

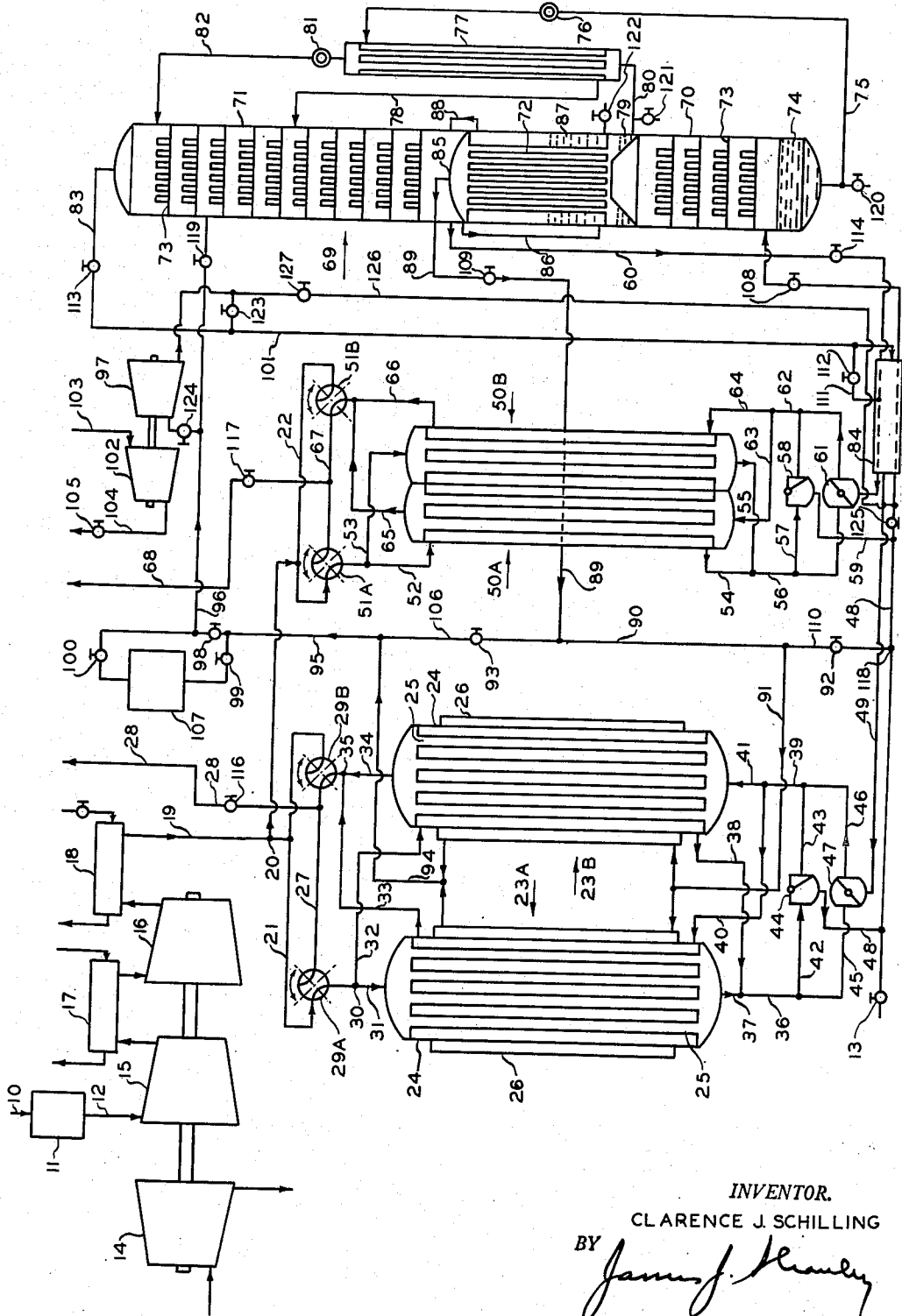
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METHOD FOR DEFROSTING GAS SEPARATION SYSTEMS

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METHOD FOR DEFROSTING GAS
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This invention relates to methods for defrosting gas separation systems. More particularly, it relates to a method for defrosting an air fractionating system using the main heat exchangers to dry the warming-up air.

