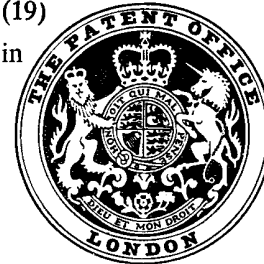


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## (54) PROCESS FOR LIQUEFYING AND RECTIFYING AIR

(71) We, NIHON SANSO KABUSHI-KI KAISHA AND TOKYO REINETSU SANGYO KABUSHIKI KAISHA, both a joint-stock company duly organized under the laws of Japan, residing at No. 16-7, 1-chome, Nishi-Shinbashi, Minato-ku, Tokyo and No. 3-18, 6-chome, Roppongi, Minato-ku, Tokyo, Japan, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:-

The present invention seeks to enable a substantial reduction of compression power required for air separation, that is, liquefaction and rectification of air to separate oxygen, nitrogen and other materials and in particular to extract them as liquid products.

Most of the cost of separating and extracting air into oxygen, nitrogen and other materials is that of power; and most of this power is consumed in the compression of feed air. Therefore a reduction of this compression power is immediately contributive to the amount of power per unit volume of the products. Various solutions have been proposed along this line e.g. utilisation of the cold of LNG, based on the fact that power required to compress gas is reduced by lowering the inlet temperature of feed air. However, in a plant which produces say 10,000 m<sup>3</sup>/h of oxygen, five times as much feed air as the resultant product is required and hence the accrued saving is counterbalanced. This happens either

(1) By larger capital and power costs of the absorbing facilities required to absorb moisture, carbon dioxide and other impurities, (necessary to avoid solidification in the process) or

(2) because in a conventional method of cooling feed air and removing impurities by the use of a regenerative cooler or a reversing heat exchanger, the required level

of feed air compression so as to enable regasification of impurities, is about 5 kg/cm<sup>2</sup>G. This means that despite the use of LNG the power saving is not as large as expected, and the contribution of the cold of LNG is limited to the supplementation of cold energy in liquefaction/rectification stages.

According to the present invention therefore, there is provided a process for separating nitrogen and oxygen from air by liquefaction and rectification which comprises:-

supplying cooled purified air to a rectification step,

separating air into nitrogen and oxygen,

cooling said supplied air by heat exchange with a portion of said separated nitrogen,

recooling said nitrogen by heat exchange with LNG, and subsequently compressing and supplying the so cooled nitrogen to a nitrogen condenser whereby rectification is effected substantially at atmospheric pressure.

According to another aspect of the invention there is provided an apparatus for separating nitrogen and oxygen from air by liquifaction and rectification which comprises:-

means for supplying cooled purified air to a rectification column substantially at atmospheric pressure,

rectification column means for separating said air into nitrogen and oxygen substantially at atmospheric pressure,

means for cooling said supplied air by heat exchange with a portion of said separated nitrogen, means for recooling said nitrogen by heat exchange with LNG,

and means for subsequently compressing and supplying the so cooled nitrogen to a nitrogen condenser: thereby to provide liquified nitrogen and oxygen by rectification

substantially at atmospheric pressure.

Thus, oxygen is evaporated in a rectifying operation by pressurized circulating nitrogen, so that this operation can be effected at about 0.5 Kg/cm<sup>2</sup>G (rectifying column pressure). The compression of feed air thus is only necessary to a pressure where feed air can still be fed into the rectifying step. That is to say, since a part of separated nitrogen is used for a pressurized heating gas which is utilized to evaporate oxygen in the rectifying operation (instead of compressed air which is generally provided), the air supply may be effected by blowing instead of compressing the same.

Thus, the compression of said circulating nitrogen can be effected at an extremely low temperature of about -140°C for example by making effective use of the cold of LNG; resulting in a marked reduction in the power. The present invention is illustrated, merely by way of example, in the accompanying drawing which is a flow chart showing one embodiment according to the invention.

