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APPROATUS FOR PURIFYING LIQUEFIED GASES

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APPARATUS FOR PURIFYING LIQUEFIED GASES
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This invention relates to a process and apparatus for purifying liquefied gases, for example, liquid oxygen, nitrogen, and the like, which are produced in a manufacturing cycle, where they are generally contaminated with undesirable impurities and has for its main object an improved procedure whereby such impurities are readily and expeditiously removed.

More specifically, the invention relates to the provision of suitable steps for concentrating undesirable impurities occurring in the production of liquefied gases of the character indicated in a manner in the cycle of production such that the impurities may be separated readily by mechanical means, whereby a gas product is finally obtained in the liquid phase from which substantially all impurities are practically completely removed.

Another object is to produce a liquefied gas in a purified condition with an expenditure of a relatively small amount of additional energy and at relatively little additional cost.

Still another object is to separate the desired liquefied gas from the undesired impurity by the step either of partial evaporation or partial condensation and thereafter causing the gas material to revert to the liquid phase with an expenditure of a relatively small amount of energy.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

This application is a division of my original application Serial No. 607,032, filed April 23, 1932.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, and the apparatus embodying features of construction, combinations of elements and arrangement of parts which are adapted to effect such steps, all as exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawings, in which:

Fig. 1 is a view mainly in vertical cross section showing a simple form of apparatus adapted for carrying out the process of the present invention; and

Fig. 2 is a view mainly in vertical section showing a gas production apparatus incorporating purifying apparatus arranged for the practice of the present invention.

In the commercial production of a purified liquefied gas from which undesirable impurities are substantially completely removed, the purification may be accomplished with the expenditure of relatively little energy by taking the gas material in the liquid phase and treating it by the step of partial evaporation to produce two phases and then separating the phases; this process being generically set forth in the copending application filed in the name of Lawrence J. Bowitch on April 23, 1932, Serial No. 607,074, where it is proposed to remove those impurities which occur in relatively small amounts in liquefied gases such as liquid air, oxygen, carbon dioxide, and the like, the concentration being such that they do not materially change the boiling point of the liquefied gas, but are otherwise objectionable and impair the commercial qualities of the gas product.

There are certain types of impurities commonly incorporated with the gas material, on account of the source from which the gas is obtained and on account of the manufacturing steps commonly employed, which are less volatile than the gas material, i. e. have melting points materially above the boiling point of the gas material. To accomplish the removal of such impurities, it is proposed to bring the gas material containing the undesired impurity in a single phase by the step of fractional evaporation or condensation under conditions of temperature and pressure where the vapor pressure of the impurity is of a negligible value.

Two phases are thus produced that may be separated physically while the impurities remain in the liquid or solid phase. Here it is proposed to practice these steps in conjunction with a step of condensation, in order finally to obtain a liquefied gas such as liquid oxygen in substantially pure condition, this step being preferably practiced so as to accomplish the condensation by the utilization of a by-product from another portion of the cycle of production.

Referring now to the drawings, and particul-
larly to Fig. 1. A denotes generally a part of the apparatus into which the gas material in liquid phase that is to be purified is passed and caused to evaporate partially by the application of heat, while B denotes a section of condenser where the vapor produced in part A, which is conducted thereto and recondensed by being brought into thermal contact with a fluid of lower temperature than the condensing temperature of the gas; material being purified.

The vaporizing portion of part A consists of a vessel 10 having an inlet conduit 11 for the liquid, a conduit 12 for the vapor, and a drain conduit 13 with a control valve 13a.

In the lower part of vessel 10 is a suitable heating means, for example a coil 14 provided with inlet and outlet connections adapted to be supplied with a heating agent. This agent may be any convenient vehicle that will pass without difficulty at the low operating temperatures here employed, for example, compressed air. In the preferred form of the apparatus, the vapor outlet conduit 12 surrounds the inlet conduit 11 in order to effect heat exchange and separation of the liquid from the gas phase; the vapor there separated being led from the top of the evaporating vessel 10 to the condenser.

The condenser shown in connection with part B consists of an upper header 15 and a lower header 16 connected by condenser tubes 17. The conduit 12 is here shown as discharging the vapor to be condensed into the upper header 15, while a conduit 18 is connected to the lower header to withdraw the liquefied gas. The condenser is disposed within a chamber 19 having inlet and outlet conduits 20 and 21, respectively, for the purpose of circulating a cooling medium, for example, liquefied oxygen, or a cold gaseous material that may be a by-product drawn from another part of the manufacturing cycle; the cooling medium carrying away the heat from the condenser so that re-liquefaction is readily accomplished in the condenser.

