LIQUID NITROGEN GENERATOR

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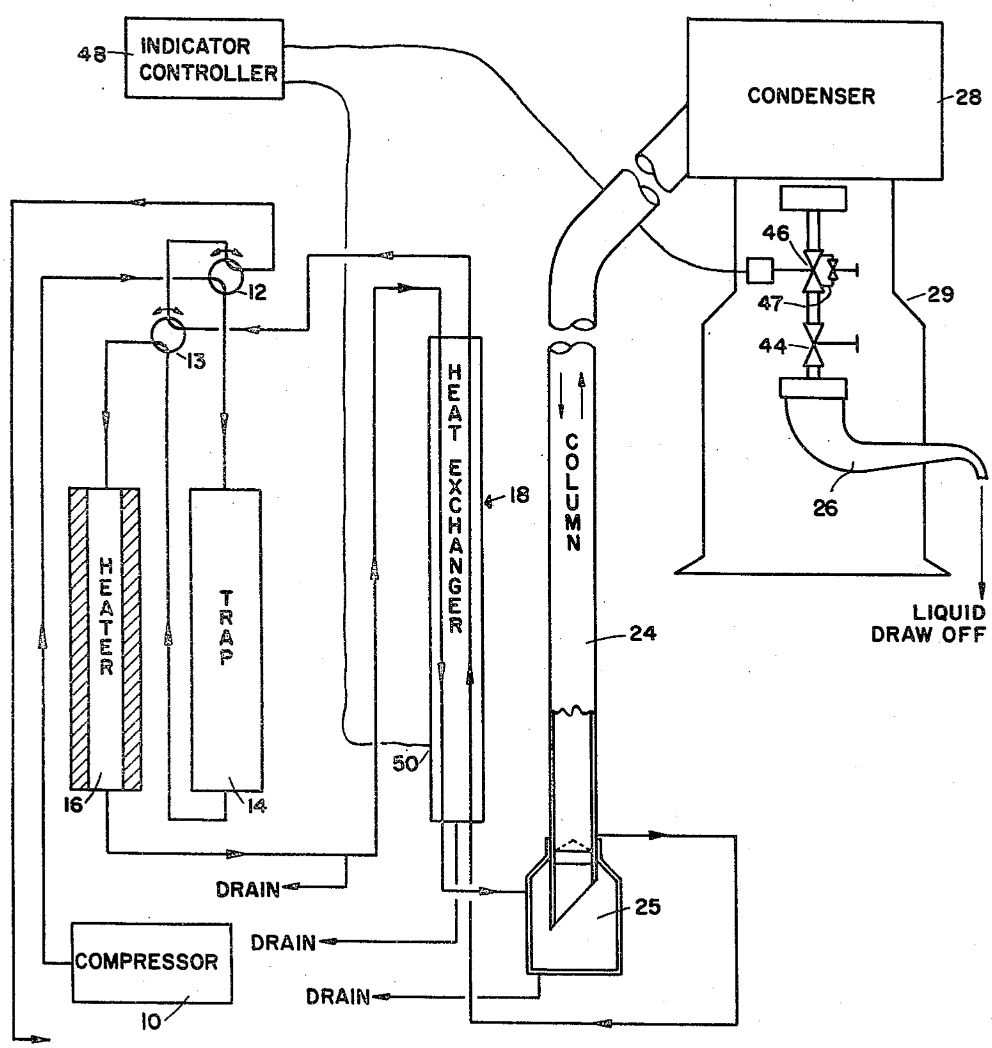

