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## (54) Dual purity oxygen generator with reboiler compressor

(57) Low and high purity oxygen products are produced by cryogenic distillation of a compressed feed air stream (10) in which a low purity (less than 97%) oxygen product (38) is provided by a conventional low purity column (25) and high purity (more than 97%) oxygen product (36) is provided by rectification in a high purity column (23) of an oxygen-rich stream (35) from the distillation system (15, 25). Boilup (31) of the low purity column (25) is provided by condensation of a suitable first process stream (30) and boilup in the high purity column is provided by condensation of a suitable second process stream (21) at a higher pressure than said first process stream (30). The oxygen rich stream (35) has an oxygen concentration at least equal to that of the feed (26') to the low pressure column (25).

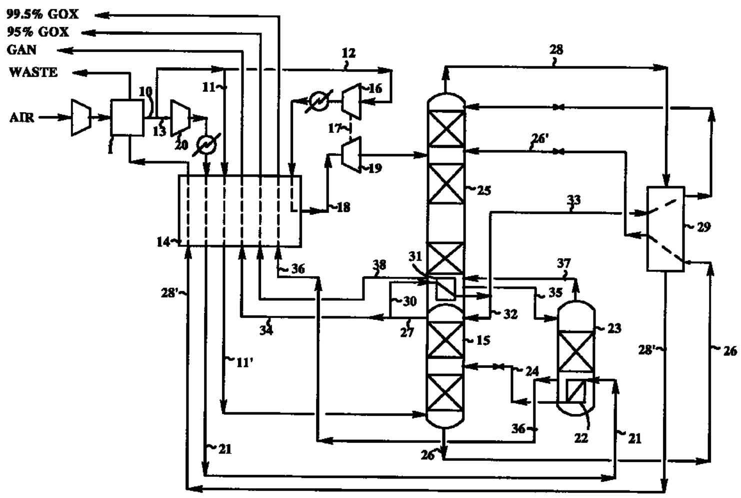


Figure 1.

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## Description

The objective of the invention is to efficiently and cost-effectively generate two different oxygen purities from the same air separation plant, when only a small amount of high purity oxygen is required. It also provides a way to make small amounts of crude argon product if required on a plant which makes predominantly low purity oxygen. This is consistent with the requirements of new grass-roots steel mills based on the COREX steel-making process.

In the past there were two commonly used alternatives to make two different oxygen purities from the same facility. One was to build two independent cryogenic trains - one for each purity. This is capital intensive and complicated. The other was to design the entire plant for the high purity, requiring a main air compressor discharge pressure at a pressure consistent with high purity oxygen. This is not energy efficient, since much of the air only needs to be raised to a lower pressure consistent with generating low purity oxygen.

A number of efficient plants for the production of low purity oxygen are known in the literature. US-A-4,702,757; US-A-4,704,148; and US-A-4,936,099 describe a number of very efficient process cycles employing multiple reboiler condensers. However, none of these cycles involve the coproduction of a portion of oxygen product at a purity greater than 95%.

US-A-5,515,833 describes a cycle in which a portion of expanded gas from a compander is used to reboil the bottom reboiler of a dual-reboiler low pressure ("LP") column. In this cycle, one double-column assembly is used for the two products. All air feed to the process is compressed to a pressure sufficient to allow the expander exhaust to reboil the high purity oxygen in the bottom reboiler of the LP column. A high pressure nitrogen stream from the top of the high pressure ("HP") column feeds an intermediate reboiler which is at a location above the low purity oxygen withdrawal location. This makes the process inefficient in that very high recoveries of oxygen and, especially, nitrogen cannot be achieved because a large quantity of air needs to be condensed in the bottom reboiler of the LP column to provide the boilup for both the high purity and the low purity gaseous oxygen products and also all the vapour stream for the distillation above the low purity oxygen withdrawal location. The large quantity of air which needs to be condensed in the bottom reboiler of the LP column decreases the amount of available liquid nitrogen reflux and negatively impacts oxygen recovery. Further, the larger portion of air needed at higher pressure to be condensed against pure oxygen increases energy consumption and thereby wastes compression energy. This process also forces the LP column to be sized for the entire air feed, leading to potential manufacturing and transportation difficulties.

US-A-5,515,833 also shows an argon sidearm column connected to the main column between the two oxygen products withdrawal locations. This feed location is necessary to eliminate nitrogen from the sidearm column feed. However, it provides a sidearm column feed with a very low argon concentration (less than 4% Ar). This makes this distillation much more difficult than where the sidearm column feed is in the 9-14% Ar range. Due to low argon concentration in the feed to the sidearm column, for a given oxygen recovery, the argon recovery will be poor.

Clearly there is a need for more efficient cycles to produce high purity as well as low purity oxygen with high efficiency and ease of operability.

The present invention relates to cryogenic distillation of a stream containing nitrogen and oxygen to efficiently produce oxygen at at least two levels of purity. The first product is a low-purity oxygen stream containing less than 97% oxygen (but generally greater than 80% oxygen) and the second product is a high purity oxygen product stream containing more than 97% oxygen, preferably more than 99.5% oxygen. The high efficiency is achieved by taking a high efficiency process cycle for the production of low purity oxygen and modifying it according to the invention. The high efficiency process cycle consists at least a distillation column where feed stream is distilled to produce low purity oxygen from the bottom and a nitrogen-rich stream from the top. The bottom of this column has a reboiler where a suitable process stream is condensed to provide boilup to the distillation column. According to the invention, a liquid stream having an oxygen concentration at least equal to that of said feed stream is withdrawn either from the bottom of this first column (at the location of the bottom reboiler) or from a point which is some separation stages above the withdrawal location of the low purity oxygen and fed to the top of a sideleg column. The bottom of the sideleg column is boiled by a suitable process fluid and high-purity oxygen product is withdrawn from the bottom of this sideleg column. The vapour from top of the sideleg column is returned to the first column, preferably at same separation stage from where the liquid feed stream was withdrawn.

When coproduction of argon is desired, an argon sidearm column is attached at a proper location of the sideleg column producing high-purity oxygen, i.e., a vapour feed from an intermediate location of the sideleg column is fed to the argon sidearm column to produce argon from the top of this column and the liquid stream from the bottom of this column is returned back to the sideleg column.

The advantage of this method is that boilup at the high temperature of the high purity oxygen is kept at minimum. A much larger amount of heat is provided at the location of the production of low purity oxygen. This leads to substantial energy savings.

In its broadest aspect, the present invention provides a process for cryogenic distillation of a compressed feed air stream in a distillation system comprising a low purity column producing a low purity (less than 97%) oxygen product stream and a nitrogen-rich stream, said column having a bottom reboiler in which a suitable first process stream is condensed to provide boilup to the column, characterized in that an oxygen-rich stream having an oxygen concentration at least equal to that of the feed to the low purity column is withdrawn from the distillation system and rectified in a high purity column whose bottoms reboiler heat is provided by condensation of a suitable second process stream to provide a high-purity (more than 97%) oxygen product stream, said second process stream being at a higher pressure than said first process stream.

Usually, the low purity oxygen product stream will contain more than 70% oxygen and preferably at least 90% oxygen. The high purity oxygen product stream preferably contains at least 99.5% oxygen.

In a presently preferred embodiment of the invention,the distillation system comprises a high pressure column and a low pressure column and:

(a) at least a portion of the compressed air feed is fed to the high pressure column in which the air feed is rectified into a high pressure nitrogen overhead and a high pressure crude liquid oxygen bottoms;
(b) at least a portion of the high pressure crude liquid oxygen bottoms is fed to the low pressure column in which the high pressure crude liquid oxygen bottoms is rectified into a low pressure nitrogen overhead and a low pressure liquid oxygen bottoms;
(c) at least a portion of the high pressure nitrogen overhead is condensed and at least a portion of the condensed high pressure nitrogen overhead is returned to the high pressure column as reflux;
(d) at least a portion of the low pressure liquid oxygen bottoms is boiled by a bottom reboiler in which a suitable first process stream is condensed;
(e) the low purity oxygen product stream is withdrawn from the low pressure column;
(f) the oxygen-rich stream having an oxygen concentration at least equal to that of the high pressure crude liquid oxygen bottoms is withdrawn from the distillation system and fed to a high purity column in which it is rectified into an oxygen-lean overhead vapour and a high-purity liquid oxygen bottoms;
(g) at least a portion of the high purity liquid oxygen bottoms is boiled by a bottom reboiler by condensation of a suitable second process stream compressed to a pressure higher than that of the first process stream boiling the low pressure column;
(h) the high purity oxygen product stream is withdrawn from the high purity column.

