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METHOD OF PUMPING LIQUEFIED GASES CONTAINING NITROGEN

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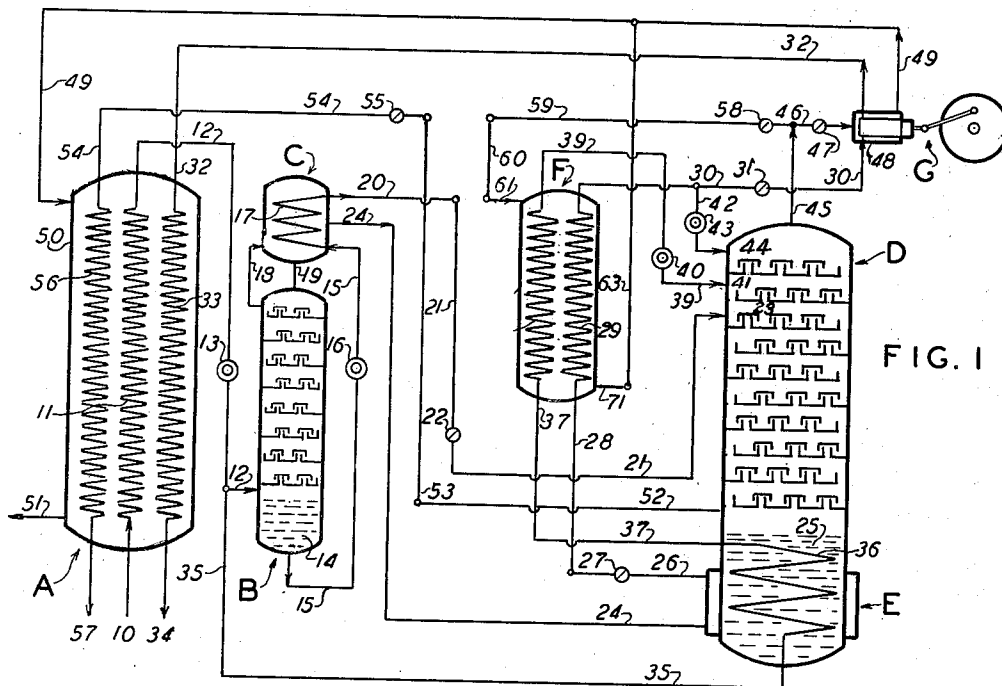