Fig. I

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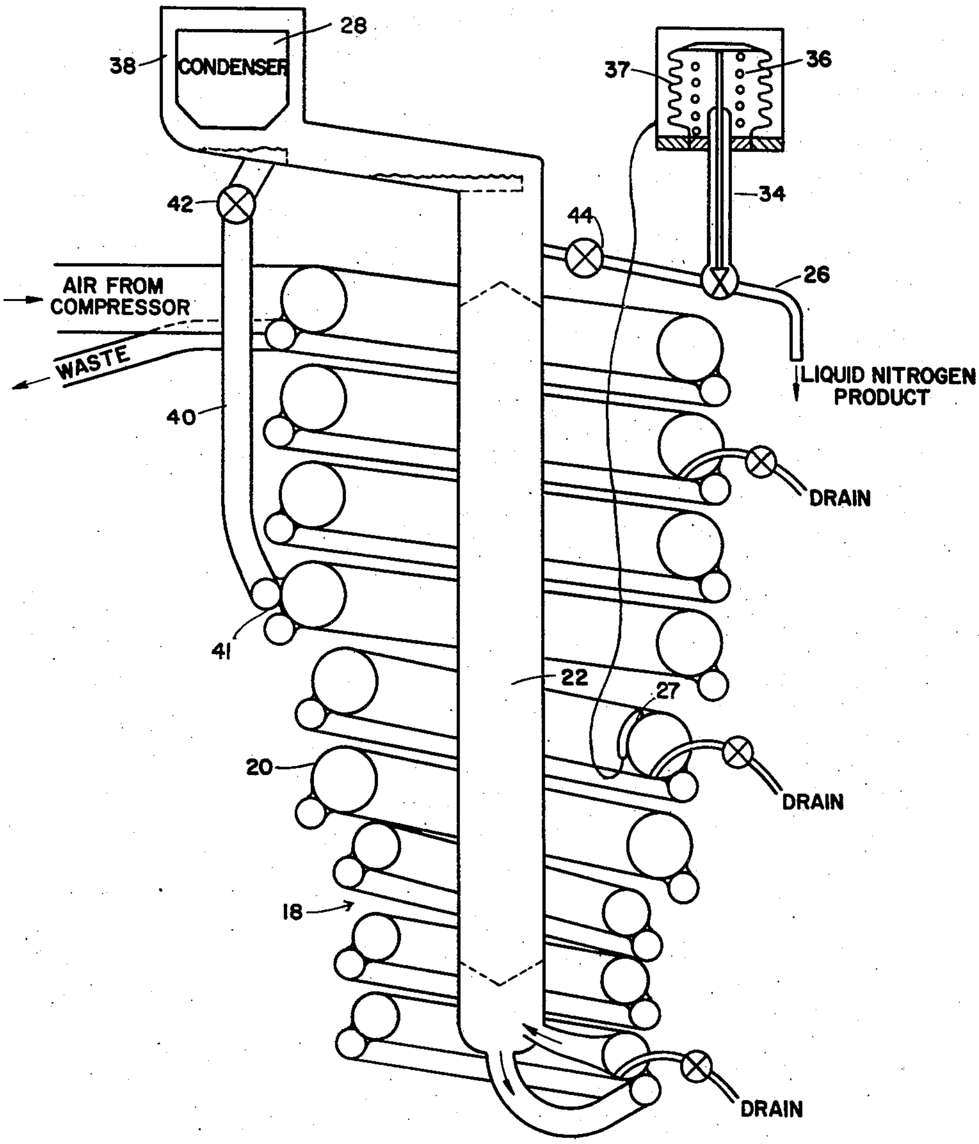

Fig. 2

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LIQUID NITROGEN GENERATOR

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11 Claims. (Cl. 62—13)

This invention relates to a liquid nitrogen generator and more particularly to a laboratory size liquid nitrogen plant employing a cold gas refrigerator as a source of refrigeration.

An object of the present invention is the provision of a system for producing liquid nitrogen having a counterflow heat exchanger for raw air entering in one direction and reflux liquid entering in an opposite direction.

Another object of the present invention is to provide means to prevent the column from being packed with water ice and carbon dioxide during the start up period of the plant.

A further object of the present invention is to provide means to effect a desired proportioning of the condensate from the condensing head of the cold gas refrigerator between product and reflux from the column.

Another object of the present invention is to provide a liquid nitrogen plant of great simplicity, compactness and ease of operation yet having a high production rate for the relatively small size of the device. In this regard, it should be apparent that the need for a simple, laboratory-size liquid nitrogen generator is ever increasing with the rapid strides made in research in the cryogenic field and low temperature physics.

A further object of the present invention is the provision of a novel heat exchanger which is traversed in one direction by a gas and in another direction by a liquid.

The above and other features, objects and advantages of the present invention will be fully understood from the following description considered in connection with the accompanying illustrative drawings.