The process stream providing bottom reboil to the high purity column is a portion of the compressed feed air which has been further compressed. At least a portion of this compressed feed air portion can be is fed to the high pressure column and/or to the low pressure column.

Alternatively, the process stream providing bottom reboil to the high purity column can be at least a portion of the high pressure nitrogen overhead which has been further compressed.

The oxygen-rich stream can be withdrawn from the low pressure column, usually the sump thereof, and, preferably, the oxygen-lean overhead vapour is returned to the low pressure column at substantially the same location as that at which the oxygen-rich stream was withdrawn.

Alternatively, the oxygen-rich stream can be withdrawn from the high pressure column, suitably as a portion of the high pressure crude liquid oxygen bottoms. A vapour stream can be withdrawn from the low pressure column at a location below the feed of the high pressure crude liquid oxygen bottoms thereto and fed to the high purity column at a location below the feed of the oxygen-rich stream thereto.

The process stream providing bottom reboil to the low pressure column can be at least a portion of the high pressure nitrogen overhead or a portion of the compressed feed air. When it is a portion of the compressed air feed, at least a portion of the resultant condensed fed can be fed to the high pressure column and/or to the low pressure column. If the high pressure nitrogen overhead is not condensed to provide low pressure column reboil, at least a portion thereof can be condensed at an intermediate location in the low pressure column.

As mentioned previously, an argon rich vapour stream withdrawn from an intermediate location of the high purity column can be separated in an argon column to produce an argon product stream and a liquid argon-depleted stream which is returned to the high purity column.

When high pressure nitrogen overhead is condensed at an intermediate location in the low pressure column, the oxygen-rich stream suitably is withdrawn from the low pressure column at a location above said intermediate location and an argon rich vapour stream withdrawn from an intermediate location of the high purity column is separated in an argon column to produce an argon product stream and a liquid argon-depleted stream which is returned to the high purity column.

Argon overhead from an argon column can be condensed by boiling a portion of the high pressure crude liquid oxygen bottoms and the vaporized high pressure crude liquid oxygen bottoms fed to the low pressure column and/or to the high purity column.

A portion of the compressed air feed can be fed to the low pressure column or a portion of the compressed air feed further compressed to a higher pressure than the main compressed air feed portion to the high pressure column and at least a portion of the further compressed feed air portion fed to the high pressure column and/or to the low pressure column.

At least a portion of the condensed high pressure nitrogen overhead can be fed to the low pressure column as reflux. Alternatively, all of the condensed high pressure nitrogen overhead can be fed to the high pressure column as reflux and the low pressure column refluxed with a sidestream withdrawn from the high pressure column. A portion of the nitrogen rich sidestream can be fed to the high purity column.

If not all of the high pressure nitrogen overhead is condensed, the remaining portion can be recovered as product.

The low purity oxygen product stream can be boiled against the portion of the compressed air feed fed to the high pressure column to at least partially vaporize the compressed air feed portion.

The low purity oxygen product stream and high purity oxygen product stream can be withdrawn from the low pressure column and high purity column respectively as liquids and pumped prior to heat exchange with the main compressed air feed to generate pressurized oxygen products.

The present invention also provides an apparatus for producing low purity oxygen and high purity oxygen products by the cryogenic distillation of a compressed air feed by the process of Claim 1, said apparatus comprising:

(i) a distillation system for rectifying the compressed air feed and comprising a low purity column for producing a low purity (less than 97%) oxygen product stream and a nitrogen-rich stream;
(ii) a bottom reboiler in said low purity column for condensing a suitable first process stream to provide boilup to the low purity column;
(iii) means for supplying said first process stream to said reboiler; and
(iv) means for withdrawing the low purity oxygen product stream from the apparatus, characterized in that the apparatus further includes
(v) a high purity column for rectifying an oxygen rich stream having an oxygen concentration at least equal to that of the feed to provide a high-purity (more than 97%) oxygen product stream;
(vi) means for withdrawing said oxygen-rich stream from the distillation system and feeding it to the high purity column;
(vii) a bottom reboiler in said high purity column for condensing a suitable second process stream to provide boilup to the high purity column;
(viii) means for supplying said second process stream to said high purity column reboiler at a higher pressure than said second process stream is supplied to the low purity column reboiler; and
(ix) means for withdrawing the high purity oxygen product stream from the apparatus.

In a presently preferred embodiment of the apparatus aspect the distillation system comprises:

(1) a high pressure column for rectifying at least a portion of the compressed air feed into a high pressure nitrogen overhead and a high pressure crude liquid oxygen bottoms;
(2) a low pressure column for rectifying the high pressure crude liquid oxygen bottoms into a low pressure nitrogen overhead and a low pressure liquid oxygen bottoms;
(3) means for feeding at least a portion of the high pressure crude liquid oxygen bottoms to the low pressure column;
(4) means for condensing at least a portion of the high pressure nitrogen overhead and returning at least a portion of the condensed high pressure nitrogen overhead to the high pressure column as reflux;
(5) a bottom reboiler in the low pressure column for condensing a suitable first process stream to provide boilup to the low pressure column;
(6) means for supplying said first process stream to said reboiler;
(7) means for withdrawing the low purity oxygen product stream from the apparatus;
(8) a high purity column for rectifying an oxygen rich stream into an oxygen-lean overhead vapour and a high-purity liquid oxygen bottoms;
(9) means for withdrawing the oxygen-rich stream from the distillation system and feeding it to the high purity column;
(10) a bottom reboiler in said high purity column for condensing a suitable second process stream to provide boilup to the high purity column;
(11) means for supplying said second process stream to said high purity column reboiler at a higher pressure than said second process stream is supplied to the low pressure column reboiler; and
(12) means for withdrawing the high purity oxygen product stream from the apparatus.

The following is a description by way of example only and with reference to the accompanying drawings of presently preferred embodiments of the invention. In the drawings:-

Figure 1 shows a first (basic) embodiment of the present invention;

Figure 2 shows a second embodiment of the present invention in which a LOX boil vaporizer is used for the low purity oxygen product;

Figure 3 shows a third embodiment of the present invention in which liquid pumps are used to generate pressurized oxygen and nitrogen products;

Figure 4 shows a fourth embodiment of the present invention which is a simplification of the embodiment of Figure 2 in which the LOX boil vaporizer is omitted;

Figure 5 shows a fifth embodiment of the present invention which is a modification of the embodiment of Figure 2 incorporating a crude argon sidearm column;

Figure 6 shows a sixth embodiment of the present invention which is a modification of the embodiment of Figure 3 incorporating a crude argon sidearm column and has the sideleg column decoupled from the LP column.

Figure 7 shows a seventh embodiment of the present invention which is a modification of the embodiment of Figure 6 in which the LP column is coupled to both the sidearm column and the sideleg column.

In all figures, the same reference numerals are used to identify the same or equivalent components.

The basics of the invention are described below with reference to Figure 1. Briefly stated, a system comprising a HP column (15), a LP column (25) and a sideleg column (23) is used. Low purity oxygen product (95% GOX) is obtained from the bottom of the LP column (25). High purity oxygen product (99.5% GOX) is obtained from the bottom of the sideleg column (23), in which a liquid stream (35) from the bottom of the LP column (25) is distilled and boiled by a portion (21) of the feed air (10) which has been boosted in pressure. Vapour (37) from the top of the sideleg column (23) is returned to the bottom of the LP column (25).

In more detail, clean, dry compressed air (10) from a front-end purification system (1) is split into three streams (11, 12, 13). A first feed air stream (11) is fed directly to a main heat exchanger (14), where it is cooled to near its dew point temperature, and then (11') to the HP column (15). A second feed air stream (12) is fed via the compressor end (16) of a compander (17) to the main heat exchanger (14) from which it is withdrawn as a side stream (18) and fed via the expander end (19) of the compander (17) to the LP column (25). The compander (17) generates refrigeration for the cycle. A third feed air stream (13) is fed via a low pressure booster air compressor (20) to the main heat exchanger (14) where it is cooled to near its dew point. The resultant cooled stream (21) is fed to a reboiler (22) in the bottom of the high purity or sideleg column (23) to provide reboil to that column. The liquified air (24) from this reboiler (22) is fed to the HP column (15).

Alternately, some or all of the liquified air (24) can be subcooled and fed to the LP column (25) rather than to the HP column (15).