It is essential during the defrosting operation that moisture be excluded from the column. Previously, it has been common to clean the moisture out of the warming-up air during defrosting of the column by means of chemical cleaners, such as silica gel and the like.

This invention has as an object to provide an improved method for defrosting an air fractionation system which is commercially more feasible than the previously used methods.

A further object of the invention is to reduce the required capacity of the chemical cleaners for the air during defrosting.

A still further object of the invention is to reduce the time period required for defrosting an entire air fractionating system.

These and other objects are accomplished by the present invention wherein an air fractionation system is defrosted by shutting down the expanders so that no refrigeration is added to the system, while fresh air taken into the system is cleaned of moisture in the heat exchangers.

In the older practice of this art, the refrigerative value of the cold fractionation products was recovered in interchangers, usually tubular, in which streams of warm air and cold fractionation products flowed continuously in heat exchange relation in separate passages. This practice required that both the carbon dioxide and the water content of the air supply be reduced as far as possible by chemical treatment and adsorption prior to the interchange, as otherwise the cold ends of the interchangers rapidly choked with ice and carbon dioxide snow.

The necessity for preliminary chemical treatment of the air may be avoided in the use of heat interchangers of the flow-reversing type. Such interchangers are known in two functionally similar forms:

The first form is the so-called "cold accumulators" which consist of shells packed with materials, such as extended surfaces of sheet metal which readily absorb and surrender heat. These accumulators are used by passing the cold fractionation products from the column through the shells and the packing until the temperature of the accumulators is reduced to the desired low temperature. The flow of these cold gases is then diverted to a second accumulator, while the warm air is passed through the cold shell and

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emerges at a low temperature. Continuity of supply is maintained by the use of duplicate accumulators, air flowing through one while the cold gas is reducing the temperature of the other.

In contradistinction to the use of "cold accumulators," a countercurrent reversing cold interchanger system may be used which permits a simultaneous heat interchange between passageways containing countercurrently flowing streams of air and backward returning cold products. This type interchanger comprises a plurality of parallel paths for the fluid in each passageway so as to establish thermal contact throughout the whole contact length of the vessel. These reversing cold interchangers are utilized to remove almost all of the higher boiling impurities from air or other gaseous mixtures, such removal being accomplished by periodically alternating the flow of warm incoming feed and a backward returning cold product between at least two passageways of the interchanger. During one-half of the reversing cycle when the air is being cooled, water and carbon dioxide, for example, are precipitated therefrom and accumulated in solid or liquid phase on the metal surfaces of the passageway through which the air at that time is flowing. Before the accumulation has become great enough to plug that passageway, the countercurrently flowing streams are interchanged to enable the backward returning product to flow over the accumulated deposits and reevaporate them. Meanwhile, the air is being cooled and precipitating further quantities of impurities on the metal surfaces of the alternate passageway through which the backward returning product previously has been flowing.

As the vapor pressures of water and carbon dioxide are extremely low at the temperatures at which the product gases emerge from the air fractionating column, these flow-reversing interchangers may readily be so operated as to refrigerate atmospheric air, without preliminary purification or dessication, and deliver the refrigerated air in condition suitable for fractionation in a column. This is possible because, unlike the continuous flow interchanger, these forms permit the solids deposited in cooling the air to be, at least in large part, removed by sublimation when the flows are reversed and the backward returning low pressure gas flows through the passage in which the solids have been deposited.

It has been recognized that this removal by sublimation is not always nor necessarily complete and that there is usually a slow accumu-

lation of deposited solids which ultimately requires that the interchanger be shut down for deriming by warming. Various methods for increasing the effectiveness of the sublimation step and thereby lengthening the period during which the interchanger may be used without warming up have been proposed. Among these are the methods proposed in my copending applications Serial No. 755,286, filed June 18, 1947, "Air Fractionating Cycle and Apparatus," now Patent No. 2,626,510, issued January 27, 1953, and Serial No. 755,287, filed June 18, 1947, "Method of Defrosting Flow-Reversing Heat Interchangers," now abandoned.