FIG. 1

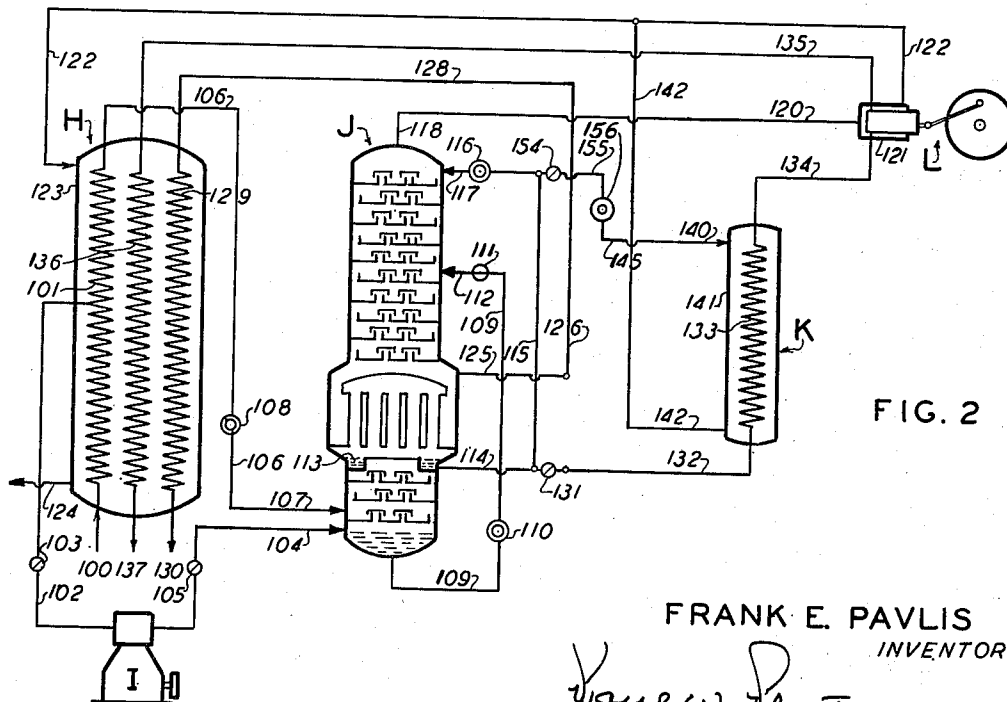


FIG. 2

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In the operation of plants producing oxygen and nitrogen by the liquefaction and fractionation of air, it is often desirable to provide a supply of nitrogen at a pressure considerably higher than that at which it exists as a separated product at any point in the fractionating cycle. Heretofore such supplies have been obtained by recompressing the final gaseous product nitrogen, which normally comes from the warm air interchanger at substantially atmospheric temperature and pressure.

The recompression of expanded gaseous products to high pressures is in several respects a wasteful and undesirable process. It requires multistage compressors which, if oil-lubricated, contaminate the recompressed gas with hydrocarbons, while if water is used for lubrication it is usually necessary to redry the compressed gas by passage through an adsorbent such as silica gel. Therefore the total plant required is costly, occupies considerable valuable space, and has an unduly high power consumption.

I have discovered that a material and useful portion of the nitrogen content of the air supplied to an air fractionating operation may be withdrawn, in liquid form or as a vapor subject to liquefaction; that this withdrawn portion may be cooled, or condensed and cooled, to a temperature at which it may be pumped in liquid form without risk of gas-locking the pump, and that the refrigeration required for such cooling or condensation and cooling may be produced through the use of higher-boiling column products by maintaining suitable pressure differences between the cooling agent and the liquid to be cooled or the vapor to be condensed and cooled.

By pumping in liquid form to the higher pressure required for storage in pressure tanks or cylinders or for delivery into pipe lines, and vaporizing and warming the high-pressure liquid by heat interchange with the entering air, material advantages are gained at the cost of only the amount of refrigeration represented by the difference between the heat contents of the gas in compressed and in expanded form. These advantages include great economy in plant and floor space in the substitution of a simple (usually plunger-type) liquid pump for the elaborate recompressing station, and the complete avoidance of contamination, such pumps needing no liquid lubrication. Safety is increased because of the reduction in the size and the temperature level of the compressing means and the freedom from contamination by lubricants. In addition, there are usually desirable reductions in power con-

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sumption, operating attention, and maintenance.

The invention may be practiced in the operation of either a single-stage or a two-stage column in the manners illustrated in the attached drawings and the following description thereof, in which:

Fig. 1 is a flow sheet of a single-column operation in which the nitrogen is withdrawn as a vapor from a preliminary partial fractionation and is condensed and cooled by the gaseous product nitrogen from the column, and

Fig. 2 illustrates in a similar manner an operation in which liquid nitrogen is withdrawn from the high-pressure stage of a two-stage column and is cooled by expanded high pressure nitrogen product from the high-pressure stage.

Referring first to Fig. 1, the essential elements of apparatus are: the primary interchanger A, which must have four flow circuits but otherwise may be any one of the many conventional types; a nitrogen separator or preliminary fractionator B, which may be either a packed or a bubble-tray column; a condenser C of the shell-and-tube or shell-and-coil type providing reflux for this fractionator; a single-stage fractionating column D, which may be of the packed type but preferably is provided with bubble trays; a nitrogen condenser E, arranged around or within the lower end of the main column D, a nitrogen subcooler F, which is basically a two-flow circuit interchanger but is frequently built as a three-flow interchanger for reasons of better operation or flexibility; and a pump G, adapted to handle highly volatile liquids.