The HP column (15) provides an initial distillation of air to generate a liquid oxygen-enriched (crude LOX) stream (26) at the bottom of the column and a HP gaseous nitrogen stream (27) at the top of the column. The crude LOX stream (26) has an oxygen content usually greater than 30% and more often greater than 35%. This stream (26) is subcooled against a LP gaseous nitrogen stream (28) from the top of the LP column (25) in a subcooler (29) and is then reduced in pressure and fed to an intermediate location of the LP column (25). The warmed LP gaseous nitrogen stream (28') is further warmed in the main heat exchanger (14) before being vented to atmosphere (WASTE) or recovered as a coproduct stream.

At least the majority (30) of the HP gaseous nitrogen (27) from the top of the HP column (15) is fed to a reboiler (31) in the bottom of the LP column (25). The condensed nitrogen stream from this reboiler (31) is split to provide two substreams (32, 33). One substream (32) is used to reflux the HP column (15) and the other substream (33) is cooled in the subcooler (29) and then used to reflux the LP column (25).

Part (34) of the gaseous nitrogen stream (27) withdrawn from the top of the HP column (15) is warmed in the main heat exchanger (14) for recovery as product (GAN). If no product nitrogen is required, all of the withdrawn HP nitrogen stream (27) can be fed to the reboiler (31). However, if large amounts of product nitrogen are required, it is typically more efficient to take this from the LP column (25).

If very high purity (ppm levels of oxygen) nitrogen product is required, it may be more efficient to reflux the LP column (25) with impure liquid nitrogen. In this case, all of the condensed nitrogen stream from the reboiler (31) is fed to the top of the HP column (15) and the LP column (25) is refluxed with a side stream (not shown) withdrawn from the HP column (15) several stages below the top and then cooled in the subcooler (29) prior to feed to the LP column (25).

A low purity liquid oxygen stream (35) is withdrawn from the sump of the LP column (25) and fed to the top of the sideleg column (23). The low purity liquid oxygen is distilled and boiled against the cooled third feed air stream (21) fed to the reboiler (22). A high purity gaseous oxygen stream (36) is removed from above the sump of the sideleg column (23) and fed to the main heat exchanger (14) from where it is recovered as product (99.5% GOX). An oxygen-lean overhead vapour stream (37) is returned from the sideleg column (23) to the LP column (25) just above the sump thereof. Since liquid feed (35) for the sideleg column (23) is withdrawn from a location of the LP column (25) which is below the crude LOX feed (26'), the concentration of oxygen in the liquid feed (35) is greater than that in the crude LOX feed (26').

A low purity oxygen product stream (38) is withdrawn from the bottom of the LP column (25) and fed to the main heat exchanger (14) from where it is recovered as product (95% GOX).

In Figure 1, a separate booster (20) is provided to compress the third feed air stream (13) used to provide reboil in heat exchanger (22) to the bottom of the sideleg column (23). However, this feed air stream (13) could be boosted in the compressor end (16) of the compander system (17).

The embodiment of Figure 2 differs from that of Figure 1 in that the low purity oxygen stream (38) is withdrawn from the LP column (25) as a liquid stream and is boiled against a portion (215) of the first cooled feed air stream (11) in a LOX boil vaporizer (210); the reboiler (31) is relocated to an intermediate location in the LP column (25); and a further reboiler (211) fed by a cooled feed air stream (212) is located in the bottom of the LP column (25). Only the differences in the two systems will be described.

A minor portion of the cooled first feed air stream (11') is withdrawn as a substream (212) and is fed to the bottom reboiler (211) in the LP column (25). The condensed air (213) from this reboiler (211) is fed to an intermediate location of the HP column (15).

The remaining (larger) portion (215) of the cooled first feed air stream (11') is partially condensed in the LOX boil vaporizer (210) by heat exchange with the low purity liquid oxygen product (38) from the LP column (25). The resultant two-phase feed air stream (214) is then fed to the bottom of the HP column (15).

If required, the condensed air (213) can be fed to the HP column (15) at the same place as the two-phase air feed (214) instead of to the intermediate location. This arrangement is simpler but less efficient than feed to the midpoint.

Some or all of the liquified air from the air-fed reboilers (22, 211) can be cooled in subcooler (29) and fed to the LP column (25) instead of to the HP column (15).

At least the major portion of the HP gaseous nitrogen stream (27) from the top of the HP column is fed to the reboiler (31) which, relative to the system of Figure 1, has been relocated at an intermediate location in the LP column (25).

The low purity liquid oxygen stream (38) withdrawn from the sump of the LP column (25), increased in pressure slightly by its own static head, is fed to the LOX boil vaporizer (210). The oxygen stream (38) is boiled by the cooled first air stream (215) and then warmed in the main heat exchanger (14) for recovery as product (95% GOX). The LOX boil vaporizer (210) increases the pressure of the low purity oxygen vapour thus reducing compression power.

The embodiment of Figure 3 differs from that of Figure 2 in that the second feed air stream (12) is boosted in pressure by a compressor (310); the exhaust (311) from the expander end (19) of compander (17) is fed to the HP column (15) instead of to the LP column (25); additional air feed (312/313, 312/314) is provided to the LP column (25) and/or to the HP column (15); the non-reflux portion (33) of the condensed HP nitrogen stream (315) is withdrawn as eventually gaseous product (GAN) instead of being fed to the LP column (25); the LOX boil vaporizer is omitted; the high purity oxygen product (36) stream is withdrawn from the sideleg column (23) as liquid; the low purity oxygen stream (38), high purity oxygen stream (36) and nitrogen product stream (33) are pumped using liquid pumps (316, 317, 318) to generate pressurized oxygen and nitrogen products; and the LP column (25) is refluxed with a side stream (319) from the HP column (15). Only the differences in the two systems will be described.

A portion (215) of the cooled first feed air stream (11') remaining after withdrawal of the substream (212) is fed directly to the bottom of the HP column (15).

The second feed air stream (12) is compressed in a high pressure booster air compressor (310) and split into two substreams (312, 320). The larger of the substreams (312), containing the majority of the compressed air, is fed to the main heat exchanger (14) where it is condensed to vaporize the liquid products. As shown in Figure 3, part of the liquified air (314) can be fed to the HP column (15) and the remainder (313) cooled in the subcooler (29) and fed to the LP column (25). However, all of the liquified air could be fed to the HP column (15) or to the LP column (25).

The smaller substream (320) of the air from the booster compressor (310) is fed to the compressor end (16) of the compander (17) and, after partial cooling in the main heat exchanger (14) is fed to the expander end (19) of the compander (17) to generate refrigeration for the plant. The expander exhaust (311) is combined with the cooled first feed air stream portion (215) fed to the bottom of the HP column (15). The compander feed can be taken as a sidestream from an inter-stage of the booster compressor (310) instead of from the booster compressor discharge as shown in Figure 3.

It may be preferred to simply expand the second feed air substream (320) instead of companding it. In that case, the compander (17) is omitted and the partially cooled substream (320) is fed from the main heat exchanger (14) to an expander replacing the expander end (19) of the compander (17).

It also may be advantageous to use a common booster compressor to replace the two booster compressors (20, 310). In this case, the compressed third feed air stream (13) required to reboil the sideleg column (23) can be withdrawn as a sidestream from the common booster compressor or from the common booster compressor discharge product.

A side stream (319) of impure reflux is withdrawn from the HP column (15) several stages below the top, cooled in the subcooler (29) and fed (319') to the top of the LP column (25).

The HP nitrogen stream (27) withdrawn from the top of the HP column (15) is condensed in the intermediate reboiler (31) and the condensed HP nitrogen stream divided into a reflux substream (32) and a product stream (33). The product stream (33) is pumped by a liquid pump (318) prior to vaporization in the main heat exchanger (14) for collection as gaseous nitrogen product (GAN).

The low purity liquid oxygen stream (38) withdrawn from the sump of the LP column (25) also is pumped by a liquid pump (316) prior to vaporization in the main heat exchanger (14) for collection as low purity gaseous oxygen product (95% GOX).

Similarly, the high purity liquid oxygen stream (36) from the sideleg column (23) is pumped by liquid pump (317) prior to vaporization in the main heat exchanger (14) for collection as high purity gaseous oxygen product (99.5% GOX)

In the embodiment of Figure 3, it is not necessary to recover all of the products (GAN, 95% GOX, 99.5% GOX) via liquid pumps and vaporization in the main heat exchanger (14). Any combination of liquid and gaseous products from the columns is allowed.