It may be found necessary, however, after many hours of operation, to defrost the heat interchangers. This is a relatively simple operation, since the flow pipes to and from the fractionation column may be closed off by shutting valves, and the warm air blown into the interchangers readily defrosts the passageways and passes out through valve 13.

After defrosting the interchangers, the normal cycle of operation may be resumed, wherein the impurities in the air may again be removed by the operation of the heat interchangers.

A defrosting of the complete system, including the fractionating column as well as the heat interchangers, is necessary when the gradual accumulation of congealed deposits eventually clogs the circuits. This operation may be necessary only a few times a year, but while it is conducted, it is necessary to discontinue production for a comparatively long period of time. The present invention enables the operator to speed up the defrost and results in an economical manner of operation. The term defrosting as used in this specification and claims is meant to define the gradual warming up of the system so that the congealed solids which have accumulated in the apparatus are melted. The warming up of the system must be gradual so that the temperature differential in the system is not too great. If one point in the system were warmed too rapidly, due to the uneven expansion resulting at that point, great damage could be done to the apparatus.

The means for accomplishing this result will be described with reference to the attached drawing, which will be understood to be a flow sheet in which apparatus elements are indicated by symbols.

Referring to the drawing, air enters the system at 10 and is substantially freed from dust in an air cleaner 11. This element may be an electrostatic precipitator, a scrubber or a simple air filter. It is not essential to remove the dust completely, but only the coarser particles which might cause abrasion in the compression unit.

The cleaned air passes at 12 to a compression unit consisting of a steam turbine or other power source 14, a first and a second stage turbo-compressor 15 and 16, a water-cooled intercooler 17 and an aftercooler 18.

The compressed air leaves the aftercooler via conduit 19 at about 100 pounds absolute and at a temperature of about 300° Kelvin. This conduit is branched at 20, about 80% of the air supply passing to a header 21 and thence to the nitrogen interchangers and about 20% to header 22 and thence to the oxygen interchangers.

The system includes a pair of "switching" or deriming heat interchangers 23A—23B which are used in parallel for cooling the larger portion of the air supply by heat interchange against the cold gaseous nitrogen produced by the column.

Each of these units consists generally of a shell 24, a gaseous nitrogen passage consisting of a plurality of tubes 25 and an auxiliary gas passage illustrated as an external gas jacket 26.

The upper ends of the interchangers communicate with the air inlet manifold 21 and with a nitrogen outlet manifold 27, which in turn is vented from the system through a nitrogen vent pipe 28, through a pair of reversing valves 29A—29B. These valves are reciprocated through quarter turns, in synchronism and at suitable intervals, by means not shown. With these valves in the position shown in the figure, the right end of air manifold 21 is closed and air passes from the left end of the manifold through valve 29A to a conduit which is branched at 30 to deliver air through conduit 31 to the tubes of interchanger 23A and through conduit 32 to the shell of interchanger 23B. In this position, the left end of nitrogen manifold 27 is closed and the right end is in communication with the shell of interchanger 23A through conduit 33 and with the tubes of interchanger 23B through conduit 34, these conduits connecting at 35. When the valves are simultaneously reversed in position, as by a quarter turn clockwise, the functions of the described conduits are reversed, conduits 31 and 32 carrying vent nitrogen and conduits 33 and 34 carrying entering air.

The coupling of the interchangers in such manner that each divided stream flows always through the tubes of one interchanger and the shell of the other is important in avoiding variations in resistance to flow of low pressure gaseous nitrogen which often accompany valve reversals when a pair of interchangers are so connected that the flow of nitrogen is directed first through two sets of tubes and then through two shells.