The cycle illustrated in Fig. 1 functions in the following manner: Air under a desired relatively high pressure, for example from 500 to 2500 pounds per square inch gauge, and at least substantially freed from vapors of water and carbon dioxide, is introduced at 10 into a coil 11 in interchanger A, in which it is in counterflow heat transfer with product nitrogen and oxygen and with the pumped nitrogen stream. Leaving the interchanger through conduit 12, the stream of partially refrigerated air is expanded in passing through expansion valve 13 to an intermediate pressure which for example usually varies from 75 pounds to 200 pounds per square inch gauge. This expansion liquefies part of the air, a portion of which enters separator B as a mixture of liquid and saturated vapor.

The oxygen-enriched air collecting in liquid pool 14 in the base of the separator passes through conduit 15 and an expansion valve 16, by which its pressure is still further reduced, for example

to 10 pounds per square inch gauge or less, into a coil 17 in condenser C. The jacket of this condenser is supplied with nitrogen vapor through conduit 18, the condensate returning at 19 to supply reflux liquid to the column. The oxygen-enriched air stream then flows through conduits 20 and 21 and stop valve 22 into column D at a medial height, as at 23.

In this and the following description, it will be understood that valves mentioned by number are open unless otherwise stated.

The portion of the nitrogen vapor that is not liquefied in condenser C passes through conduit 24 to nitrogen condenser E, in which it is liquefied by heat interchange with pure oxygen boiling in a pool 25 in the base of column D. The resultant liquid flows through conduit 26, stop valve 27, and conduit 28 to a coil 29 in interchanger F, in which it is subcooled in a manner later described. The subcooled liquid stream passes through conduit 30 and valve 31 to the suction side of pump G, at which point it is still at substantially the intermediate pressure. Being raised by the liquid pump to a desired higher pressure, the stream flows through conduit 32 to coil 33 of interchanger A, in which the liquid is vaporized and the vapor brought to atmospheric temperature by heat interchange with the entering air feed. The gaseous stream is finally discharged at 34, at the elevated pressure produced by the liquid pump, into cylinders, a storage tank, or a pipe line, not shown.

The low-pressure gaseous nitrogen separated in column D flows through conduits 45 and 46 and valve 47 to a jacket 48 surrounding the cold end of pump G, in which it functions to absorb heat entering the cold portion of the pump through the insulation or generated within the pump as friction, and thence through conduit 49 to the shell 50 of interchanger A, in which it is brought to atmospheric temperature and is vented from the cycle at 51.

The pure oxygen separated in the column flows through conduits 52, 53, and 54 and valve 55 to a coil 56 in interchanger A, from which it is vented at 57 at substantially atmospheric temperature and pressure.

When the apparatus represented by Fig. 1 is used to produce oxygen, and no compressed nitrogen is desired, the nitrogen stream liquefied in condenser E passes through nitrogen subcooler F, through conduits 30 and 42 and expansion valve 43 to the top of the column D as at 44, where it functions as a useful reflux liquid.

As mentioned above, the liquefied stream of nitrogen is subcooled in the interchanger F as it flows through the coil 29 on its way to the suction side of the pump. This subcooling is accomplished by passing a stream of gaseous product nitrogen to the shell of the interchanger F in heat exchange relation with the coil 29. For this purpose a portion of the gaseous nitrogen product stream vented from the column D by the conduit 45 is conducted past a valve 58 and through conduits 59, 60 and 61 into the shell 62 of the interchanger F. After passing in heat exchange relation with the coil 29, the stream of gaseous product nitrogen leaves the shell 62 by way of a conduit 71 from whence it is conducted through conduit 63 to the conduit 49 where it merges with the stream of gaseous product nitrogen leaving the pump jacket 48 on its way to the main interchanger A. The valves 47 and 58 are adjusted to provide the necessary flow of gaseous

product nitrogen through the interchanger F to provide the desired degree of subcooling.