The embodiment of Figure 4 is a simplification of that of Figure 2 in which the LOX boil vaporizer (210) is omitted and the (larger) portion (215) of the cooled first feed air stream (11') is fed directly to the bottom of the HP column (15). The low purity oxygen product stream (438) is withdrawn from the sump of the LP column (25) as gas instead of as liquid.

The embodiment of Figure 5 differs from that of Figure 2 in that the feed to and return from the sideleg column (23) is at a location of the LP column (25) above the intermediate reboiler (31) and an argon sidearm column (510) has been added. Only the differences in the two systems will be described.

In Figure 5, the feed stream (35) to the sideleg column (23), instead of being withdrawn from the sump of the LP column (25) as in Figure 2, is withdrawn from the middle of that column, above the intermediate reboiler (31). Alternatively, the feed stream (35) to the sideleg column (23) could be withdrawn at a location below the intermediate reboiler (31) but above the sump of the LP column (25). The vapour stream (37) from the top of the sideleg column (23) is returned preferably to this same point in the LP column (25). High purity oxygen product stream (36) is withdrawn from the bottom of the column (23) either as gas (as in Figure 2) or as liquid (as in Figure 3).

An argon rich vapour sidestream (511) is withdrawn from the middle of the sideleg column (23) at a point where there is low nitrogen content in the column vapour. The nitrogen concentration in the argon rich vapor sidestream (511) usually is less than 1%, preferably less than 0.5% and especially less than 100 ppm. The argon rich sidestream (511) is distilled further in a crude argon or sidearm column (510). The product from this column is mostly argon and can contain as much a 4% or as little as 1 ppm of oxygen, depending on the number of stages in the column. The sidestream (511) can be further purified if necessary with any suitable purifier. An argon depleted liquid stream (512) from the bottom of the sidearm column (510) is returned to the middle of the sideleg column (23) preferably at the same point where the vapour sidestream (511) was withdrawn. The argon at the top of the sidearm column (510) is condensed by boiling a portion (513) of the subcooled crude LOX in a reboiler (514). The vaporized crude LOX (crude GOX, 515) is fed to a suitable point in the LP column (25). A portion of the argon stream from the top of the sidearm column (510) is recovered as an argon product stream. Preferably, said argon product stream is a portion (520) of the condensed stream from the reboiler (514).

The embodiment of Figure 6 differs from that of Figure 3 in that the sideleg column (23) is decoupled from the LP column (25) and an argon sidearm column (510) has been added. Only the differences in the two systems will be described.

In Figure 6, the feed to the sideleg column, instead of being withdrawn from the LP column (25), is provided by feeding to the top of the sideleg column (23) a portion (610) of the subcooled nitrogen rich impure reflux (319) and feeding to an intermediate location of the sideleg column (23) a subcooled portion (611) of the crude LOX reflux (26) to the LP column (25).

The vapour stream (612) from the top of sideleg column (23) is oxygen depleted waste and is mixed with the oxygen depleted waste (28) from the top of the LP column (25).

High purity oxygen product (36) is withdrawn from the bottom of the sideleg column (23) either as liquid (as in Figure 3) or as gas (as in Figure 2).

An argon rich vapour sidestream (511) is withdrawn from the middle of the sideleg column (23) at a point where there is low nitrogen content in the column vapor. The nitrogen concentration in the argon rich vapor sidestream (511) usually is less than 1%, preferably less than 0.5% and especially less than 100 ppm. The argon rich sidestream (511) is distilled further in the sidearm column (510). The product from this column is mostly argon and can contain as much a 4% or as little as 1 ppm of oxygen, depending on the number of stages in the column. The sidestream (511) can be further purified if necessary with any suitable purifier. An argon depleted liquid stream (512) from the bottom of the sidearm column (510) is returned to the middle of the sideleg column (23) preferably at the same point where the vapour sidestream (511) was withdrawn. The argon at the top of the sidearm column (510) is condensed by boiling a portion (513) of the subcooled crude LOX in a reboiler (514). The vaporized crude LOX (crude GOX, 613) is fed to a suitable point in the sideleg column (23). A portion (520) of the condensed argon stream from the reboiler (514) is recovered as an argon product stream.

In some other variations of the process in Figure 6, a vapour stream can be withdrawn from a stage below the crude LOX feed (26) to the LP column (25) and fed to the sideleg column (23) at a location which is below the feed point of the vaporized crude LOX (613).

The embodiment of Figure 7 differs from that of Figure 6 in that the vaporized crude LOX (713) from the top of the sidearm column (510) is fed to the LP column (25) and a vapour stream (711) withdrawn from the LP column (25) from a location below this feed is fed to the sideleg column (23).

In all of the embodiments described above, it is possible to use nitrogen to reboil the sideleg column instead of air. For example, the nitrogen product stream (GAN) is compressed in the LP booster compressor (20) instead of the third feed air stream (13). After cooling to cryogenic temperatures in the main heat exchanger (14), the compressed nitrogen product stream is fed to the reboiler (22) in the sideleg column (23). The resultant condensed nitrogen can be fed to any suitable point of the HP column (15).

In all of the embodiments, the pressure of the supply air from the front end (1) is only as high as it needs to be to boil low purity oxygen. This is consistent with the low pressure of a dual-reboiler cycle. Only the portion of the air necessary to make the sideleg oxygen is compressed to the pressure required to boil high purity oxygen. This reduces the compression power over cycles where the entire air feed is used to make high purity oxygen and compared with the cycle of US-A-5,515,833.

The new cycle of the invention also permits of reduction in size of the LP column (25). If the sideleg column (23) is decoupled as in Figure 6, the overall diameter of the LP column (25) is smaller because it is not purifying the entire air stream. It also permits of reduction in the number of stages which must be of a large diameter, since the large main column (15, 25) no longer has to make high purity oxygen.

By taking the feed (511) to the sidearm column (510) from the midpoint of the sideleg column (23), this feed is rich in argon (typically 6-22%, preferably 9-15%, Ar). This not only simplifies and shortens the sidearm column over the cycle of US-A-5,515,833 which must start with a feed severely depleted in argon (typically less than 4% Ar), but provides much higher recoveries of argon.

## Claims

1. A process for cryogenic distillation of a compressed feed air stream (10) in a distillation system (15, 25) comprising a low purity column (25) producing a low purity (less than 97%) oxygen product stream (38) and a nitrogen-rich stream (28), said column (25) having a bottom reboiler (31; 211) in which a suitable first process stream (30; 212) is condensed to provide boilup to the column (25), characterized in that an oxygen-rich stream (35; 611) having an oxygen concentration at least equal to that of the feed (26') to the low purity column (25) is withdrawn from the distillation system (15, 25) and rectified in a high purity column (23) whose bottoms reboiler heat is provided by condensation of a suitable second process stream (21) to provide a high-purity (more than 97%) oxygen product stream (36), said second process stream (21) being at a higher pressure than said first process stream (30; 212).

2. A process as claimed in Claim 1, wherein said distillation system comprises a high pressure column (15) and a low pressure column (25) and wherein:

   (a) at least a portion (11) of the compressed air feed (10) is fed to the high pressure column (15) in which the air feed (11) is rectified into a high pressure nitrogen overhead (27) and a high pressure crude liquid oxygen bottoms (26);

   (b) at least a portion of the high pressure crude liquid oxygen bottoms (26) is fed to the low pressure column (25) in which the high pressure crude liquid oxygen bottoms (26) is rectified into a low pressure nitrogen overhead (28) and a low pressure liquid oxygen bottoms;

   (c) at least a portion (30) of the high pressure nitrogen overhead (27) is condensed (31) and at least a portion (32) of the condensed high pressure nitrogen overhead is returned to the high pressure column (15) as reflux;

   (d) at least a portion of the low pressure liquid oxygen bottoms is boiled by a bottom reboiler (31; 211) in which a suitable first process stream (30; 212) is condensed;

   (e) the low purity oxygen product stream (38) is withdrawn from the low pressure column (25);

   (f) the oxygen-rich stream (26; 35) having an oxygen concentration at least equal to that of the high pressure crude liquid oxygen bottoms (26) is withdrawn from the distillation system (15, 25) and fed to a high purity column (23) in which it is rectified into an oxygen-lean overhead vapour (37) and a high-purity liquid oxygen bottoms;

   (g) at least a portion of the high purity liquid oxygen bottoms is boiled by a bottom reboiler (22) by condensation of a suitable second process stream (21) compressed to a pressure higher than that of the first process stream (30; 212) boiling the low pressure column (15);

   (h) the high purity oxygen product stream (36) is withdrawn from the high purity column (23).