The lower ends of the interchangers are coupled in a similar manner to manifolds which alternately convey air and nitrogen. Thus, manifold 36 is branched at 37 to the tubes of interchanger 23A and at 38 to the shell of interchanger 23B. Manifold 39 is oppositely branched, i. e., at 40 to the shell of interchanger 23A and at 41 to the tubes of interchanger 23B. These manifolds are also branched at 42 and 43 to opposite sides of a flap valve 44, and at 45 and 46 to opposite sides of a flap valve 47.

With the reversing valves 29A—29B in the positions shown, manifold 36 is conveying air under relatively high pressure while manifold 39 is conveying nitrogen at a much lower pressure. The overbalancing pressure in branch 42 swings the flap of valve 44 to the right, as illustrated, preventing the air from entering the opposite manifold through branch 43 and directing it into conduit 48 which leads to the fractionating column. The flap in valve 47 being pivoted below its center line, the excess pressure in branch 45 tips it to the right, as illustrated, affording a passage for nitrogen from conduit 49, leading from the fractionating column, into branch 46 and manifold 39 which passes gas upwardly through the interchangers.

The oxygen interchangers 50A—50B are structurally identical with the nitrogen interchangers above described except for the omission of the jackets 26. Air from the compression unit passes from manifold 22 through reversing valve 51A and branch conduits 52 and 53 to the shell of interchanger 50A and to the tubes of interchanger 50B and through bottom connections 54 and 55, manifold 56, branch 57, flap valve 58 and conduit 59 to air conduit 48 leading to the column. Oxy-

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gen in gaseous form, withdrawn from the fractionating column through conduit 60, flows through flap valve 61, manifold 62 and branches 63 and 64 to the tubes of interchanger 50A and the shell of interchanger 50B, escaping at the upper ends of the exchangers through conduits 65 and 66 to valve 51B, manifold 67 and product oxygen delivery pipe 68.

Instead of using two separate sets of heat interchangers—one for the nitrogen product and a second for the oxygen product—a single set of heat interchangers containing a plurality of passageways may be used. In this manner of operation, the oxygen product, nitrogen product and the unbalancing medium all pass in heat exchange with the air feed in a single interchanger. For convenience, one particular system is shown and described but the invention could be applied to any low temperature gas separation system.

The fractionating column generally indicated at 69 may be any conventional or preferred two-stage column. In any case, it consists of a high pressure section 70 and a low pressure section 71 separated by a partition plate and a refluxing nitrogen condenser 72. Each of the sections is provided with bubble plates 73.

Liquid crude oxygen collecting in a pool 74 in the base of the high pressure section passes through a conduit 75 and an expansion valve 76 to an interchanger 77 in which it is in counter-flow heat interchange with high pressure liquid nitrogen, the expanded crude oxygen then passing through conduit 78 to an intermediate point in the low pressure section. A crude oxygen defrost outlet valve 120 is also provided in the base of the high pressure section.

The high pressure liquid nitrogen collecting in pool 79 below the nitrogen condenser passes through conduit 80 to the opposite side of interchanger 77 in which it is cooled and stabilized by the expanded crude oxygen, flowing thence through expansion valve 81 and conduit 82 to the upper end of the low pressure section. A high pressure liquid nitrogen defrost outlet valve 121 is also provided leading from pool 79.

Gaseous low pressure nitrogen is withdrawn from the top of the column through conduit 83, flowing to an exchanger 84 which is also connected to air feed line 48 and oxygen product line 60. The nitrogen product leaves exchanger 84 through line 49 above referred to as leading to the nitrogen interchangers.

Oxygen in a desired state of purity, ordinarily 95% or over, collects over the head 85 of condenser 72 and flows through conduit 86 to form a pool 87 surrounding tubes 72.

The high pressure nitrogen vapor within the tubes of the condenser are condensed by the liquid oxygen boiling around the tubes.

The oxygen vapor travels through bypass 88 to the vapor space above head 85, from which it is withdrawn through conduit 60 and the air-nitrogen interchanger 84 to the oxygen interchangers as above described. A pure oxygen defrost outlet valve 122 is also provided leading from the pool 87.

