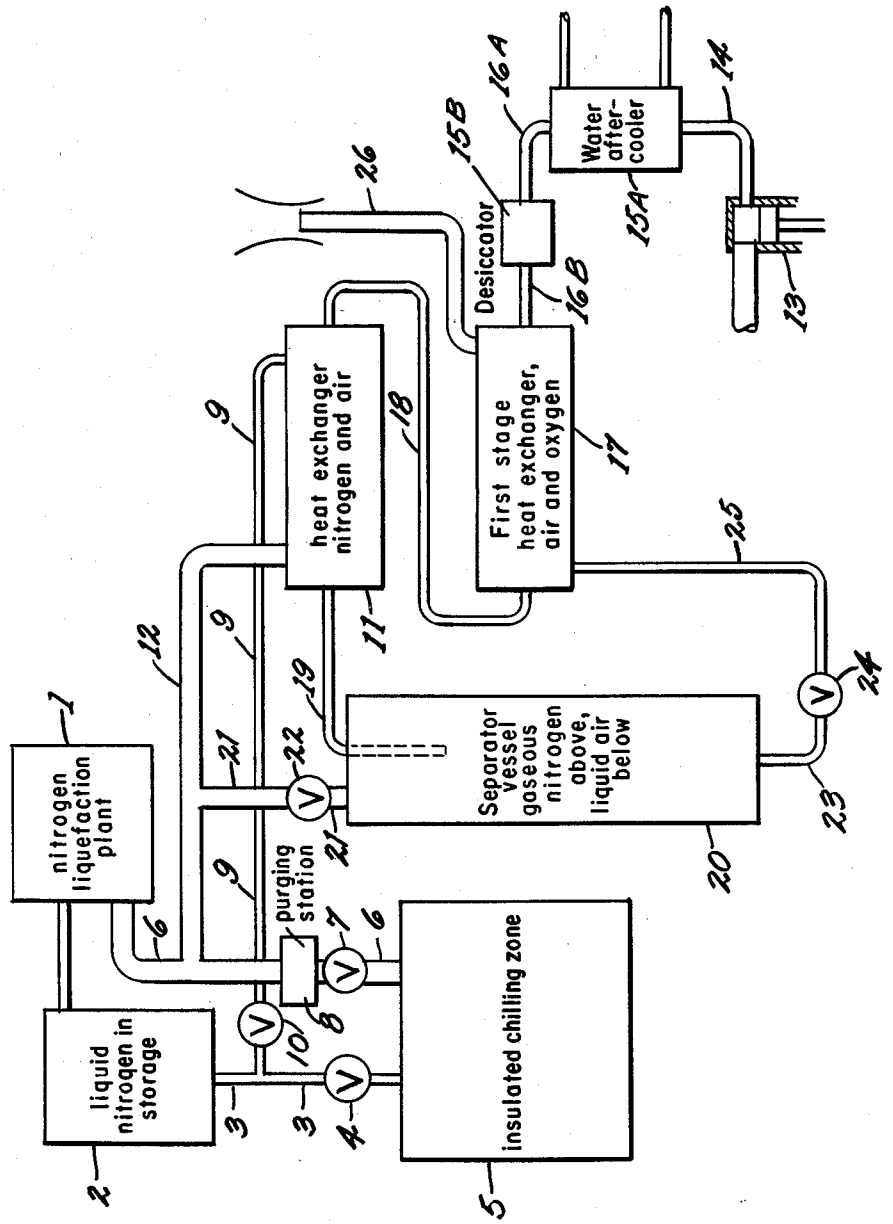
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METHOD OF AND APPARATUS FOR RECOVERING NITROGEN FROM AIR BY USE OF A COLD BOILING LIQUID SUCH AS NITROGEN

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METHOD OF AND APPARATUS FOR RECOVERING NITROGEN FROM AIR BY USE OF A COLD BOILING LIQUID SUCH AS NITROGEN

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My invention relates to method of and apparatus for recovering nitrogen from air by the use of a cold boiling liquid such as nitrogen. By cold boiling liquid I mean a liquid the boiling point of which at atmospheric pressure is far below zero degrees F.

