages in the turboexpander as it further chills the gas. Thus, in such processes the water vapor must be removed down to a dew point preferably as low as −90° F. where the remaining water vapor content is too low to be very troublesome. If carbon dioxide is present, its content must be reduced to a point where the lowest temperature point to which the gas is cooled does not reach its frost point. The removal of water vapor and carbon dioxide to such a low level prior to introducing the feed gas into the liquefication system is difficult and costly.

Accordingly, it is a general object of this invention to provide a novel process for the low temperature separation of pressurized raw gas into a condensed portion and a residue gas which eliminates the necessity of reducing the water vapor, carbon dioxide or other frost forming constituents contents to extremely low levels.

Another object is to render a system for the low temperature separation of pressurized gas into a condensed portion and a residue gas more efficient by preventing the formation of frost on the solid portions of the system.

Still another object is to prevent the formation of frost on heat exchange equipment during the cooling of a raw feed gas which may contain a frost forming constituent.

A further object is to improve the low temperature separation of a pressurized gas into a condensed portion and a residue gas by recovering the refrigeration content of the product liquid.

A still further object is to improve the chilling and recovering of a partial condensate from a frost forming raw feed gas by collecting and rejecting any frost forming constituents.

Still another object is to provide purification system of the type described in which the removed frost forming constituents are not re-introduced into the residue gas, thereby leaving the residue gas pure.

In accordance with the present invention, these and other objects are accomplished in a gas liquefication system operating through a cycle in which the feed gas is chilled and partly reduced to a liquid phase and a residue gas, with the residue gas being recycled for chilling the feed gas in a heat exchange relation by passing the feed gas through successively colder bodies of liquid so that any frost formed is at the interfaces between the gas and the bodies of liquid and not on solid surfaces of the system.

More specifically in the system which is presently disclosed as an illustration of the invention, a series of successively colder bodies of liquid which may be chill chambers, each containing a liquid medium at successively lower temperatures is utilized to cool the feed gas. The feed gas is passed through a series of the successively colder bodies of liquid in gas-liquid contact. Any frost which tends to form as the feed gas is being chilled forms at the interfaces between the gas and the liquid. The resulting ice crystals float or are dispersed in the cold liquid rather than building up a frost or ice cake on the equipment. The chill chambers are kept chilled by refrigerant coils which are submerged in the liquid. The coils are in series and are refrigerated by recycling the chilled residue gas stream. In other words, the bodies of liquid are chilled by passing the return stream of chilled residue gas in non-contact heat exchange relation thereto.

The liquefiable constituents are accumulated in the successively colder chill chambers. As the accumulated volume of product liquid in a chill chamber becomes excessive it flows over into the next succeeding chamber along with the chilled gas. The final chill chamber has withdrawal means for removing the product liquid.

The cold end temperature difference in the heat exchange system is maintained by any suitable means, as for example, a turboexpander which expands the uncondensed gas from the coldest body of liquid. The expander or other refrigeration source also supplies the additional refrigeration for the latent heat of the condensables and frost as the feed gas is chilled.

If it is anticipated that the frost will deposit at a higher temperature and in advance of any deposit of product liquid, a suitable chilled liquid medium is provided in such chill chamber. In such case, a down flow tube is provided from the next colder chill chamber to spill some of the accumulated product liquid into the warmer chill chambers so that it acts as a contact medium for chilling the gas. Although this product liquid gradually evaporates, the evaporated portion is recovered in succeeding chambers and flows back so that its inventory is maintained and heavier constituents concentrated. Such down flow tubes have the further advantage that they bring the ice laden accumulated product liquid down to a warmer zone where the frost forming constituents may be segregated, heated to melt the ice and drawn off as a liquid.