The interchangers are operated in the customary manner, the warm air passing through one side of each unit in counterflow to one of the cold product gases, until the air passages become fouled by the accumulation of water ice and solid carbon dioxide to give rise to a high pressure differential, or to fall below a predetermined heat transfer efficiency. At this point, the reversal of the valves 29A, 29B, 51A and 51B causes the air

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stream to flow through the passage previously occupied by the cold gas while the cold gas stream flows through the passage previously occupied by air, vaporizing and removing the ice and carbon dioxide snow.

It is a well known drawback to this procedure that the total products of fractionation flowing through a cold accumulator or its functionally equivalent deriming interchanger do not completely and dependably remove the accumulation of carbon dioxide snow and water ice from the surfaces on which they are deposited, and that such substantially complete removal may be effected by passing through the interchanger a quantity of cold gas materially greater than the quantity of air from which these deposits are accumulated.

To provide complete deriming for long period operation, it is essential that the cold end temperature difference between incoming air and purging product be 5° C. or less. To accomplish this, it is necessary to compensate for the higher specific heat of air under pressure especially at lower temperature. Adding quantity to the effluent product makes this possible by bringing the temperature-enthalpy curves of the counterflowing gases into approximate parallelism.

It is not necessary that the excess cold gas be in contact with the deposited solids, but only that it be in heat interchange relation with them. In consequence there are numerous ways in which this compensation may be effected, in any interchanger, direct contact or passageway type, in which the gas to be cooled and the gas to be heated flow alternately through the same passage.

In the operating cycle here described, the oxygen interchangers 50A—50B are provided with an excess of the cold gas by passing through them a smaller quantity of air than that which corresponds to the quantity of oxygen produced, for example, say 20% of the total air supply instead of the 21% to 22% which would correspond with the oxygen yield.

The remainder or say 80% of the air supply passes through the nitrogen interchangers 23A—23B and the excess of cold gas is provided by nitrogen withdrawn in gaseous form from the high pressure section of the column, heated by passing through the nitrogen interchanger, cooled by expansion and returned at low pressure to pass again through the interchanger with the low pressure nitrogen taken from the top of the column, thus passing twice through the step of interchange.

In more detail, a sufficient quantity of gaseous nitrogen, which may for example be perhaps 20% of the total nitrogen content of the air fractionated, is withdrawn from the dome 85 of the column condenser 72, carrying with it any incondensable gases which might otherwise tend to accumulate there. The withdrawn gas, at about 100 pounds absolute and about 100° K., passes through conduits 89, 90 and 91 and is equally divided between the auxiliary gas passages 26 of the nitrogen interchangers, in which its temperature is raised to about 145° K. by interchange with entering warm air. These streams, which flow continuously through the two interchangers in parallel and constantly from the cold to the warm end, are collected in conduit 94 and pass through conduits 95 and 96 to a turbo-expander 97. During normal operation, valve 98 in conduit 95 is open and valves 99, 100 and 119 are closed.

In the expander 97, the pressure is reduced to

about 24 pounds absolute in doing work and the temperature is thus reduced to about 100° K. The expanded nitrogen stream then passes through valve 123 to conduit 101 to mix with the colder nitrogen stream passing through conduit 83, the temperature of the mixed nitrogen stream at the cold end of the interchangers being thus raised to about 96° K.

The withdrawal of as much as 20% of the total nitrogen made in this manner does not reduce the quantity of reflux liquid sufficiently to interfere with efficient operation of the low pressure column section, so long as oxygen of the highest purity is not required.

The expander 97 is coupled with a turbo-compressor 102 or other means for applying a power load. The compressor is desirable as providing a steady and readily controllable load. It is illustrated as taking air through conduit 103 and discharging it through a conduit 104 controlled by a valve 105. If the oxygen produced by the column is to be delivered into a pipe line at a pressure above that available at the interchanger outlet, compressor 102 may be utilized for that purpose.