Fig. 1 is a schematic diagram of the liquid nitrogen generator constructed in accordance with the present invention, and

Fig. 2 is a schematic diagram of an alternate construction with the part of the system omitted which is the same as that shown in Fig. 1.

In accordance with the invention it has been recognized that because of the nature of room air as a mixture that it is not practicable to remove as a liquid nitrogen product more than half of the air supplied to the plant. The remainder of the mixture, being gas enriched in oxygen, must be ejected into the atmosphere. This waste stream leaves the column at the temperature of liquid air. However, in order to conserve refrigeration the waste stream is warmed substantially to room temperature in a counterflow heat exchanger by absorbing heat from incoming raw air going in the opposite direction in the heat exchanger. The moisture and carbon dioxide present in the raw air will be deposited on the walls of the air conduit in the heat exchanger.

Referring to the drawings and more particularly to Fig. 1 a schematic diagram of the liquid nitrogen generator constructed in accordance with the present invention discloses a compressor 10 which compresses atmospheric air of pressures in the order to 2–3 p.s.i. and the compressed air is fed through valve 12 to preferably a chemical drier 14 which may, for example, be filled with silica gel. Thus, during the cooldown period of the nitrogen plant the moist air passes through the drier and moisture is removed chemically. The air mixture is then passed through a heater 16 and thereafter into the counterflow heat exchanger referred to generally by the numeral 18 and including an outside coil arrangement 20 for the inlet air and a central tube 22 for the reflux gas bonded to the outside coil so that the outside coil and central tube are in heat exchanging relationship. The outside coil may be provided with fins (not shown). The air mixture then leaves the heat exchanger 18 and is supplied to the column 24 which is in this instance a packed column having a liquid pot 25, but may take the form of a column with vertically superposed trays or extensions projecting from the wall of the column. The nitrogen component of the air mixture rises in column 24 and is drawn into the consensing head 28. Liquid nitrogen is removed from the tap conduit 26 of gas refrigerator 29. The cold-gas refrigerator contemplated as part of the present system is disclosed in detail in U.S. Patent No. 2,750,765 to J. W. L. Kohler et al. dated June 19, 1956.

As stated before because of the nature of air as a mixture, reflux is required in the distillation column for 99.5% nitrogen to be produced, therefore about half the feed stream is withdrawn as product and half is returned countercurrent to the feed in the column 24 and the heat exchanger 18. In the heat exchanger the reflux is turned into a gaseous state and is warmed substantially to room temperature in the counterflow heat exchanger 18 by absorbing heat from the incoming air. Thus, since the reflux or waste stream is the cooling means moisture and CO_2 in the air are frozen out on the walls of the inlet passage of the heat exchanger 18. Thereafter, the reflux is conducted through a valve 13, valve 12 and thence to the atmosphere.

During normal operation of the nitrogen plant moisture and CO_2 in the air are frozen out on the walls of the inlet passage of the exchanger, the cooling medium, as stated before, is the flux or waste stream. In order to insure stable and acceptable conditions in the heat exchanger it is necessary that this waste stream be returned through the heat exchanger 18. It is desirable to return approximately one-half of the entering air as liquid waste. If too little waste liquid enters the heat exchanger and too much liquid nitrogen product taken from the plant the compressed air mixture is introduced into the column at too high a temperature and will not be free of carbon dioxide. In Fig. 2 valve 34 responds to the aforesaid condition and partially closes thereby reducing the product rate. Valve 34 contains a condensable fluid such as Freon, CH_4, O_2 and is mounted on tap conduit 26 and is also connected to the outside coil arrangement 20 at a predetermined location 27. This location is chosen on the basis that the normal temperature of this area partially condenses the charge of gas in the valve 34. Thus, the fluid in valve 34 is very sensitive to changes in temperature and thereby causes pressure on the valve elements to close the same. A compression spring 36 and bellows 37 urges the valve 34 to its open position. The present liquid nitrogen generator represents a considerable mass of metal which must be cooled through various ranges of temperature in order to reach the operating level. This is accomplished by the liquefaction of air and the subsequent dispersion of the liquid over the surfaces to be cooled. There is no net flow of air through the plant during this period. It is not desirable, therefore, to turn on the compressor until the column is cold and charged with the proper amount of liquid air. Therefore, in normal operation of the present liquid nitrogen generator as seen in Fig. 2 the condensing head 28 is surrounded with a jacket **38** and has a conduit **40** which is connected to the outside coil arrangement **20** of the heat exchanger at a region **41** near the center thereof. Solenoid valve **42** in conduit **40** is held open upon starting up of the plant for approximately 30 minutes. During this interval of time all the liquid is re-evaporated as rapidly as it is formed by heat provided by the relatively warm metallic surfaces upon which it falls. The amount of air and its accompanying moisture which is drawn into the system thus far is negligibly small because there has been no accumulation of liquid air. In addition, the lower part of heat exchanger **18**, in the 30 minutes that valve **42** is open, is cooled to a temperature far below the freezing point of water. Thus, the incoming air is dehydrated. After the foregoing operation, valve **42** is closed and thereafter the compressor **10** commences to operate and solenoid valve **44** mounted on top conduit **26** opens. Valve **44** which is controlled by a timing device (not shown) is initially closed during the "warm up" period of the apparatus but opens after a predetermined time interval. Valve **44** remains wide open during normal operation. Partition of the liquid for product is accomplished by valve **34**.