Accordingly, the incoming feed gas is chilled and a portion of it condenses. The uncondensed portion is then further chilled by a gas expansion device which will usually condense an additional portion. If the process is for the recovery of a vaporizable hydrocarbon liquid from a pipeline gas, it may be desired to produce a product liquid reasonably free from the more volatile constituents. In such case, a fractionating column may be used. The discharge from the turboexpander is fed into a liquid disengaging zone of the fractionating column which disengages the product liquid and discharges the uncondensed gas as residue. The disengaged product liquid is used as a top tray feed. The uncondensed residue gas discharged from the fractionating column which is free of the removed frost forming constituents, is fed into non-contact heat exchange relation to at least one of the bodies of liquid. Condensed liquid from the colder chambers may be introduced into the fractionating columns at appropriate levels as a side feed. The fractionator may be at a pressure lower than the chill chambers in which case a portion of lower feed liquid flash vaporizes. In order to strip the down flowing product liquid in the fractionator, a small side stream of feed gas from one of the warmer chill chambers is introduced into the bottom of the fractionator. This recovers the refrigeration by countercurrent contact with the product liquid and cools the side stream of feed gas. This warmer gas acts as a stripper vapor and also as a source of heat to warm the down flowing cold product liquid so that it is partially stripped of its more volatile constituents while giving its refrigeration content over to the stripper stream of raw gas to simultaneously treat the latter.

The use of a fractionating column has two advantages. One is that it acts to recover the refrigeration content in the product liquid while it processes a parallel stream of partially chilled feed gas. This aids in balancing the temperature difference in the chill chambers. Another advantage is that the parallel side stream is chilled by contact with the surfaces of the down flowing product liquid so that any frost it tends to deposit will likewise be precipitated on the surface of the product liquid and carried down with the product liquid instead of being deposited on members where it could build up and interfere with the performance of the process.

The frost forming constituents may be separated and disengaged from the product liquid and a deliquescent liquid may be introduced into the disengaged portion to dissolve the frost forming constituents which may then be withdrawn as a liquid.

The principal advantage of the improved process of the present invention is that any water vapor, carbon dioxide, or other frost forming constituents form frost on the interfaces between the gas and the bodies of liquid and thus the resulting ice crystals float or are dispersed in cold bodies of liquid rather than forming frost on a solid portion which interferes with the operation of the system.

The operation of the present invention will be described in detail hereinafter with reference to the drawings in which:

The single FIGURE is a schematic drawing of a system for the low temperature separation of pressurized raw gas into a condensed portion and a residue gas in accordance with the present invention.

It will be apparent to those skilled in the art that the teachings of the present invention are not restricted to the particular type of liquefication cycle employed in the disclosed illustrative embodiment, but may be used advantageously in conjunction with any suitable liquefication cycle in which similar problems are encountered, particularly where a feed gas stream which may contain frost forming constituents is chilled in a heat exchange relation with a cold fluid or cold surface.

A typical application for the invention is the recovery of a vaporizable hydrocarbon liquid from a pipeline gas. This liquid can be stored at a moderately low temperature in large quantity and be used for "peak shaving" by vaporization and reinjection into the gas pipeline. In such case the raw pipeline gas often contains water vapor and carbon dioxide and other frost forming constituents.

Referring to the drawings, a stream of feed gas from a pipeline at a pressure of several hundred p.s.i. is introduced at ambient temperature into the system at inlet **1** of a chill chamber **2.**

The chill chamber is an insulated compartment having said inlet **1** at its bottom, an outlet **4** at its top, and a set of refrigerant coils **5** which are submerged in a liquid medium **6** therein. Positioned over inlet **1** is a perforated distributor plate **7** which causes the feed gas stream entering at inlet **1** to be distributed over refrigerant coils **5** as the bubbles of gas rise through the liquid medium. The liquid medium in chill chamber **2** is colder than the feed gas in the inlet stream; accordingly, the feed gas is cooled and partially condensed as it passes up through the liquid medium. The cooled gas leaves the chill chamber **2** through outlet **4** which is connected to inlet **9** of the next succeeding chill chamber **10,** which is similar in construction to chill chamber **2.**

Chill chamber **10** has a refrigerant coil **11,** submerged in a liquid medium **12,** which is connected in series with refrigerant coil **5** of chill chamber **2.** Positioned over inlet **9** is a distributor plate **14** which distributes the incoming gas around refrigerant coil **11** as the incoming gas bubbles through liquid medium **12.** Chill chamber **10** has an outlet **15** through which the further chilled gas exits. If sufficient gas is condensed it may overflow the chill chamber in which event it will be entrained with the feed stream exiting through outlet **15.** Outlet **15** is connected to inlet **16** of chill chamber **17** which is at a lower temperature than the preceding chill chambers.