It is desirable to provide a crossover line 106 to admit a controlled quantity of cold nitrogen into conduit 95 in case the temperature of the high pressure nitrogen passing from the jackets to the expander becomes too high. This quantity is controlled by regulation of valve 93.

When using a single set of multi-passageway heat interchangers, all of the entering air is passed therethrough in heat interchange with the oxygen product, the nitrogen product and the high pressure nitrogen removed from the column through conduit 89.

It should be noted that the drawing shows only one turbo-expander 97. This unit, expanding the withdrawn high pressure nitrogen, suffices to provide make-up refrigeration for the cycle but when of proper size for that purpose is insufficient to provide refrigeration for starting up a warm apparatus. For this purpose, it is desirable to provide the expander in duplicate or even in triplicate to ensure quick starts after a shut-down.

In the drawing, 107 indicates any conventional air drier, such unit consisting ordinarily of two shells filled with silica gel or activated alumina, through which the gas to be dried is passed alternately and from which the adsorbed water is driven out by a stream of heated air or other gas. As these drying units are in common use and well known, they are not illustrated but are indicated by a symbol.

The normal method of defrosting this type system is as follows:

The heat exchangers are first warmed up. This is accomplished by shutting off the water to the aftercooler 18 so as to permit the temperature of the discharged air to attain relatively high values. This hot air is introduced in the normal manner through the switching valves to the air-to-nitrogen and air-to-oxygen exchangers and passed out through open valve 13. As soon as the heat exchangers have been warmed up to defrosting temperature, the defrosting of the remainder of the system is accomplished. The liquids are drained from the column through the drain valves 120, 121 and 122 which are opened fully. Valves 114 and 117 in the oxygen outlet lines and valve 116 in the nitrogen outlet line are opened. The crude oxygen and crude nitrogen expansion valves 76 and 81 are opened. Valve 109 in the air line to the column is closed. By opening valves 92, 93, 99, 100 and 119, and closing valve 92, the

flow of the warming up air is diverted through the air dryer 107 and thence into the column at the low pressure section. Thus, during the entire warming up of the column, chemical drying of the warming up gas is utilized. The air flows through the low pressure section of the column to the nitrogen and oxygen outlet lines 83 and 60 leading to the heat exchangers. The air also flows from the low pressure section to the high pressure section through lines 82 and 78 which normally carry the crude products to the low pressure column. The air is bled from the high pressure section through the drainage valves. When the proper temperature is observed at all outlets from the system, the defrosting operation is complete and the system is again started up into normal operation. It will be observed that in this method of operation, during the defrosting of the column, all the moisture is removed from the warming up air by use of the air dryer 107. This method of operation is costly and requires a large capacity of air dryers.

In the method of defrosting according to the present invention, the heat exchangers and the column warm up simultaneously in a gradual manner, the speed of which may be closely controlled. Uneven expansion of the apparatus due to warming one point too rapidly is prevented. In general, according to this invention, the operation of the system during defrosting is as follows: Expander 97 is shut down so that no refrigeration is added to the system. With all other operations being continued as though oxygen product is being produced, the liquids are drained from the column through crude oxygen drain valve 120, crude nitrogen drain valve 121, and pure oxygen drain valve 122. Air is continually fed to the system. A portion of the air being fed is continuously bled off at the crude oxygen defrost outlet valve 120 and at the pure oxygen defrost outlet valve 122. The proportion bled off at these points will determine the speed of the warm-up of the column and may be controlled. During this phase of the warm-up, the compressed air taken into the system is cooled in the heat interchangers as in normal operation of the system. The warm air enters at 10, is compressed and passes through the manifolds 21 and 22 to the heat interchangers 23 and 50, respectively. The moisture is removed from the air during its passage through the interchanges. The dry cold air in line 48 is passed through exchanger 84 through valve 108 into the high pressure section of the column. A portion of the air is bled from the high pressure section through valve 120. The remainder passes through conduit 75, expansion valve 76, heat interchanger 77, and conduit 78 into the low pressure section of the column. Air also leaves the high pressure section through line 80, interchanger 77, expansion valve 81, and conduit 82 into the low pressure section. Thus, it will be seen that the control of the pressure drop through the column may be manipulated by the proper setting of valves 108, 76, 81, 109, 113 and 114. A portion of the cold air leaves the low pressure section of the column through conduit 83, valve 113, line 101, exchanger 84 and thence to the heat interchangers 23, leaving the system through pipe 28. A further portion of the cold air leaves the low pressure section through conduit 60, exchanger 84, and thence to the heat interchangers 50, leaving the system through pipe 68. Air is also bled from the low pressure section through valve 122. The continuous deriming of the heat interchangers 23 is accomplished by removing a stream

