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(54) **Process to produce a krypton-xenon concentrate and a gaseous oxygen product.**

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FR-A- 2 113 843
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US-A- 3 768 270
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US-A- 4 421 536

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Description

This invention relates to the production of a krypton-xenon concentrate and is an improvement whereby the krypton-xenon concentrate is produced at high efficiency and a gaseous oxygen product substantially free of rare gases is also produced.

Krypton and xenon are undergoing increasing demand in a number of applications. Krypton is being widely used in high quality lighting including long-life light bulbs and automotive lamps. Xenon is being used for medical applications including special x-ray equipment. Both of these gases are commonly used in many laboratory and research applications.

The principle source of krypton and xenon is the atmosphere. Atmospheric air contains about 1.1 ppm (parts per million) of krypton and about 0.08 ppm of xenon. Generally, krypton and xenon are recovered from the air in conjunction with a comprehensive air separation process which separates air into oxygen and nitrogen.

Due to the lower vapor pressure of krypton and xenon, these gases concentrate in the oxygen rather than in the nitrogen during the air separation. The concentration of the atmospheric krypton and xenon in the oxygen increases their concentration by a factor of five because oxygen comprises only about one-fifth of the atmospheric air. It is desirable to further concentrate the krypton and xenon so that they may be effectively recovered in a rare gas recovery facility.

A process for the production of a krypton-xenon concentrate and the recovery of a gaseous product substantially free of rare gases, comprising:

(1) taking from an air separation plant a feed stream comprising oxygen, krypton and xenon and providing said feed stream to a reboiling zone;

(2) partially vaporizing a reboiling liquid to produce a vapor, and a liquid krypton-xenon concentrate;

(3) recovering krypton-xenon concentrate;

(4) introducing into a stripping column, reflux liquid having a krypton-xenon concentration less than that in said vapor;

(5) passing said vapor against the reflux liquid downflowing in the stripping column;

(6) stripping krypton and xenon from the vapor into the reflux liquid to produce a lean vapor and a richer liquid;

(7) passing the richer liquid to the reboiling zone to form part of the reboiling liquid;

(8) withdrawing lean vapor from the stripping column; and

(9) recovering withdrawn lean vapor as gaseous product substantially free of rare gases

is known from US-A 4 401 448. In this prior process a gaseous feed stream is taken from the air separation plant and passed to the reboiling zone.

In a similar prior process (US-A 3 768 270) the gaseous oxygen from the air separation plant, including krypton and xenon, likewise is fed as the feed stream to the reboiling zone, whereas liquid oxygen taken from the air separation plant is pumped into an adsorber for removal of acetylene, other higher hydrocarbons and carbon dioxide, thereafter is vaporized and then is combined with the oxygen taken in gaseous form from the air separation plant, for introduction into the reboiling zone.

Generally one wishes to produce gaseous oxygen from the air separation process. Therefore, in order to produce both gaseous oxygen product, and a further concentration of krypton and xenon, the entire amount of gaseous oxygen is passed through the concentrating process, i.e. is passed through the stripping column which must be relatively large. Furthermore, the oxygen passing through the stripping column is subject to pressure drop which adds to the costly compression if the oxygen product is desired at elevated pressure. This is costly from both a capital and operating cost standpoint.

In still another known process (GB-A 1 189 975) for obtaining a krypton-xenon mixture from air part of the oxygen is recovered directly from the low pressure column of a double column air separation process, whereas the liquid oxygen of the low pressure column, including krypton and xenon, is fed to the krypton/xenon concentration process. In the latter concentration process the oxygen is exchanged with argon in an exchange column. The argon is supplied from an argon section of the main air separation plant. This process has the disadvantage of tying the rare gas recovery with the notoriously sensitive argon section; often this results in a undesirable impact upon argon recovery.

It would be very desirable to have a krypton-xenon concentration process which produces gaseous oxygen but can employ a stripping column significantly smaller than heretofore considered necessary for conventional processes.