3. A process as claimed in Claim 2, wherein the process stream providing bottom reboil (22) to the high purity column (23) is a portion (21) of the compressed feed air (10) which has been further compressed (20).

4. A process as claimed in Claim 2, wherein the process stream providing bottom reboil (22) to the high purity column (23) is at least a portion of the high pressure nitrogen overhead (27) which has been further compressed (20).

5. A process as claimed in any one of Claims 2 to 4, wherein the oxygen-rich stream (35) is withdrawn from the low pressure column (25).

6. A process as claimed in Claim 5, wherein the oxygen-lean overhead vapour (37) is returned to the low pressure column (25) at substantially the same location as that at which the oxygen-rich stream (35) was withdrawn.

7. A process as claimed in Claim 5 or Claim 6, wherein the oxygen-rich stream (35) is withdrawn from the sump of the low pressure column (25).

**8.** A process as claimed in any one of Claims 2 to 4, wherein the oxygen-rich stream (611) is a portion of the high pressure crude liquid oxygen bottoms (26).

**9.** A process as claimed in Claim 8, wherein a vapour stream (711) is withdrawn from the low pressure column (25) at a location below the feed of the high pressure crude liquid oxygen bottoms (26) thereto and is fed to the high purity column (23) at a location below the feed of the oxygen-rich stream (611) thereto.

**10.** A process as claimed in any one of Claims 2 to 9, wherein the process stream providing bottom reboil (211) to the low pressure column is a portion (212) of the compressed feed air (10) and the condensation of at least a portion (30) of the high pressure nitrogen overhead (27) is in a reboiler (31) at an intermediate location in the low pressure column (25).

**11.** A process as claimed in Claim 10, wherein the oxygen-rich stream (35) is withdrawn from the low pressure column (25) at a location above said intermediate reboiler (31) and an argon rich vapour stream (511) withdrawn from an intermediate location of the high purity column (23) is separated in an argon column (510) to produce an argon product stream and a liquid argon-depleted stream (512) which is returned to the high purity column (23).

**12.** A process as claimed in any one of Claims 2 to 11 comprising a distillation system as defined in Claim 2, wherein all of the condensed high pressure nitrogen overhead (27) is fed (30) to the high pressure column as reflux and the low pressure column (25) is refluxed with a sidestream (319) withdrawn from the high pressure column (15).

**13.** A process as claimed in Claim 12, wherein a portion (610) of the nitrogen rich sidestream (319) is fed to the high purity column (23).

**14.** An apparatus for producing low purity oxygen and high purity oxygen products by the cryogenic distillation of a compressed air feed (10) by the process of Claim 1, said apparatus comprising:

(i) a distillation system (15, 25) for rectifying the compressed air feed (10) and comprising a low purity column (25) for producing a low purity (less than 97%) oxygen product stream (38) and a nitrogen-rich stream (28);
(ii) a bottom reboiler (31; 211) in said low purity column (25) for condensing a suitable first process stream to provide boilup to the low purity column (25);
(iii) means (27, 30; 11, 212) for supplying said first process stream to said reboiler; and
(iv) means (38) for withdrawing the low purity oxygen product stream from the apparatus,

characterized in that the apparatus further includes

(v) a high purity column (23) for rectifying an oxygen rich stream having an oxygen concentration at least equal to that of the feed (26') to the low purity column (25) to provide a high-purity (more than 97%) oxygen product stream;
(vi) means (35; 26, 611) for withdrawing said oxygen-rich stream from the distillation system (15, 25) and feeding it to the high purity column (23);
(vii) a bottom reboiler (22) in said high purity column (23) for condensing a suitable second process stream to provide boilup to the high purity column (23);
(viii) means (20, 21) for supplying said second process stream to said high purity column reboiler (22) at a higher pressure than said first process stream is supplied to the low purity column reboiler (31; 211); and
(ix) means (36) for withdrawing the high purity oxygen product stream from the apparatus.

**15.** An apparatus as claimed in Claim 14, wherein said distillation system comprises:

(1) a high pressure column (15) for rectifying at least a portion of the compressed air feed (10) into a high pressure nitrogen overhead and a high pressure crude liquid oxygen bottoms;
(2) a low pressure column (25) for rectifying the high pressure crude liquid oxygen bottoms into a low pressure nitrogen overhead and a low pressure liquid oxygen bottoms;
(3) means (26, 29) for feeding at least a portion of the high pressure crude liquid oxygen bottoms to the low pressure column (25);
(4) means (27, 31, 32) for condensing at least a portion of the high pressure nitrogen overhead and returning at least a portion of the condensed high pressure nitrogen overhead to the high pressure column (15) as reflux;
(5) a bottom reboiler (31; 211) in the low pressure column (25) for condensing a suitable first process stream to provide boilup to the low pressure column (25);
(6) means (27, 30; 11, 212) for supplying said first process stream to said reboiler (31, 211);
(7) means for withdrawing the low purity oxygen product stream (38) from the apparatus;
(8) a high purity column (23) for rectifying an oxygen rich stream into an oxygen-lean overhead vapour (37) and a high-purity liquid oxygen bottoms;
(9) means (26; 35) for withdrawing the oxygen-rich stream from the distillation system (15, 25) and feeding it to the high purity column (23);

(10) a bottom reboiler (22) in said high purity column (23) for condensing a suitable second process stream to provide boilup to the high purity column (23);

(11) means (20, 21) for supplying said second process stream to said high purity column reboiler (22) at a higher pressure than said first process stream is supplied to the low pressure column reboiler (31; 211); and

(12) means (36) for withdrawing the high purity oxygen product stream from the apparatus.

**16.** An apparatus as claimed in Claim 15 including the components required to carry out an individual process as claimed in any one of Claims 3 to 13.

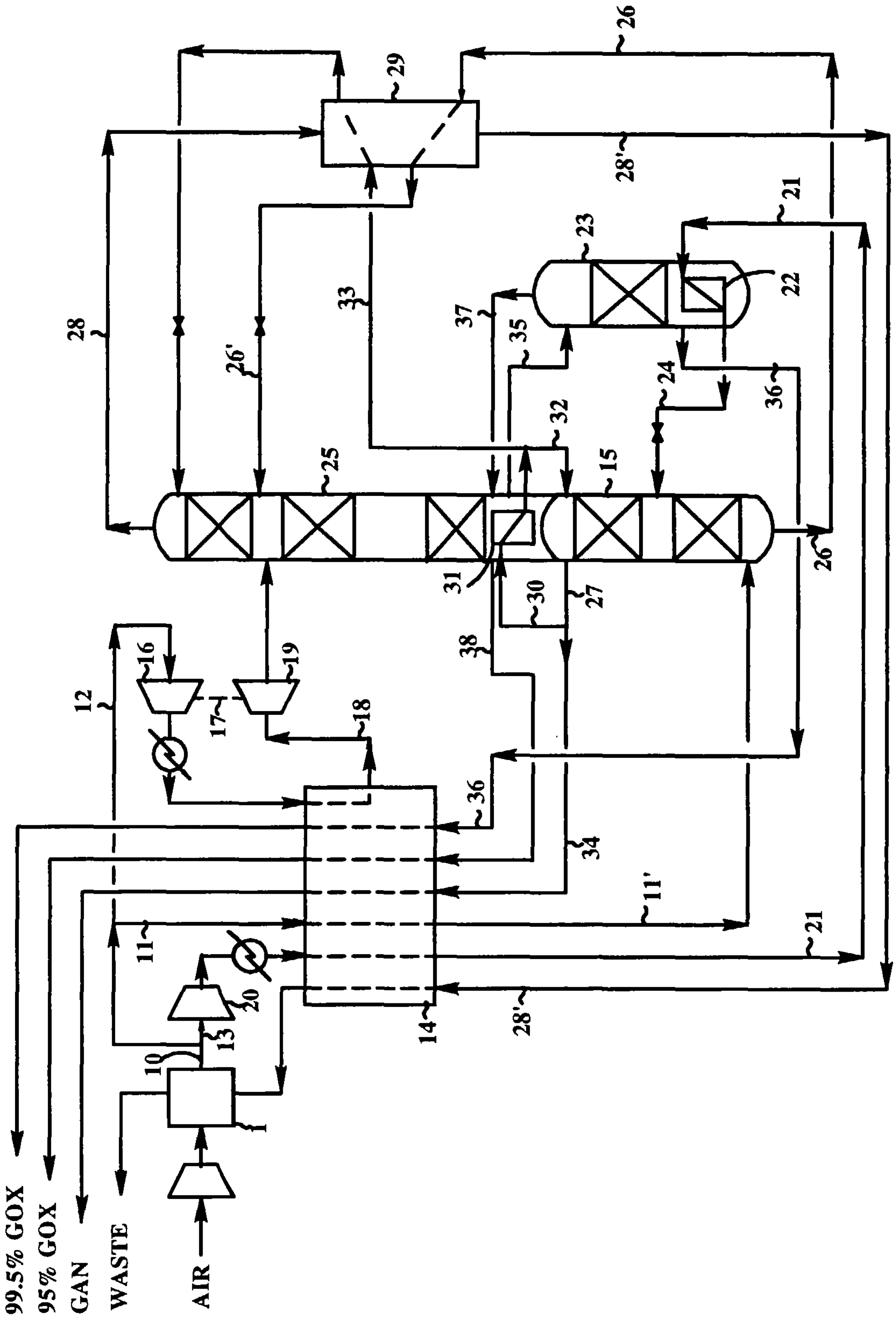
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Figure 1.