Chill chamber **17** is similar in construction to the preceding chill chamber having a refrigerant coil **18** submerged in a liquid medium **19** and perforated distributor plate **20.** While only three chill chambers are illustrated it is to be understood that there may be twenty or more in a series arrangement extending from a warmer temperature to a colder temperature.

The refrigerant coils in the chill chambers are connected in series. One means of providing refrigerant to the refrigerant coils is to recycle the residue gas. In any event, the coldest chamber receives the refrigerant at the lowest temperature.

Chill chamber **17** or whichever chill chamber is the coldest has an outlet **21** through which the uncondensed feed gas leaves the heat exchanger. The feed gas will still be at substantially the same pressure as at the time of its entry into the heat exchanger through inlet **1.** From outlet **21** the feed gas stream flows into an expansion engine **22** which is illustrated as being a turboexpander wherein the feed gas stream is further chilled and an additional portion condensed. The discharge from the expansion engine **22** flows through line **23** into a liquid disengaging zone **24** of a fractionating column **25.** The uncondensed cold gas from the turboexpander is disengaged from the liquid portion and flows through a residue gas discharge line **26,** through a processing zone **27** and then through line **28** which connects with the series of refrigerant coils of the chill chamber. The frost forming constituents are not reintroduced into the residue gas, therefore, the residue gas is clean. The refrigeration content of the uncondensed gas stream is given up to the liquid medium in the successive chill chambers and provides the refrigeration needed to chill the incoming feed stream and condense some of its constituents.

The residue gas by giving up its refrigeration content is warmed and leaves the heat exchanger through an outlet line 29 extending from warmest chill chamber 2. If turboexpander 22 is utilized, the power from it may be used by a compressor 30 to recompress the warmed residue gas and discharge it through discharge line 31. Ordinarily the pressure at which residue gas is discharged is controlled and the utilization of the turboexpander power in compressor 30 permits a lower level of pressure in the system and thus provides a wider expansion ratio for the turboexpander which then produces a greater amount of refrigeration and accordingly a higher recovery of liquefiable constituents.

As was previously mentioned, the discharge from the turboexpander is fed into liquid disengaging zone 24 of fractionating column 25. This space is the top compartment of fractionating column 25 and separates the turboexpander discharge into a gas phase and a liquid phase. The disengaged liquid acts as a top tray feed or reflux.

Each of the successively colder chill chambers accumulate condensed liquid. As mentioned, this liquid may be allowed to simply flood the chambers and flow over along with the partially chilled gas into the next succeeding cold chamber so that the condensed product along with the uncondensed gas all finally reaches the coldest chill chamber. This has the desirable effect of introducing the heavier constituents into the fractionator to absorb additional liquefiable constituents. The liquid accumulating in the coldest chill chamber may be discharged through a liquid discharge line 32 which is controlled by a level control valve 33. This stream is introduced as a side feed stream into fractionator 25.

The lower part of fractionator 25 is a stripping section. A stream of feed gas or partially processed gas may be delivered into the stripper section of fractionator 25 through a valved line 34 which is connected to outlet 4 of warmest chill chamber 2. This warm stream of gas passes up through fractionator 25 to strip the more volatile constituents from the liquid product stream coming down through the fractionator. At the same time, the gas stream is itself stripped of liquefiable constituents because it contacts successively colder product liquid as it passes up the fractionator toward liquid disengaging zone 24 at the top of the fractionator. The uncondensed portion of the gas feed stream which reaches liquid disengaging zone 24 passes out through residue gas discharge line 26. Processing this stream of stripping gas requires refrigeration which is recovered from the chilled raw product liquid disengaged from the gas phase in liquid disengaging zone 24 and from the raw liquid from line 32. The product liquid stripped of its refrigeration content leaves the fractionator through a flow line 35 attached to the bottom of fractionator 25.

As was previously mentioned, the feed gas stream entering inlet 1 may contain frost forming constituents such as water or carbon dioxide. Such water vapor successively condenses in the chill chambers and becomes suspended particles of ice in the product liquid. The frost dispersed in the product liquid passes through flow line 23 from coldest chill chamber 19 and down through the fractionator so that the product liquid coming out of flow line 35 at the bottom of the fractionator may contain suspended frost particles. If the temperature in flow line 35 is above the freezing point, these frost particles will melt and there will be suspended particles of water in the flow stream. The flow stream may be fed into a separator 36 where the water and dispersed frost will settle to the bottom. The hydrocarbon liquid may be withdrawn through top discharge line 37 as product liquid.