Referring now to Fig. 2, the essential elements of apparatus are: the primary interchanger H; an expansion engine I, which may be of the reciprocating or the turbine type; a two-stage fractionating column J, which may be of any conventional or preferred form; a two-pass heat interchanger K used as a liquid-nitrogen subcooler, and a liquid pump L, which may be a rotary pump or a two-stage centrifugal but is preferably a simple plunger pump.

The cycle illustrated in Fig. 2 functions in the following manner. Air previously raised to a desired pressure, for example from 250 pounds to 1000 pounds per square inch gauge, and suitably purified, is introduced at 100 into coil 101 of primary interchanger H, in which it is refrigerated by heat interchange with cold column products. A portion of the air stream is withdrawn at a medial point in the length of the coil and passes through conduit 102 and a regulating and shut-off valve 103 to an expansion engine I, by which its pressure is reduced to that carried in the high-pressure stage of the column, for example, 5 to 6 atmospheres absolute. The exhaust from the engine passes through conduit 104 and a stop valve 105 to the lower end of column J.

The remainder of the air supply traverses the full length of coil 101 and is likewise introduced into the lower end of the column through conduits 106 and 107 and expansion valve 108. The air supplies passing through the expansion engine and the expansion valve may be proportioned as desired, and it is possible, by suitably controlling the initial air pressure, to dispense with the use of the expansion engine.

The crude oxygen separating in the high-pressure-column stage passes in the usual manner through conduit 109, expansion valve 110, and conduit 112 into the low-pressure-column section, which is maintained at a lower pressure, as for example from 2 to 10 pounds per square inch gauge.

The liquid nitrogen collecting in a pool 113 in the upper end of the high-pressure section flows through conduits 114 and 115, expansion valve 116, and conduit 117 into the upper end of the low-pressure section.

Low-pressure gaseous nitrogen separating in the upper section of the column flows through conduit 118 and conduit 120 to a jacket 121 surrounding the cold end of liquid pump L, from which it passes through conduit 122 to the shell 123 of primary interchanger H, from which it is vented at 124.

Pure oxygen separating in the low-pressure stage flows through conduits 125, 126 and 128 to coil 129 of the primary interchanger, from which it is vented at 130.

A desired portion of the liquid nitrogen collecting in pool 113 may be directed to the intake side of pump L through valve 131 and conduit 132, the stream passing through a coil 133 in cooler K in which it is brought to a desired lowered temperature in a manner described below. The subcooled liquid reaches the suction side of the pump through conduit 134 at substantially the pressure maintained in the high-pressure-column section, is raised by the pump to a desired higher pressure, and is discharged through conduit 135 to a coil 136 in the primary interchanger. In this coil the liquid is vaporized and brought substantially to atmospheric temperature by heat

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interchange with the entering air feed and is discharged at 137, in gaseous form and under the pressure created by the pump, into storage or transportation vessels or into a pipe line, not shown.

The subcooling of the liquid nitrogen stream in the coil 133 of the cooler K may be produced by passing an expanded stream of high pressure liquid nitrogen in heat exchange relation with the coil 133. For this purpose a portion of the liquid nitrogen stream withdrawn from the pool 113 is diverted from the conduit 115 through a valve 154, and the diverted stream is conducted through a conduit 155, expansion valve 156 and conduit 140, to the shell 141 of the cooler K. After passing in heat exchange relation with the coil 133 the expanded diverted stream of liquid nitrogen flows from the shell 141 through a conduit 142 which connects with the conduit 122 to merge the diverted stream with the stream of gaseous product nitrogen on its way to the main interchanger H.

The liquids separating in a gas-fractionating column are normally in equilibrium with the vapors above them. In pumping such liquids out of the column, fluid friction in the intake line and the weight of the intake valve of a reciprocating pump cause a drop in pressure between the point of supply and the barrel of the pump, resulting in the evolution of vapor in reaching a new equilibrium at the lowered pressure. In the handling of these extremely volatile liquids, even a very slight drop in pressure causes sufficient vapor evolution to make it difficult or impossible to maintain the pump in smooth and continuous operation.