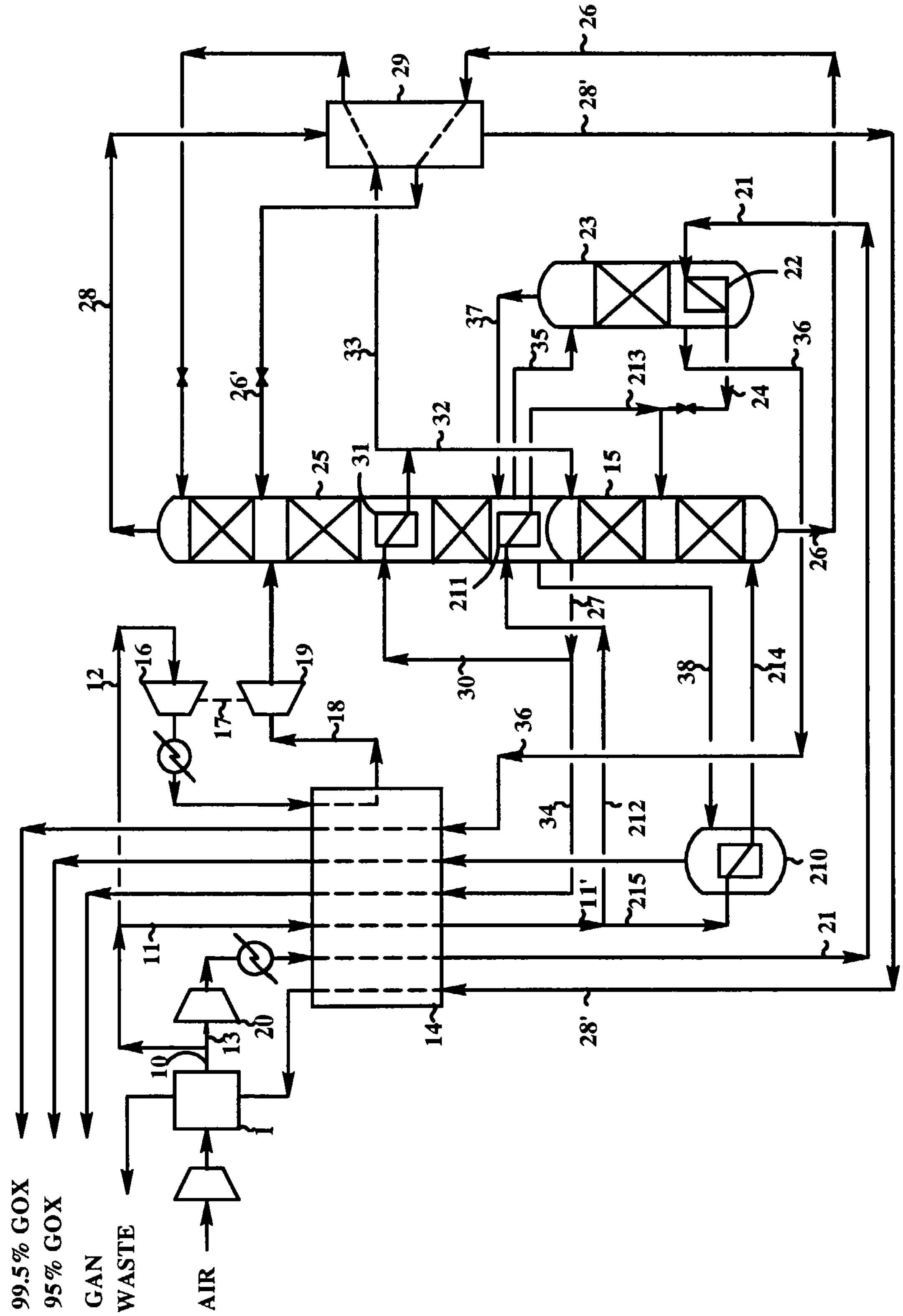


Figure 2.

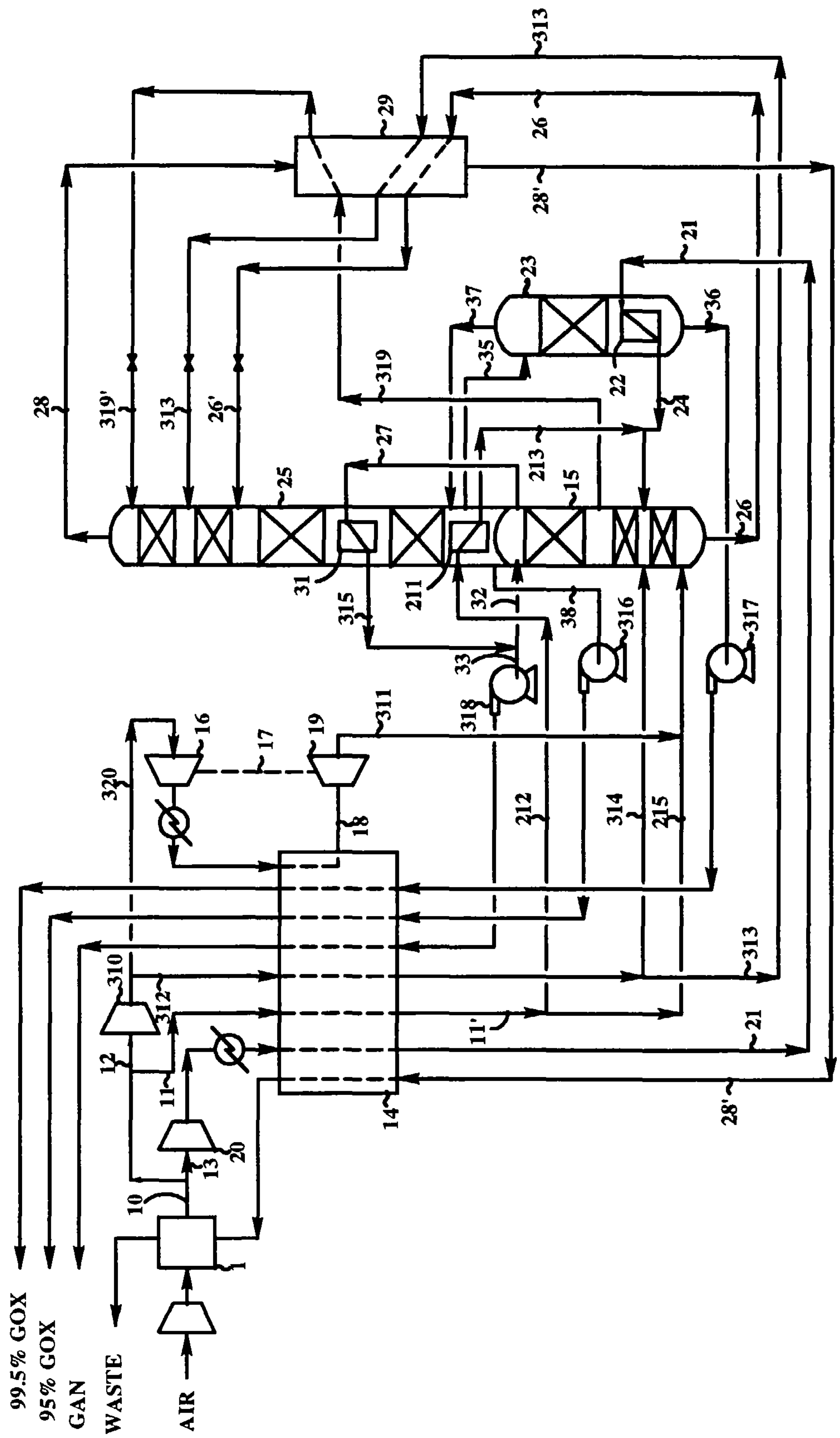


Figure 3.