31,800 m<sup>3</sup>/h of feed air enters air compressor (2) through piping (1) where it is compressed to 1.2 kg/cm<sup>2</sup>G. Upon removal of the heat of compression in heat exchanger (3) the air is introduced into heat exchanger (4). This heat exchanger (4) is cooled by Freon (Registered Trade Mark) which is cooled by LNG and which circulates in a closed cycle as described later. The cooled air then enters absorber (5) for moisture removal and then passes to heat exchanger (6) cooled by the separated, low-temperature nitrogen gas. After passing through heat exchanger (6), the air passes into absorber (7) for removal of carbon dioxide, is further cooled in heat exchanger (8) and led through piping (9) to the first rectifier (10). This first rectifier (10) corresponds to a high pressure tower in a conventional plant and is operated at approximately 0.5kg/cm<sup>2</sup>G, whereas a conventional pressure tower is usually operated under pressure of 4.5kg/cm<sup>2</sup>G. This means that the necessary pressure for compressing feed air is such that the air can only just reach the rectifying process after passing through the pretreatment stages necessary for rectification.

The feed air is rectified in this first rectifier (10), so that nitrogen is separated to the upper part of the column and oxygen-rich liquid air to the lower. For further rectification, the oxygen-rich liquid is passed to the second rectifier (12) through piping (11). The second rectifier (12) is operated under generally the same pressure as the first rectifier (10), so that nitrogen is separated at the upper part of the column and liquid oxygen above the condenser (13) at the lower part. 6,000 m<sup>3</sup>/h of liquid

oxygen thus produced is extracted as product from piping (14).

Meanwhile nitrogen forming in the upper part of the rectifier 12 is extracted through piping (15), and is used to cool feed air by counterflow through heat exchangers (8) and (6). This gas is consequently warmed and discharged. Nitrogen extracted through piping (16) joins nitrogen issuing from the top of the first rectifier (10). A part of this nitrogen goes to piping (17). The remainder is brought into countercurrent contact with feed air in the heat exchangers (8), (6) and (3) for cooling, so that it is warmed almost to ambient temperature. This nitrogen, passing through piping (18), is subsequently cooled to -140°C in heat exchanger (19) which constitutes a part of the Freon cooling cycle, and after joining the flow of nitrogen in piping (17), it enters nitrogen compressor (20) where it is compressed to 5 kg/cm<sup>2</sup>G. This compressed nitrogen is introduced through piping (21) into heat exchanger (22) constituting a part of the Freon cycle as in the case of heat exchanger (19), where it is cooled to -132°C. A part of this gas is separated to piping (23). The remainder is compressed to 30 kg/cm<sup>2</sup>G in nitrogen compressor (20) and then goes through heat exchangers (24) and (25) where it is cooled by LNG. It is further super-cooled in heat exchange with the separated, low-temperature nitrogen having been bypassed to piping (17) in heat exchanger (26), expanded to 5 kg/cm<sup>2</sup>G through expansion valve (27) and is introduced into condenser (13) in the second rectifier (12). The flow of nitrogen of 5 kg/cm<sup>2</sup>G at -132°C, which is bypassed into piping (23), is also introduced into condenser (13) through heat exchanger (26). The two flows of nitrogen are condensed so that liquid nitrogen is collected at the bottom of condenser (13). This liquid nitrogen is extracted by piping (28) and 6,000 m<sup>3</sup>/h is collected as a product through piping (29). The remainder is expanded to 0.5 kg/cm<sup>2</sup>G through expansion valves (31) and (32) and is refluxed into the first and second rectifiers (10) and (12).

LNG is supplied through piping (33). A part of it is expanded through expansion valve (34) and is introduced into LNG heat exchanger (25) to cool compressed nitrogen at 30 kg/cm<sup>2</sup>G. LNG *per se* is gasified and leaves through piping (35), and is compressed to a suitable pressure in compressor (36) for supply as gaseous fuel or feedstock. The rest of the LNG is separately supplied to LNG heat exchanger (24) and Freon heat exchanger (38) by way of piping (39) and (40) respectively, and imparts its cold to compressed nitrogen and Freon in those heat exchangers, whereby this LNG *per se* is again gasified and flows into piping (41) for supply as gaseous fuel or feedstock.

Reference numeral (42) in the diagram is a Freon circulating pump. Freon is cooled by LNG in freon heat exchanger (38) and is separately introduced into heat exchangers (22), (19) and (4). The warmed freon joins together and returns to circulating pump (42).

While this example shows the first and second rectifiers, it is possible to eliminate the first rectifier by feeding air directly into the middle of the second rectifier.