If the ice particles have melted and are present as water, the water can be withdrawn through line 39 at the bottom of separator 36. If there are still ice particles, then means may be provided to melt the dispersed frost so that it can be withdrawn as a liquid through line 39. For example, a deliquescent liquid, such as alcohol, may be introduced into separator 36 through line 40. The alcohol will mix with the settling ice crystals and dissolve them and the alcohol water solution is withdrawn through line 39. The alcohol may be recovered and returned through line 40. The alcohol is not intimately in contact with the product stream which might dissolve a portion of it and carry it away. The settling particles of water or ice settling to the bottom of separator 36 act to absorb any alcohol which may have dissolved in the hydrocarbon and be diffusing upward. Thus, the settling water and dispersed frost act to recover the alcohol should it tend to dissolve.

Since frost may form at a temperature higher than the hydrocarbon dew point, it is desirable that liquid be supplied to the warmest chill chamber so that there will be a liquid medium for the feed gas to contact and therefore no frost will form on the refrigerant coil in such chamber. Accordingly, the various chill chambers may be provided with overflow tubes 41 each of which may be provided with a pump 42. Therefore, there will be a supply of liquid medium in each of the chill chambers. While a portion of this overflow liquid may evaporate by moving to a warmer chill chamber, it will be recovered in the colder chill chambers and returned. Accordingly there will be an inventory of liquid in each of the warmer chill chambers so that the incoming feed gas will make contact with a liquid medium. Accordingly, any frost which forms will form on the surface of the liquid medium and become suspended as particles of ice in the liquid medium rather than be deposited on a solid surface which could impede the operation of the system.

If the liquid flowing down overflow tubes 41 floods the lower chamber, a portion of it will be carried back into the colder chambers by being entrained along with the partially chilled gas. Thus, the frost is carried into the cold end of the cascade of chill chambers.

However, the warmest chill chamber 2 may be provided with a settling section 43 in which event the dispersed frost which is brought to the warmest chamber may be melted and withdrawn as a liquid through line 44. In such event, settling section 43 may be provided with a heater 45 to melt the dispersed frost. A line 46 to introduce alcohol or a like diliquescent liquid into settling section 43 may also be provided.

As can be seen, from the foregoing by passing the feed gas in heat exchange relation with successively colder bodies of liquid any frost forming occurs at the interfaces between the gas and the bodies of liquid rather than on a solid surface of the system. Accordingly the present invention fulfills the objectives of chilling and recovering a partial condensate from a raw feed gas having a frost forming constituent. Moreover, any frost is collected and rejected. The process partially stabilizes the product liquid so as to minimize the refrigeration necessary for its storage, or conversely minimizes the pressure at which it would be stored at any temperature above its boiling point. It provides a simple system capable of operation unattended. The process is adaptable to installations of moderate size and is capable of operation over a wide range of capacity by utilization of simple controls.

From the foregoing it will be seen that this invention is one well adapted to attain all of the ends and objects hereinabove set forth, together with other advantages which are obvious and which are inherent to the process.

It will be understood that certain features and subcombinations are of utility and may be employed without reference to other features and subcombinations. This is contemplated by and is within the scope of the claims.

As many possible embodiments may be made of the invention without departing from the scope thereof, it is to be understood that all matter herein set forth or shown in the accompanying drawings is to be interpreted as illustrative and not in a limiting sense.

The invention having been described, what is claimed is:

1. A process for the low temperature separation of a pressurized feed gas into a condensed portion and a residue gas without frost accumulation, said process comprising first passing the pressurized feed gas at initial feed temperatures into and through a body of liquid colder than the incoming feed gas and in gas-liquid contact therewith to condense and separate from the gas and incorporate into the liquid any constituents condensible at the temperature of said liquid body, and repeating contact of the residue of said gas with successively colder bodies of liquid until the condensible constituents remaining the gas are removed to a desired degree, whereby any frost formed from said constituents is at the interface between the gas and liquid and incorporated into the liquid, and separating the residue gas from the various bodies of liquid, wherein the bodies of liquid are chilled by further chilling a portion of the residue gas separated from said liquid bodies after passing therethrough by expanding said portion of said residue gas exiting from the coldest body of liquid in a turboexpander, and passing a returned stream of such chilled residue gas in heat exchange relation to the liquid bodies while keeping the liquid and the residue gas in such relation out of direct engagement with one another.