It is therefore an object of this invention to provide an improved process to produce a krypton-xenon concentrate.

It is another object of this invention to provide an improved process to produce a krypton-xenon concentrate while also producing a gaseous oxygen product substantially free of rare gases.

It is still another object of this invention to provide an improved process to produce a krypton-xenon concentrate and a gaseous oxygen product while employing a stripping column significantly smaller than employed by conventional processes.

The above and other objects which will become apparent to one skilled in the art upon a reading of this disclosure are attained by:

a process for the production of a krypton-xenon concentrate and the recovery of a gaseous product substantially free of rare gases, comprising:

(1) taking from an air separation plant a feed stream comprising oxygen, krypton and xenon and providing said feed stream to a reboiling zone;

(2) partially vaporizing a reboiling liquid to produce a vapor, and a liquid krypton-xenon concentrate;

(3) recovering krypton-xenon concentrate;

(4) introducing into a stripping column, reflux liq-

uid having a krypton-xenon concentration less than in said vapor;

(5) passing said vapor against the reflux liquid downflowing in the stripping column;

(6) stripping krypton and xenon from the vapor into the reflux liquid to produce a lean vapor and a richer liquid;

(7) passing the richer liquid to the reboiling zone to form part of the reboiling liquid;

(8) withdrawing lean vapor from the stripping column; and

(9) recovering withdrawn lean vapor as gaseous product substantially free of rare gases;

which process is characterized in that said feed stream is taken from said air separation plant as a stream of feed liquid which forms a further part of said reboiling liquid in said reboiling zone; and

that the major portion of the gaseous oxygen product is directly recovered from said air separation plant.

As used herein, the term "rare gas" means krypton and xenon.

As used herein, the terms "lean", "leaner", "rich" and "richer", refer to the concentration of rare gases, unless specifically indicated otherwise.

As used herein the term "reboiling zone" means a heat exchange zone where entering liquid is indirectly heated and thereby partially vaporized to produce gas and remaining liquid. The remaining liquid is thereby enriched in the less volatile components present in the entering liquid.

As used herein, the term "indirect heat exchange" means the bringing of two fluid streams into heat exchange relation without any physical contact or intermixing of the fluids with each other.

As used herein, the term "equilibrium stage" means a vapor-liquid contacting stage whereby the vapor and liquid leaving that stage are in mass transfer equilibrium. For a separation column that uses trays or plates, i.e. separate and discrete contacting stages for the liquid and gas phases, an equilibrium stage would correspond to a theoretical tray or plate. For a separation column that uses packing, i.e. continuous contacting of the liquid and gas phases, an equilibrium stage would correspond to that height of column packing equivalent to one theoretical plate. An actual contacting stage, i.e. trays, plates, or packing, would have a correspondence to an equilibrium stage dependent on its mass transfer efficiency.

As used herein, the term "column" means a distillation or fractionation column, i.e., a contacting column or zone wherein liquid and vapor phases are countercurrently contacted to effect separation of a fluid mixture, as for example, by contacting of the vapor and liquid phases on a series of vertically spaced trays or plates mounted within the column or alternatively, on packing elements with which the column is filled. For an expanded discussion of fractionation columns see the Chemical Engineer's Handbook, Fifth Edition, edited by R.H. Perry and C.H. Chilton, McGraw-Hill Book Company, New York Section 13, "Distillation" B.D. Smith et al, page 13–3, The Continuous Distillation Process.

The term "double column" is used herein to mean a high pressure column having its upper end in heat exchange relation with the lower end of a low pressure column. An expanded discussion of double columns appears in Ruheman. "The Separation of Gases" Oxford University Press, 1949, Chapter VII, Commercial Air Separation, and Barron, "Cryogenic Systems", McGraw-Hill, Inc., 1966, p. 230, Air Separation Systems.