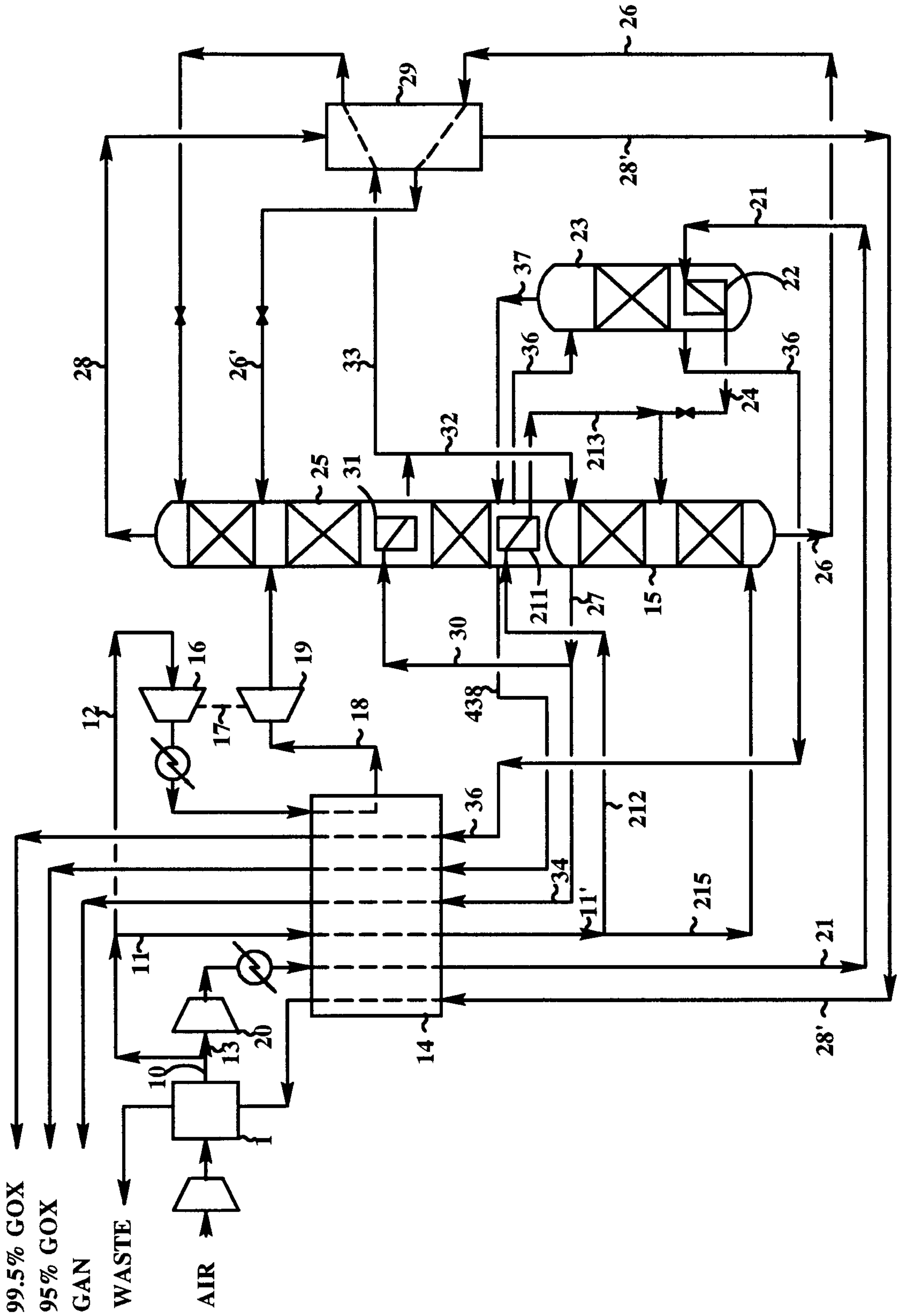
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Figure 4.

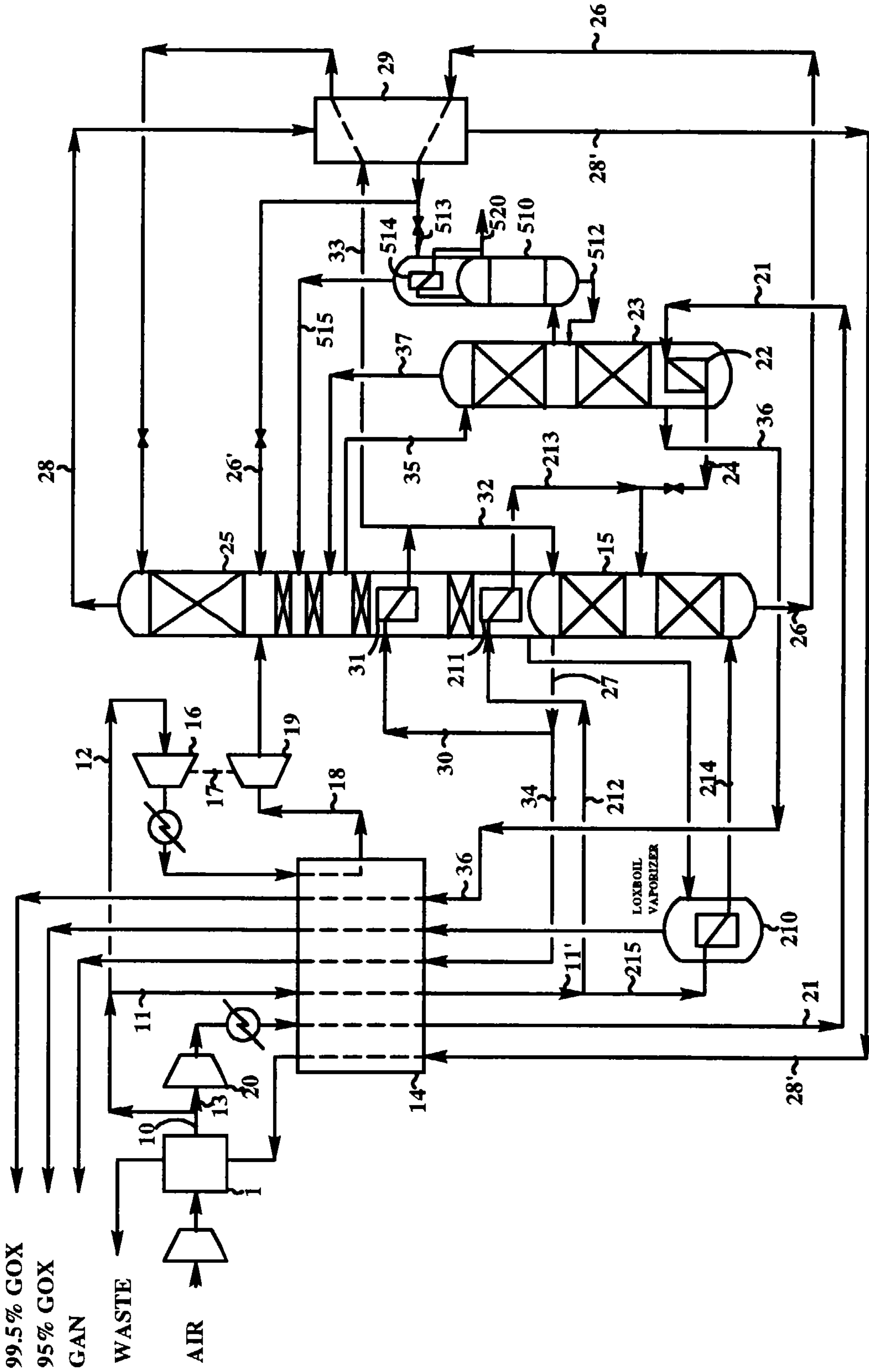
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Figure 5.