of air from the high pressure section through dome 85 of the condenser 72, conduits 89, throttling valve 109, conduits 90, 91 and passage through the jackets 26 of the heat interchangers 23. This air leaves the heat interchangers through conduits 94, 95, 96, valve 124 and through the now idle expander 97, valve 123 and discharged into conduit 101 to merge with the air leaving the column through conduit 83. The expander 97 is defrosted by the passage of this air through it.

If the heat interchangers warm up more quickly than the column is being defrosted so that their cold ends approach about minus 40° C., then additional cold may be supplied to the interchangers without deleteriously affecting the defrosting of the column. This may be accomplished in one of two ways. With valve 108 fully open, and expansion valves 76 and 81 maintaining the pressure differential between the two sections of the column, valve 109 may be fully opened. In this manner, air under pressure from the high pressure section of the column is removed from the column through conduit 89 and is conveyed to the expander 97, utilizing conduits 106, valve 93, conduits 95, valve 98, conduit 96 and valve 124. The pressure of the air is reduced in passing through the expander and the stream of air is thus cooled. The cooled gas is recycled to the interchangers after it is merged with the air flowing from the column in line 101.

The second method of adding cold is to bypass a portion of the feed air flowing from the interchangers to the column through line 48 so that a stream of compressed air flows through lines 110, 90, 106, valve 93, conduit 95, valve 98, conduit 96 and valve 124 to the expander 97. From the expander, the expanded cooled air is merged with the air flowing in line 101 and is returned to the interchanger. In this method of operation, valve 109 is closed and, by relative manipulation of valves 76, 81 and 108, the pressure drop is controlled so that pressure is maintained in line 48.

When the column has reached a temperature, for example, minus 40° C., which is so high that the clean-up of the air in the main interchangers is no longer practical, then valves 125, 123 and 98 are closed and valves 92, 93, 99, 100 and 124 are opened. In this manner of operation, all of the air from the heat interchangers is diverted through conduits 110, 90, 106, 95, through valve 99, air dryer 107, valve 100 and thence to conduit 96. The stream of dry warm air may be divided and a portion admitted to the low pressure section of the column through valve 119. The remainder of the air stream passes through valve 124, idle expander 97, conduit 126 controlled by valve 127, and thence through line 48 and valve 108 into the high pressure section of the column.

During the final step of defrosting when the air dryers 107 are being utilized to dry the warming up gas, the compressors must be slowed down, sometimes to one-half capacity, and pressure must be maintained on the air passing through the dryers by throttling valves 119 and 124. When the entire system is completely defrosted, the plant may again be started up.

In operating in this manner, a great saving may be realized as the cold in the system that has previously been wasted during defrosting may be utilized for drying the warming-up air as long as possible. This results in a much speedier operation, the air being blown through the system at full capacity of the compressors. The process is also more economical in that the size of the

chemical drying installation may be greatly reduced.

I claim:

1. In the method of defrosting a low temperature gas separation system by flowing a warming-up gas therethrough, the step of removing from the warming up gas an impurity that is solid at temperatures above the normal operating temperature of the system by cooling the warming-up gas entering the system to a temperature below the freezing point of the impurity but above the normal operating temperature of the system by heat exchange with the gas leaving the system that has been cooled in defrosting the gas separation system.