An alternate to valve **34** liquid control is shown in Fig. 1 in which a solenoid valve **46** with a built-in bypass **47** is operated by a controller **48** which in turn is controlled by a thermocouple **50** located in the lower region of heat exchanger **18**. The bypass **47** allows some minimum liquid flow at all times. Any excess liquid is discharged by the solenoid valve **46** when the temperature in the heat exchanger becomes too low.

Another method of liquid control is a liquid level sensing device which may be located in liquid pot **25** of column **24** (not shown).

It should be noted that the net amount of air delivered to the column by the compressor **10** is exactly the amount called for by the condensing head **28**. Any excess which may be provided by the compressor enters the bottom of the column **24** but leaves immediately along with the waste liquid or reflux. This does not hinder the operation of the plant with the minor exception that it does leave additional ice in the heat exchanger and places an additional heat load upon the heat exchanger.

While we have shown and described the preferred embodiment of our invention, it will be understood that the latter may be embodied otherwise than as herein specifically illustrated or described and that in the illustrated embodiment certain changes in the details of construction and in the arrangement of parts may be made without departing from the underlying idea or principle of the invention within the scope of the appended claims.

What we claim:

1. A process for the manufacture of liquid nitrogen from an air mixture comprising supplying said air mixture to a counterflow heat exchanger at slightly superatmospheric or subatmospheric pressure and thence to a gas fractionating column, connecting the upper part of said column to a condenser of a cold-gas refrigerator whereby the liquid nitrogen product of said air mixture is formed, and returning approximately one-half of said air mixture as liquid reflux through said heat exchanger and then to the atmosphere, said liquid reflux being in heat exchanging contact with said air mixture being conducted in the opposite direction in the counterflow heat exchanger whereby moisture and CO_2 is frozen out.

2. A process for the manufacture of liquid nitrogen from an air mixture comprising compressing said air mixture to slightly above atmospheric pressure, supplying said air mixture to a counterflow heat exchanger and thence to a gas fractionating column, connecting the upper part of said column to a condenser of a refrigerator whereby the liquid nitrogen product of said air mixture is formed, and returning part of said air mixture as liquid reflux through said heat exchanger and then to the atmosphere, and controlling automatically the ratio of reflux being in heat exchanging contact with said air mixture in the counterflow heat exchanger whereby moisture and CO_2 is frozen out.

3. A process for the manufacture of liquid nitrogen from an air mixture comprising removing moisture from said mixture before the commencement of the liquid nitrogen manufacturing process compressing said air mixture to slightly above atmospheric pressure, supplying said air mixture to a counterflow heat exchanger and thence to a gas fractionating column, connecting the upper part of said column to a condenser of a cold-gas refrigerator whereby the liquid nitrogen product of said air mixture is formed, and returning approximately one-half of said air mixture as liquid reflux through said heat exchanger and then to the atmosphere, said liquid reflux being in heat exchanging contact with said air mixture being conducted in the opposite direction in the counterflow heat exchanger whereby moisture and CO_2 is frozen out.