In connection with the use of liquid nitrogen as a refrigerant brought directly into contact with the foodstuffs or other perishable material, or their direct containers, especially when a multiplicity of interchangeable containers must be presented to the nitrogen supply, chilled and then removed, there is a continuous depletion of the nitrogen in the system even though care is taken to insure that a maximum percentage of the gaseous nitrogen boiled off from the liquid by contact with the stuff being cooled be returned for reliquefaction and reuse.

It is therefore necessary to provide means for supplying make up nitrogen. Make up nitrogen in liquid form is an article of commerce and can be purchased but the expense is high and such refrigeration devices may work in places where liquid nitrogen make up is hard to come by. It has been suggested that such refrigerating plants be accompanied by a separate liquid air, liquid oxygen, liquid nitrogen recovery plant. This again is expensive and complicated.

This invention discloses a method and apparatus whereby with the simple addition of certain heat exchangers and a separating device and an air compressor with conventional after-cooler, the liquid nitrogen used as a refrigerant or rather some of that liquid nitrogen may be used to produce the necessary low temperatures to obtain make up nitrogen.

Other objects will appear from time to time in the course of the specification and claims.

My invention is illustrated in the accompanying flow sheet wherein like parts are indicated by like characters throughout the specification and drawing.

A nitrogen liquefaction plant **1** is adapted to liquefy gaseous nitrogen supplied to it and discharge the liquid nitrogen to storage at **2**, preferably though not necessarily at atmospheric pressure. A liquid nitrogen supply duct **3** controlled by the valve **4**, leads to a chilling zone **5**, which preferably takes the form of an insulated container adapted to hold the material to be chilled and adapted to be easily connected to and separated from the nitrogen system. The liquid nitrogen entering the chamber **5** is gasefied, the contents of the chamber furnishing the latent heat to change the nitrogen from liquid to gaseous state. This gas escapes through the duct **6** controlled by the valve **7** and is led to the reliquefying plant **1** through a purging station **8**. The valves **4** and **7** make it possible to connect and disconnect successive freezer containers without loss of nitrogen but since every time a freezer container is filled with nitrogen and taken away, some nitrogen is lost and since some nitrogen is lost in purging or purifying the nitrogen gas from the

freezer container before it reaches the reliquefier, make up nitrogen must be provided.

Liquid nitrogen at —320° F. may also be conducted through the pipe **9** controlled by the valve **10** to a heat exchanger **11** where as will hereafter appear by heat exchange the liquid nitrogen is vaporized and returned at —320° F. through the duct **12** to the reliquefier.

Atmospheric air is drawn in and compressed by an air compressor **13** which for example may be two-stage, with a small horsepower percentage of that required by 1. The air will be compressed to a pressure of one hundred pounds p.s.i.g. and discharged through the duct **14** to a water after-cooler **15***a* which will extract most of the heat of compression. Air may then travel through the duct **16***a* to air cleaning or desiccating equipment **15***b* at for example 125° F., then through duct **16***b* to a first stage heat exchanger **17** where by heat exchange this compressed air will be cooled to —227° F. by oxygen and will then as will hereafter appear be led through duct **18** to the heat exchanger **11** where by heat exchange with the liquid nitrogen, it will be cooled to —249° F. and will be discharged through the duct **19** to the separator vessel or tall column **20**. The pressure in the separator vessel is 100 pounds p.s.i.g. The temperature, —249° F. at 100 pounds p.s.i.g. is such that oxygen will liquefy but nitrogen will not, so in the vessel we have at the bottom oxygen enriched liquid and at the top gaseous nitrogen in a substantially pure state because nitrogen will not remain a liquid at —249° F. and 100 pounds p.s.i.g. In order to get satisfactory effective separation, the duct **19** extends down to a point well below the top of the separator vessel and gaseous nitrogen will be discharged therefrom through the duct **21** controlled by the pressure reduction valve **22** and led through ducts **12** and **6** to the reliquefier.