As seen from the above description, this invention has many characteristic features which are not found in the existing facilities, and an appropriate combination of such features leads to a considerable reduction power requirement. For instance, a conventional plant is generally designed to compress feed air to 5 kg/cm<sup>2</sup>G and rectify the compressed air at 4.5 kg/cm<sup>2</sup>G in a pressure tower followed by further rectification at about 0.5 kg/cm<sup>2</sup>G; hence, there is limit on the possibility of reduction in the pressure of feed air. However, pressure for compressing feed air under the process of the present invention need only be such that the air reaches the rectifying stage through pre-treatment stages, since the rectifiers operate only at about 0.5 kg/cm<sup>2</sup>G. This is achieved by arranging that nitrogen circulation is via condenser (13) which performs the function of rectification and reboiling in a conventional pressure tower and also by making effective use of the cold of LNG. In addition, the effective use of the cold of LNG renders it possible to compress the circulating nitrogen at an extremely low temperature in the order of -140°C. This also serves to reduce power requirement which is not attainable in the known process. In this connection, a comparison is made between this and conventional processes i.e. in the case of process in which LNG is not employed, power consumption per unit liquid product is about 1.2 KWH/Nm<sup>3</sup>, whereas it is about 0.76 KWH/Nm<sup>3</sup> in the case of usual process but in which LNG is used and feed air is compressed to 5 kg/cm<sup>2</sup>G. However, the process according to the present invention gives this unit of about 0.5 KWH/Nm<sup>3</sup>. This is a considerable reduction over both the first and second cases. In addition, the lower rectifying pressure gives the higher efficiency of separation and also makes it possible to save capital costs.

Since the pressure of the feed air is set at 1.2 kg/cm<sup>2</sup>G in this invention, removal of impurities contained in the air is done by absorbents rather than by cooling by a regenerative cooler or a reversing heat exchanger. This gives no demerit in the facilities but instead, because of the merits as discussed above, it enables larger extraction of nitrogen product. Extracting oxygen and nitrogen as liquid products has been

exemplified here, but it is possible to collect them as gaseous products. In addition, it goes without saying that the utilization of the cold of LNG can be expanded to replace the Freon cycle by making simple modifications to the design.

#### WHAT WE CLAIM IS:

1. A process for separating nitrogen and oxygen from air by liquifaction and rectification which comprises:-  
supplying cooled purified air to a rectification step,  
separating air into nitrogen and oxygen,  
cooling said supplied air by heat exchange with a portion of said separated nitrogen,  
recooling said nitrogen by heat exchange with LNG, and  
subsequently compressing and supplying the so cooled nitrogen to a nitrogen condenser whereby rectification is effected substantially at atmospheric pressure.
2. A process according to claim 1 wherein heat exchange with LNG is effected indirectly by means of a closed heat exchange loop containing a second heat exchange medium.
3. A process as claimed in claim 1 or claim 2 in which two rectifying columns are used.
4. A process as claimed in any preceding claim in which said rectifying step is effected at about 0.5 kg/cm<sup>2</sup>G.
5. A process as claimed in any preceding claim in which the separated, low temperature nitrogen gas is compressed to 30 kg/cm<sup>2</sup>G at -140°C.
6. A process according to any preceding claim and substantially as hereinbefore set forth.
7. A gas separated according to a process as claimed in any one of claims 1 to 7.
8. An apparatus for separating nitrogen and oxygen from air by liquifaction and rectification which comprises:-  
means for supplying cooled purified air to a rectification column substantially at atmospheric pressure,  
rectification column means for separating said air into nitrogen and oxygen substantially at atmospheric pressure,  
means for cooling said supplied air by heat exchange with a portion of said separated nitrogen,  
means for recooling said nitrogen by heat exchange with LNG,  
and means for subsequently compressing and supplying the so cooled nitrogen to a nitrogen condenser; thereby to provide liquified nitrogen and oxygen by rectification substantially at atmospheric pressure.
9. An apparatus according to claim 8 wherein the recooling means includes a closed loop heat exchange circuit provided

with a second heat exchange medium, said loop being adapted to cool the air supply.

10. An apparatus according to either of claims 8 or 9 comprising two rectifying columns in series.

- 5 11. An apparatus according to claim 8 substantially as hereinbefore set forth with reference to the accompanying drawing.

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