4. An apparatus for the manufacture of liquid nitrogen comprising a compressor for creating a pressure slightly different from atmospheric, a counterflow heat exchanger, first means for conducting an atmospheric air mixture through said compressor to said heat exchanger, a gas fractionating column, a refrigerator provided with a condenser, second means for conducting said air mixture from said heat exchanger to said column, said column being operatively connected to said condenser whereby the liquid nitrogen product is formed, means to tap off said liquid nitrogen product so formed, and means to conduct at least half of said air mixture as a liquid reflux back through said heat exchanger and discharged into the atmosphere, said liquid reflux being in heat exchanging contact in said heat exchanger with said air mixture being conducted in the opposite direction and means for controlling automatically the ratio of reflux being in heat exchanging contact with said air mixture in the counterflow heat exchanger whereby moisture and CO_2 is frozen out.

5. An apparatus for the manufacture of liquid nitrogen comprising a device for removing moisture from said air mixture before the commencement of the liquid nitrogen manufacturing process, a compressor, a counterflow heat exchanger, first means for conducting an atmospheric air mixture through said compressor to said heat exchanger, a gas fractionating column, a refrigerator provided with a condenser, second means for conducting said air mixture from said heat exchanger to said column, said column being operatively connected to said condenser whereby the liquid nitrogen product is formed, means to tap off said liquid nitrogen product, and means for conducting at least half of said air mixture as a liquid reflux back through said heat exchanger and discharged into the atmosphere, said liquid reflux being in heat exchanging contact in said heat exchanger with said air mixture being conducted in the opposite direction and means for controlling automatically the ratio of reflux being in heat exchanging contact with said air mixture in the counterflow heat exchange whereby moisture and CO_2 is frozen out.

6. An apparatus for the manufacture of liquid nitrogen as claimed in claim 5 wherein said device is a receptacle containing a chemically absorbent material which removes the moisture from the air during start-up of the apparatus.

7. An apparatus for the manufacture of liquid nitrogen as claimed in claim 6 wherein said receptacle is a silica gel trap.

8. An apparatus for the manufacture of liquid nitrogen as claimed in claim 4 further comprising a valve containing a condensable fluid which is sensitive to changes in temperature, means operatively connecting said valve to a predetermined location on said heat exchanger whereby changes in temperature of said heat exchanger causes said valve to automatically open and

close thereby automatically adjusting the ratio of liquid nitrogen product and reflux in said apparatus.

9. An apparatus for the manufacture of liquid nitrogen as claimed in claim 4 further comprising a valve provided with a bypass, a thermocouple secured to a lower region of said heat exchanger and operatively connected to said valve, said thermocouple controlling the opening and closing of said valve thereby automatically adjusting the ratio of liquid nitrogen product and reflux in said apparatus.

10. An apparatus for the manufacture of liquid nitrogen from atmospheric air and dividing the liquid nitrogen produced between reflux and product comprising a compressor, a counterflow heat exchanger including an outside coil for said atmospheric air and a central tube positioned within said outside coil for said reflux, first means for conducting an atmospheric air mixture through said compressor to said heat exchanger, a gas fractionating column, a refrigerator provided with a condenser, second means for conducting said air mixture from said heat exchanger to said column, said column being operatively connected to said condenser whereby the liquid nitrogen product is formed, means to tap off said liquid nitrogen product, and means to conduct at least half of said air mixture as a liquid reflux back through said heat exchanger and discharged into the atmosphere, said liquid reflux being in heat exchanging contact in said heat exchanger with said air mixture being conducted in the opposite direction.

11. A process for the manufacture of liquid nitrogen from an air mixture comprising supplying said air mixture to a counterflow heat exchanger at slightly superatmospheric or subatmospheric pressure and thence to a gas fractionating column, connecting the upper part of said column to a condenser of a refrigerator whereby the liquid nitrogen product of said air mixture is formed, returning part of said air mixture as liquid reflux through said heat exchanger and then to the atmosphere, said liquid reflux being in heat exchanging contact with said air mixture being conducted in the opposite direction in the counterflow heat exchanger whereby moisture and CO_2 is frozen out, and automatically adjusting the ratio of liquid nitrogen product and reflux through heat-responsive means.

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