This difficulty is avoided in the operations herein described by cooling the liquid, in transit to the pumping step, to such degree that the unavoidable reduction in pressure is insufficient to bring the liquid to a new equilibrium at the reduced temperature. Assuming sufficient temperature reduction, this step is a positive cure for gas-locking and ensures continuous pump operation without short-stroking.

The extent to which the pumped liquid must be subcooled will, of course, depend on the pressure drop between the source of supply and the pump barrel in the particular installation, and on the amount of heat conducted to the liquid line through the insulation material surrounding it. Ordinarily a temperature reduction equivalent to that required to reduce the vapor pressure of the liquid by about one atmosphere will be sufficient, but this figure may be departed from rather widely with differences in actual pressure drop and in heat leakage into the intake line.

As nitrogen is the lowest-boiling component present in any appreciable quantity in air, and as no lower-boiling refrigerant is ordinarily available at the same absolute pressure, the cooling fluid must be expanded to a pressure below that under which the liquid passes to the pump. The necessary pressure difference may be produced by applying artificially created pressure to the liquid flowing through the cooling coil, or by evacuating the chamber in which the coolant is flashed, but in the instant disclosure it is produced in the simplest possible manner by withdrawing the liquid to be pumped from the point in the fractionating cycle at which it already exists at a pressure materially above atmospheric, and by expanding the coolant to substantially atmospheric pressure, that is, to the lowest pressure at which it will flow at the re-

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quired velocity through the interchangers in which its remaining refrigerative value is recovered.

The pressures existing in practice in the intermediate stage of the cycle of Fig. 1 or the high-pressure stage of the cycle of Fig. 2 are ample to permit the cooling of even pure liquid nitrogen to a temperature of stability in the pump section by interchange with even what is normally the highest boiling product of the fractionation.

The method herein described is useful and valuable for the subcooling and pumping of all liquefied gases and gaseous mixtures rich in nitrogen and having vapor pressures approaching those of nitrogen. Such gases, referred to in some of the claims as nitrogenous gases, include nitrogen in a state of more or less purity, atmospheric air, and oxygen-enriched air containing not less than about fifty per cent nitrogen by weight.

I claim as my invention:

1. The method of transferring liquefied product of a fractionating operation, in which operation a mixture of compressed and cooled component gases having boiling points substantially below atmospheric temperature is expanded and the effluent of the expansion step subjected to the fractionating operation to produce a liquefied higher boiling point fraction and a gaseous lower boiling point fraction at relatively low pressure, comprising forming as a step in the fractionating operation liquefied lower boiling point fraction at a pressure substantially equal to the vapor pressure of the liquefied lower boiling point fraction, withdrawing a stream of the liquefied lower boiling point fraction from the fractionating operation, subcooling the stream of liquefied lower boiling point fraction by heat exchange with a relatively colder fluid from the fractionating operation at a pressure lower than the pressure of the liquefied lower boiling point fraction, the last named pressure being such that the temperature of the liquefied lower boiling point fraction is reduced to a point below the boiling point of the liquefied lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the subcooled liquefied lower boiling point fraction in liquid phase to a relatively high pressure.

2. The method of transferring liquefied product of a fractionating operation, in which operation a mixture of compressed and cooled component gases having boiling points substantially below atmospheric temperature is expanded and the effluent of the expansion step subjected to the fractionating operation to produce a liquefied higher boiling point fraction and a gaseous lower boiling point fraction at relatively low pressure, comprising forming as a step in the fractionating operation liquefied lower boiling point fraction at a pressure substantially equal to the vapor pressure of the liquefied lower boiling point fraction, withdrawing a stream of the liquefied lower boiling point fraction from the fractionating operation, subcooling the stream of liquefied lower boiling point fraction by heat exchange with the gaseous lower boiling point fraction product from the fractionating operation to reduce the temperature of the liquefied lower boiling point fraction to a point below the boiling point of the liquefied lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the subcooled liquefied lower boiling point fraction in liquid phase to a relatively high pressure.