2. The method of defrosting a low temperature gas separation system by flowing a warming-up gas therethrough comprising the steps of removing from the warming-up gas an impurity that is solid at the normal operating temperature of the system by cooling the warming-up gas entering the system to a temperature below the freezing point of the impurity but above the normal operation temperature of the system by heat exchange with the gas leaving the system that has been cooled in passing through the gas separation system, and bleeding off part of the cooled warming-up gas at at least one point in the system to remove cold from the system.

3. The method of defrosting an air separation system by flowing a warming up air stream therethrough comprising the step of removing moisture from the incoming compressed warming-up air stream by cooling the warming up air stream entering the system in a reversing heat exchange zone to a temperature below the freezing point of the moisture but above the normal operating temperature of the system by heat exchange with the air stream leaving the system that has been cooled by the defrosting process while passing through the separation system.

4. The method of defrosting an air separation system by flowing a warming-up gas therethrough comprising the steps of removing the moisture from the compressed warming up air by cooling the warm air entering the system in a reversing heat exchange zone to a temperature below the freezing point of the moisture by heat exchange with the outgoing air that has been cooled in passing through the system, and bleeding off part of the cooled warming-up air at at least one point in the separation system.

5. The method of defrosting a low temperature gas separation system by flowing a warming-up gas therethrough comprising the steps of removing from the compressed warming-up gas an impurity that is solid at the normal temperature of the system by cooling the warming-up gas entering the system in a reversing heat exchange zone to a temperature below the freezing point of the impurity but above the normal operating temperature of the system by heat exchange with the gas leaving the system that has been cooled in passing through the separation system, and preventing the temperature of the gas leaving the system from rising to said freezing point by passing a portion of the compressed gas through an expansion step in which work is done and combining the expanded gas with the remainder of the gas going to the heat exchange step, the temperature of the expanded gas being such that the combined gases are at a temperature below the freezing point of the impurity.

6. The method of defrosting a low temperature gas separation system by flowing a warming-

up gas therethrough comprising the steps of removing from the compressed warming-up gas an impurity that is solid at the normal operating temperature of the system by cooling the warming-up gas entering the system to a temperature below the freezing point of the impurity but above the normal operating temperature of the system by heat exchange with the gas leaving the system that has been cooled in passing through the system, bleeding off part of the cooled warming-up gas at at least one point in the system to control the rate of warm-up, and preventing the temperature of the gas leaving the system from rising to said freezing point by passing a portion of the compressed gas through an expansion step in which work is done and combining the expanded gas with the remainder of the gas going to the heat exchange step, the temperature of the expanded gas being such that the combined gases are at a temperature below the freezing point of the impurity.

7. The method of defrosting a low temperature gas separation system by flowing a warming-up gas therethrough comprising the steps of compressing a stream of warming-up gas, passing the compressed stream through the system, expanding the compressed stream in the course of its passage through the system, bleeding off a portion of the low pressure gas which has been cooled in the system, and removing an impurity from the compressed stream before the expansion step that is solid at the normal operating temperature of the system by cooling the compressed stream in a reversing heat exchange zone to a temperature below the freezing point of the impurity therein but above the normal operating temperature of the system by heat exchange with the remainder of the low pressure gas which has been cooled in passing through the system.

8. In the method of defrosting a low temperature fractionation system wherein a compressed gaseous stream of warming-up gas is passed in one direction of flow through a reversing heat

exchange zone along a precooled path therein progressively decreasing in temperature from end to end to effect cooling of the stream and resultant precipitation of an impurity that freezes in a colder portion of said path and wherein a second gaseous stream of outgoing warming up gas with a lower concentration of the impurity and under lower pressure and at lower temperature than said colder portion is passed subsequently through the same path in the opposite direction of flow after the first stream has ceased flow, the step of controlling the temperature of said colder portion of said path by passing a portion of the cooled compressed warming-up gas of a composition essentially the same as that leaving the colder end of the reversing heat exchange zone through a separate path in said heat exchange zone disposed in heat exchange relation with at least a part of the colder portion of the first mentioned path, combining this portion with the second stream, and passing the combined stream through the exchange zone.

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