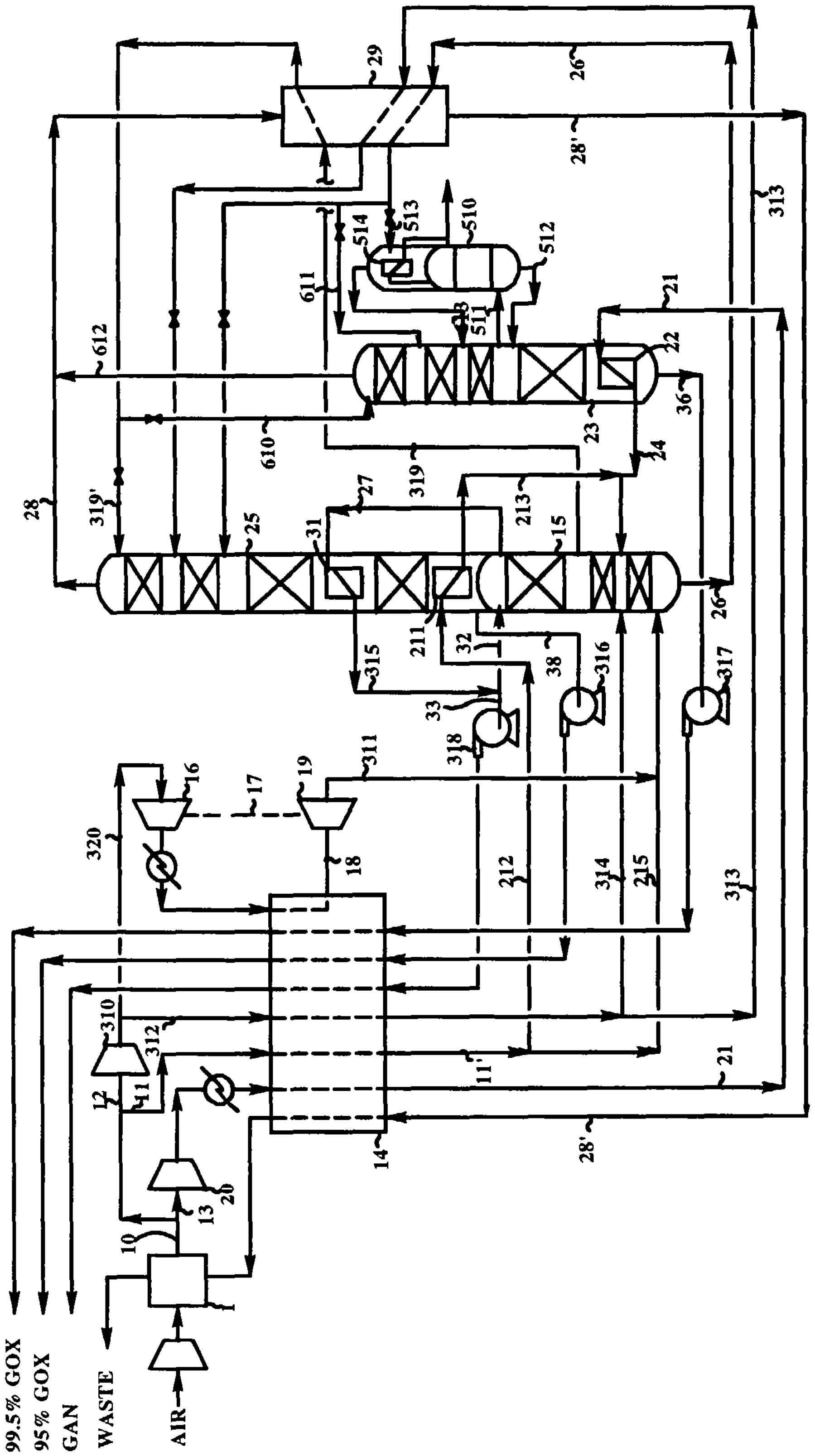


Figure 6.

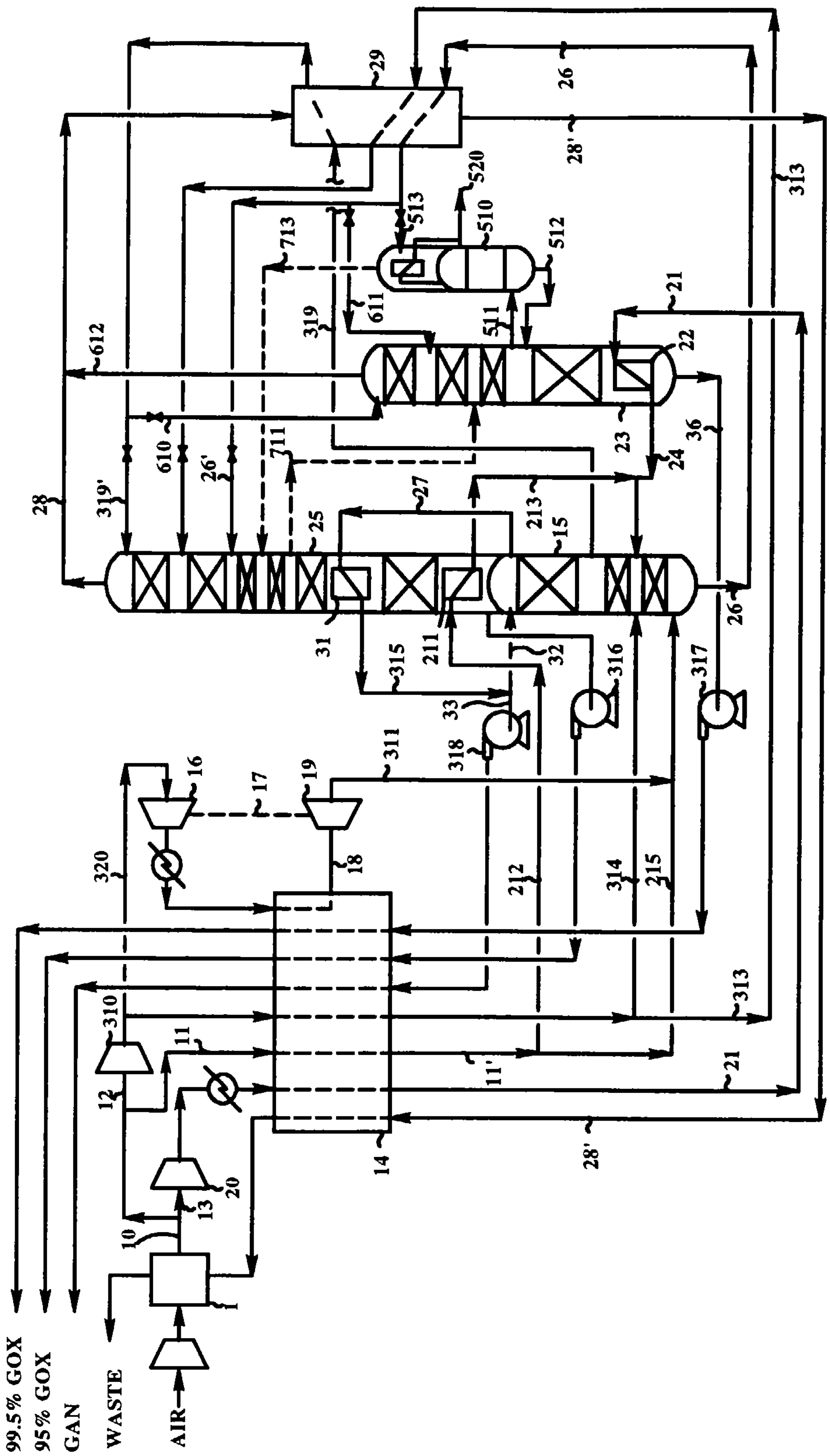


Figure 7.

European Patent Office

# EUROPEAN SEARCH REPORT

Application Number
EP 96 30 1423

## DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (Int.Cl.6) |
|---|---|---|---|
| X | GB-A-1 216 192 (BRITISH OXYGEN COMPANY) 16 December 1970<br>* claims; figure * | 1-5,8, 11,13-15 | F25J3/04 |
| X | EP-A-0 376 464 (BOC GROUP PLC) 4 July 1990<br>* claims; figure * | 1-6,9, 11,14-16 | |
| A | US-A-5 425 241 (AGRAWAL RAKESH ET AL) 20 June 1995 | | |
| A | EP-A-0 446 004 (AIR PROD & CHEM) 11 September 1991<br>* claims; figures 3-7,11-13 * | | |
| A | EP-A-0 538 117 (LIQUID AIR ENGINEERING) | | |

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

F25J

The present search report has been drawn up for all claims

| Place of search | Date of completion of the search | Examiner |
|---|---|---|
| THE HAGUE | 9 August 1996 | Meertens, J |

CATEGORY OF CITED DOCUMENTS

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