3. The method of transferring liquefied product of a fractionating operation, in which operation a mixture of compressed and cooled component gases having boiling points substantially below atmospheric temperature is expanded and the effluent of the expansion step subjected to the fractionating operation to produce a liquefied higher boiling point fraction and a gaseous lower boiling point fraction at relatively low pressure, comprising forming as a step in the fractionating operation liquefied lower boiling point fraction at a pressure substantially equal to the vapor pressure of the liquefied lower boiling point fraction, withdrawing a stream of the liquefied lower boiling point fraction from the fractionating operation, subcooling the stream of liquefied lower boiling point fraction by heat exchange with expanded liquefied lower boiling point fraction from the fractionating operation to reduce the temperature of the liquefied lower boiling point fraction to a point below the boiling point of the liquefied lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the sub-cooled liquefied lower boiling point fraction in liquid phase to a relatively high pressure.

4. The method of transferring liquefied nitrogen product of a fractionating operation, in which operation compressed and cooled air is expanded and the effluent of the expansion step subjected to the fractionating operation to produce liquefied oxygen higher boiling point fraction and the gaseous nitrogen lower boiling point fraction, comprising forming as a step in the fractionating operation liquefied nitrogen lower boiling point fraction at a pressure substantially equal to the vapor pressure of the liquefied nitrogen lower boiling point fraction, withdrawing a stream of the liquefied nitrogen lower boiling point fraction from the fractionating operation, subcooling the stream of liquefied nitrogen lower boiling point fraction by heat exchange with a stream of gaseous nitrogen at a pressure lower than the pressure of the liquefied nitrogen lower boiling point fraction, the last-named pressure being such that the temperature of the liquefied nitrogen lower boiling point fraction is reduced to a point below the boiling point of the liquefied nitrogen lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the sub-cooled liquefied nitrogen lower boiling point fraction in liquid phase to a relatively high pressure.

5. The method of transferring liquefied nitrogen product of a fractionating operation, in which operation compressed and cooled air is expanded and the effluent of the expansion step subjected to the fractionating operation to produce liquefied oxygen higher boiling point fraction and gaseous nitrogen lower boiling point fraction, comprising forming as a step in the fractionating operation liquefied nitrogen lower boiling point fraction at a pressure substantially equal to the vapor pres-

sure of the liquefied nitrogen lower boiling point fraction, withdrawing a stream of the liquefied nitrogen lower boiling point fraction from the fractionating operation, subcooling the withdrawn stream of liquefied nitrogen lower boiling point fraction by heat exchange with an expanded stream of liquefied nitrogen lower boiling point fraction at a pressure lower than the pressure of the withdrawn stream of liquefied nitrogen lower boiling point fraction, the last-named pressure being such that the temperature of the liquefied nitrogen lower boiling point fraction is reduced to a point below the boiling point of the liquefied nitrogen lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the sub-cooled liquefied nitrogen lower boiling point fraction in liquid phase to a relatively high pressure.

6. The method of transferring liquefied nitrogen product of a fractionating operation, in which operation compressed and cooled air is expanded and the effluent of the expansion step subjected to the fractionating operation to produce liquefied oxygen higher boiling point fraction and gaseous nitrogen lower boiling point fraction, comprising forming as a step in the fractionating operation liquefied nitrogen lower boiling point fraction at a pressure substantially equal to the vapor pressure of the liquefied nitrogen lower boiling point fraction, withdrawing a stream of the liquefied nitrogen lower boiling point fraction from the fractionating operation, expanding a stream of cold fluid from the fractionating operation to a pressure lower than the pressure of the liquefied nitrogen lower boiling point fraction, subcooling the stream of liquefied nitrogen lower boiling point fraction by heat exchange with the expanded stream of cold liquid from the fractionating operation, the pressure of the expanded stream being such that the temperature of the liquefied nitrogen lower boiling point fraction is reduced to a point below the boiling point of the liquefied nitrogen lower boiling point fraction at the minimum momentary pressure reached in an ensuing pumping step, and pumping the sub-cooled liquefied nitrogen lower boiling point fraction in liquid phase to a relatively high pressure.

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