2. A process for the low temperature separation of a pressurized feed gas into a condensed portion and a residue gas without frost accumulation, said process comprising first passing the pressurized feed gas at initial feed temperatures into and through a body of liquid colder than the incoming feed gas and in gas-liquid contact therewith to condense and separate from the gas and incorporate into the liquid any constituents condensible at the temperature of said liquid body, and repeating contact of the residue of said gas with successively colder bodies of liquid until the condensible constituents remaining in the gas are removed to a desired degree, whereby any frost formed from said constituents is at the interface between the gas and liquid and incorporated into the liquid, and separating the residue gas from the various bodies of liquid, wherein the bodies of liquid are chilled by further chilling a portion of the residue gas separated from said liquid bodies after passing therethrough by expanding said portion of said residue gas exiting from the coldest body of liquid in a gas expansion device, feeding the gas discharge from the gas expansion step into a liquid disengaging zone of a fractionating column - disengaging the product liquid from the gas in said column and using the liquid from said disengaging step as a top feed for said column, passing a returned stream of such chilled residue gas from said disengaging step in heat exchange relation to the liquid bodies while keeping the liquid and the residue gas in such relation out of direct engagement with one another, and discharging the uncondensed residue gas.

3. The process specified in claim **2**, including feeding the uncondensed gas discharged from the fractionating column into heat exchange relation to at least one of said successively colder bodies of liquid while keeping said liquid and gas in such relation out of direct engagement with one another.

4. The process specified in claim **2**, including introducing overflow accumulation of liquid from one of the successively colder chill chambers into the fractionating column at an appropriate level as a side feed.

5. The process specified in claim **2**, including introducing a stream of warm feed gas into the bottom of the fractionating column to recover the refrigeration content of the product liquid and cool a parallel stream of feed gas.

6. The process specified in claim **2**, including separating and disengaging any frost forming constituents from the overflow accumulation of liquid from the successively colder chambers to obtain a product liquid.

7. The process specified in claim **6**, including introducing a deliquescent liquid during the separating step to dissolve the frost forming constituents.

8. The process specified in claim **7**, including withdrawing the solution of deliquescent liquid and frost forming constituents from the separator.

9. A process for the low temperature separation of a pressurized feed gas into a condensed portion and a residue gas, said process comprising passing the pressurized feed gas through a series of successively colder bodies of liquid in gas-liquid contact whereby any frost formed is at the interface between the gas and liquid, wherein the bodies of liquid are cooled by refrigerant coils submerged therein and the process includes the step of distributing the feed gas throughout the bodies of liquid so that the feed gas moves past the refrigerant coils.

10. A process for the low temperature separation of a pressurized feed gas into a condensed portion and a residue gas without frost accumulation, said process comprising first passing the pressurized feed gas at initial feed temperatures into and through a body of liquid colder than the incoming feed gas and in gas-liquid contact therewith to condense and separate from the gas and incorporate into the liquid any constituents condensible at the temperature of said liquid body, and repeating contact of the residue of said gas with successively colder bodies of liquid until the condensible constituents remaining in the gas are removed to a desired degree, whereby any frost formed from said constituents is at the interface between the gas and liquid and incorporated into the liquid, and separating the residue gas from the various bodies of liquid, including feeding the condensed liquid produced from the condensation of the chilled gas in the colder of said successively colder bodies to the warmer of said successively colder bodies to provide an adequate supply of liquid to act as a contact medium with the feed gas in such warmer bodies.

11. The process specified in claim **10,** including accumulating the condensed liquid containing frost forming

constituents into the warmest of the bodies of liquid and disengaging said frost forming constituents and withdrawing them.

12. The process specified in claim **11,** including segregating the condensed liquid containing dispersed frost, heating it and melting the dispersed frost to permit its withdrawal as a liquid.

* * * * *