The operation of the process in the apparatus described is as follows: In previous portions of the manufacturing cycle, certain impurities are not entirely removed and hence appear, though in relatively small quantities in the liquefied gas which enters the vaporizer A through conduit 11. The flow of liquid into the vaporizer and the rate at which heat is supplied by the heating fluid passing through the coil 14 are adjusted by the operator to maintain a certain liquid level in the evaporator. The supply of heat causes evaporation of the liquid to occur, but the impurities, which are solid or in solution have a negligible vapor pressure at the temperature of the evaporating liquid, and maintain in the liquid and are concentrated at this point in the cycle. The concentrated impurities may be drawn off at will through conduit 13 by means of valve 13a, together with some of the liquefied gas.

The vapor is conducted through conduit 12 into the condenser at its upper header 15 whence it passes into the tubes 17 and is condensed because it loses heat through the tube walls to the fluid of lower temperature surrounding the tubes. The liquefied gas collects in the lower header 16 from which it is withdrawn through conduit 18 in a purified state.

In the modified form of apparatus shown in Fig. 2, a heat exchanger for the manufacture of liquefied oxygen is modified and arranged to separate out the impurities which have relatively high boiling points and are in general inflammable, and then recondense the purified oxygen in order to supply it in the liquid phase. Here a conduit 25 provided with an expansion valve 26 is arranged to supply compressed air drawn from a suitable high pressure source to the rectifying column shown generally at 27. This rectifying column is provided with a kettle or evaporator 28 at its lower end and a condenser 29 at its upper end. A conduit 30 provided with an expansion valve 31 and distributor 32 is arranged to draw liquid from the evaporator 28 and to discharge it into the upper portion of a second rectifying column 33 disposed above the column 27 and arranged to be in heat exchanging relation with the condenser 29. Each of these columns is provided with suitable counter-current contact cooling means, for example, a series of perforated trays, as shown at 27' in the column 27 and at 33' in the column 33. The evaporator 28 associated with the column 27 is provided with suitable heating means, for example, a heating coil as shown at 28'.

Within the column 27 is a tray 27'' arranged to collect the condensate which drips from the condenser 29 and to convey the same to a conduit 34 having a controlling valve 35 and a distributor 36 discharging into the top of the column 33, the distributor 36 being preferably disposed above the distributor 32. From the top of the condenser 29 is a gas withdrawal conduit 37 which is arranged for withdrawing non-condensible vapors, while a liquid withdrawal conduit 38 is arranged to withdraw liquid from the base of the column 33 from about the condenser 29.

From this arrangement, it is seen that the compressed air introduced into the column 27 is liquefied by the passage through the expansion valve 26 and through the trays 27''; the liquid collecting in the evaporator 26. Gas or vapor rising through the column 27 passes into the condenser 29 where the less volatile portion is recondensed to produce a reflux in the column 27. Liquid nitrogen collects in the tray 27''', whereas the liquid in the evaporator 28 comprises a relatively large percentage of liquid oxygen. Any non-condensable vapor that is more volatile than liquid nitrogen or oxygen is withdrawn through the column 37. Liquefied gas comprising mainly liquid oxygen and the impurities of relatively high melting point are withdrawn through the conduit 30 and introduced into the column 33 through the distributor 32.

In order to effect the final stage of purification, in accordance with the present invention, a second condenser shown generally at 40 is incorporated at a convenient point in the column 33. This condenser comprises upper and lower portions 41 and 42, connected by an intermediate portion 43. One or more passages or fluids are associated with the condenser in order to establish communication between the space above the condenser with the space below; a chamber for this condenser being preferably provided in the column by means of a partition 45 disposed transversely in the column below the condenser and a second partition 46, which is disposed above the condenser. This chamber occupies a space normally occupied by counter-current contact cooling trays, and preferably has trays both above and below the same, those shown below being denoted 33''.
The partition 46 is provided with one or more passages 47 that permits communication with the flues 44 so as to establish communication in the column 33 for the space on the two sides of the condenser chamber containing condenser 40. The partition 46 is also provided with one or more liquid drains 48 which allow liquid to drain from the partition into the chamber about the condenser 40. Gaseous oxygen to be recondensed and which has been evaporated by the condenser 29 is led from a point near the lower end of the column 33 from the chamber about the condenser 29 by means of a conduit 50 and passed into the portion 41 of condenser 40. Here liquid oxygen collects in a purified state and is withdrawn from the lower portion 42 through a conduit 51.

The operation of this latter form of apparatus is seen to be as follows: The compressed air from which oxygen in the liquid phase and in a purified state is desired to be obtained is introduced into the rectifying column 27 either with or without previous partial purification. This is then passed through the conduit 30, the expansion valve 31 and the distributor 32 into the column 33, where the cooled oxygen condenses out and passes down through the trays 33' and through the drain 48 into the chamber about the condenser 40. When the desired liquid level is attained in this chamber, the cooled liquid overflows through the by-pass 44 into the lower portion of the column 33 over the trays 33" and finally collects in the chamber about the condenser 29. Here the heat absorbed from the condenser 29 in effecting rectification for the column 27 vaporizes a portion of the liquid which is now a high concentrate of liquid oxygen contaminated with the impurities of relatively high melting point. The oxygen vaporized in this chamber is substantially pure oxygen gas which is withdrawn through the conduit 50 and passed into the condenser 41, where it is recondensed by the heat abstracted by the liquid in the chamber about the condenser 41. Thus it is seen that liquidified oxygen in the purified state desired collects in the portion 42 and is withdrawn by the conduit 51.