Oxygen enriched liquid from the bottom of the vessel **20** will be discharged through the duct **23**, Joule Thompson expansion valve **24** and duct **25** to the heat exchanger **17**. The liquid oxygen expanding from 100 pounds p.s.i.g. through the Joule Thompson valve will be in part liquid and in part gas at a temperature of approximately —297° F. and it is this cold oxygen both in liquid and gaseous form which by heat exchange lowers the temperature of the compressed air in the first heat exchanger. Oxygen from heat exchanger **17** will be vented to atmosphere through the duct **26** at 70° F. and atmospheric pressure, no effort being made to recover the oxygen because in this instance, we are interested only in getting make up nitrogen gas for the refrigeration system. The fact that the make up nitrogen is supplied to the system as a gas rather than a liquid is no disadvantage because the reliquefier will always have adequate capacity to take care of and liquefy gaseous make up nitrogen just as well as gaseous nitrogen from the refrigeration chamber.

The reliquefier will, of course, include some type of compressor, presumably a multi-stage compressor. The air compressor above referred to might be built in as a part of that compressor or it might be a separate one. By control of the valves **4**, **7**, **10** and **22**, liquid nitrogen may be proportioned between refrigeration and make up or make up may be provided when refrigeration is not going on or vice versa as of course there will always be in the storage zone a substantial amount of liquid nitrogen. The temperatures and pressures above referred to are, of course, illustrative. The entire cycle might take place at atmospheric pressure but taking place at 100 pounds p.s.i.g. gives a wider range of temperature to work with, thereby improving accuracy, efficiency and making safe and effective controls possible. The after-cooler taking out the heat of compression may, of course,

be water or air cooled or be cooled by any other means as desired.

While in a simple solution of the problem as above indicated no effort would be made to recover the oxygen, it would, of course, under some circumstances be desirable to recover the gaseous oxygen for other use and my invention is not limited to any particular temperatures or pressures or methods of heat exchange provided only that by the use of the cold liquid nitrogen the air is reduced to a temperature at which some nitrogen may be separated from the oxygen and returned to the refrigeration system as make up.

I claim:

1. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit.

2. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit, means for supplying as a coolant the liquid from which gaseous nitrogen has been extracted to the heat exchange means.

3. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit, means for supplying as a coolant the liquid from which gaseous nitrogen has been extracted to the heat exchange means, means for supplying as a coolant liquid nitrogen from the refrigeration circuit to the heat exchange means, means for returning nitrogen in gaseous phase from the heat exchange means to the refrigeration circuit.

4. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit, means for supplying as a coolant the liquid from which gaseous nitrogen has been extracted to the heat exchange means, means for supplying as a coolant liquid nitrogen from the refrigeration circuit to the heat exchange means, means for returning nitrogen in gaseous phase from the heat exchange means to the refrigeration circuit, the heat exchange means preventing any mixing of the compressed air with the coolant liquids therein.

5. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit, means for supplying as a coolant liquid nitrogen from the refrigeration circuit to the heat exchange means, means for returning nitrogen in gaseous phase from the heat exchange means to the refrigeration circuit.

6. In combination, a refrigeration circuit adapted to use nitrogen as a refrigerant and a separation circuit adapted to recover nitrogen in gaseous phase from atmospheric air and supply it to the refrigeration circuit, the refrigeration circuit including in series nitrogen liquefaction means, liquid nitrogen storage means and a refrigeration vessel, wherein liquid nitrogen at approximately atmospheric pressure contacts material to be refrigerated, is evaporated thereby and from which nitrogen in gaseous phase is returned without substantial loss of temperature to the liquefaction means, the separation circuit including connected means for compressing air, heat exchange means for cooling the compressed air by liquid coolants to a temperature between the boiling points of oxygen and nitrogen, means for supplying the resultant nitrogen in gaseous phase to the refrigeration circuit, means for supplying as a coolant liquid nitrogen from the refrigeration circuit to the heat exchange means, means for returning nitrogen in gaseous phase from the heat exchange means to the refrigeration circuit, the heat exchange means preventing any mixing of the compressed air with the coolant liquids therein.

References Cited in the file of this patent

UNITED STATES PATENTS

1,843,043	Patart	Jan. 26, 1932
2,423,273	Van Nuys	July 1, 1947
2,496,380	Crawford	Feb. 7, 1950
2,708,831	Wilkinson	May 24, 1955