The liquid which collects in the chamber about the condenser 41 is seen to comprise a mixture of oxygen and nitrogen in the liquid phases, together with any substances that may be present either as solids or in solution. The vaporization which takes place in this chamber is of a fractional nature, the more volatile nitrogen being boiled off and passed up through the flues 47 into the upper portion of the column 33. This liquid nitrogen is seen to be supplied to the column mainly from the conduit 34; thus the cooling of the condenser 41 is seen to be accomplished by means of a cooling agent that is withdrawn from another portion of the cycle of production. The vaporized nitrogen is seen to pass from the top of the column 33 in the usual manner.

The agents here used for heating the liquid in the evaporators and for extracting heat from the vapor in the condensers, in order to conserve energy, are thus with advantage both taken from some other portions of the cycle of production, and since no change has occurred therein other than a change in the quantity of absorbed heat, they may be returned, if desired, to the cycle substantially without loss. Since certain changes in carrying out the above process and in the constructions set forth, which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

Having described my invention, what I claim as new and desire to secure by Letters Patent is:

1. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material in the liquid phase is introduced and an evaporator adjacent the lower end, of counter-current gas and liquid contact—means in said column between said inlet and said evaporator, said rectification on column being provided with a chamber to which a refrigerating agent is admitted from another part of the system, a condenser disposed in said chamber said stored from the walls thereof, means for supplying a gas fraction drawn from said evaporator to said condenser, and means for withdrawing the condensation product directly from said condenser to a point on the exterior of said column. 2. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material substantially in the liquid phase is introduced, an evaporator at its lower end and a condenser at its upper end, of a second rectification column having its lower end connected to be in heat exchanging relation with the fluid in said condenser, a connection leading from said first rectification column and discharging into the second near the upper end thereof, an auxiliary condenser located within said second rectification column exposed to a refrigerating agent and spaced from the column walls, a connection arranged to supply a gas fraction withdrawn from said second rectification column to said auxiliary condenser which is distilled off by the heat of said first condenser, and a withdrawal connection for withdrawing the condensate from said auxiliary condenser. 3. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material substantially in the liquid phase is introduced, an evaporator at its lower end and a condenser at its upper end, of a second rectification column having its lower end connected to be in heat exchanging relation with said condenser and a connection for receiving liquid at its upper end which is withdrawn from said evaporator, said second rectification column being formed with a chamber at a point intermediate of its ends, a second condenser disposed within and spaced from the walls of said chamber, means for supplying a refrigerating agent to said chamber, means for supplying a gas fraction distilled from said second rectification column to said second condenser whereby a purified liquefied gas is collected in said second condenser, and means for directly withdrawing the collected condensate from said second condenser. 4. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material substantially in the liquid phase is introduced, an
evaporator at its lower end and a condenser at its upper end, of a second rectification column superposed above the first column and having its lower end in heat exchanging relation with said condenser and a connection for receiving liquid at its upper end which is withdrawn from said evaporator, said second rectification column being formed with a chamber at a point intermediate of its ends, a second condenser disposed within and spaced from the walls of said chamber, means for supplying a refrigerating agent to said chamber withdrawn from another portion of the apparatus, means for supplying a gas fraction to said second condenser distilled from said second rectification column whereby the purified liquefied gas is collected in said condenser, and means for directly withdrawing the collected condensation product from said second condenser.

5. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material in the liquid phase is introduced near the upper end and an evaporator adjacent the lower end, of counter-current contact means in said column between said inlet and said evaporator, perforated partitions located so as to interrupt the regular occurrence of said counter-current contact means at an intermediate point and providing a chamber within said rectification column, a condenser disposed within and spaced from the walls of said chamber, the perforations in said partitions being arranged for admitting a refrigerating agent to said chamber, means for supplying a gas fraction drawn from said evaporator to said condenser, and means for withdrawing the condensation product from said condenser.

6. In apparatus for the commercial production of liquid oxygen free from undesired impurities, the combination with a rectification column having an inlet by which gas material in the liquid phase is introduced near the upper end and an evaporator adjacent the lower end, of counter-current contact means in said column between said inlet and said evaporator, perforated partitions interrupting said counter-current contact means at an intermediate point to provide a chamber within said rectification column, a condenser within and spaced from the walls of said chamber, pipe connections for leading a gas fraction directly from said evaporator to said condenser, means for withdrawing the condensation product from said condenser, the perforations in said partitions admitting the passage of liquefied gas material drawn from an upper part of the column as the refrigerating agent to said chamber, and means for withdrawing said gas material when the refrigerating effect is spent and returning the same to another part of said rectification column.

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