The single Figure is a schematic flow diagram of one preferred embodiment of the process of this invention. The schematic representation of the Figure is particularly preferred in that it illustrates a case where the feed to the krypton-xenon concentration process comes from a double-column air separation plant and the feed is taken from the air separation plant so as to have an increased krypton-xenon concentration over that which would conventionally be attained in oxygen.

Detailed Description

The process of this invention will be described in detail with reference to the drawing.

Referring now to the single Figure, cooled pressurized feed air 12, which has been cleaned of high boiling impurities such as carbon dioxide and water vapor, is introduced into higher pressure column 19, operating at a pressure in the range of from 5.2 to 20.7 bar (75 to 300 psia), preferably from 5.2 to 10.3 bar (75 to 150 psia). The cooling and cleaning steps, and other steps such as heat exchange with return streams, are not illustrated in the Figure since such process steps are well-known conventional steps and do not form part of this invention.

Within higher pressure column 19, the feed air is preseparated into a nitrogen-rich vapor 23 and an oxygen-enriched liquid 20. Liquid 20 is expanded through valve 21 and introduced as feed 22 into lower pressure column 17 which is operating at a pressure in the range of from 1.03 to 6.9 bar (15 to 100 psia), preferably from 1.03 to 2.1 bar (15 to 30 psia).

Nitrogen-rich vapor 23 is passed 24 to condenser 18 wherein it is condensed by indirect heat exchange with reboiling liquid from the bottom of lower pressure column 17. The resulting condensed nitrogen-rich stream 60 is divided into stream 26 which is expanded through valve 30 and passed as stream 31 into column 17 as liquid reflux, and into stream 27 which is passed into column 19 as liquid reflux.

The Figure also illustrates low pressure feed air stream 13 to column 17 which may be available from the warm end of the air separation process as obtained from development of plant refrigeration. Within column 17 the various input streams are separated by cryogenic rectification to produce nitrogen stream 14 and oxygen product. The nitrogen stream 14 may be recovered in whole or in part, or may be released to the atmosphere.

As indicated previously, virtually all of the krypton and xenon in the feed air will concentrate in the oxygen rather than in the nitrogen. As illustrated in the Figure the krypton and xenon in the oxygen are further concentrated in a liquid oxygen portion en-

abling the recovery of a major portion of the oxygen as gaseous oxygen product, relatively free of rare gases, directly from column 17. This is accomplished by removing gaseous oxygen from column 17 as stream 37 above at least 1 and preferably at least 2 equilibrium stages or actual trays above the sump of column 17 wherein bottoms are reboiled against condensing nitrogen in condenser 18. In the Figure, tray 32 is the bottom tray, tray 33 is the next higher tray, and tray 34 is the third tray in this order. As can be seen oxygen product stream 37 is taken from between trays 33 and 34. In this way, because krypton and xenon both have lower vapor pressures than oxygen, the bulk of the krypton and xenon remains in liquid oxygen and is carried down into the sump, leaving stream 37 relatively free of rare gases.

As indicated, the major portion of the krypton and xenon in the feed air is contained in the liquid in the sump of column 17. This liquid is an ideal source of a feed to the krypton-xenon cencentration process of this invention.

Referring again to the Figure, liquid stream 36 containing oxygen, krypton and xenon is provided to reboiling zone 44 to form reboiling liquid 61. Reboiling zone 44 may be separate from or may be within stripping column 38. The concentration of krypton and xenon in the feed liquid such as stream 36 may be any effective concentration, but, in general, the concentration of krypton will be at least 10 ppm and preferably at least 20 ppm, and the concentration of xenon will be at least 1 ppm, preferably at least 2 ppm, in the liquid feed stream.

In reboiling zone 44, the liquid 61 is partially vaporized to produce a vapor, which has a lower rare gas content than the remaining liquid. The vapor 41 is passed to stripping column 38 for upflow through the column. The remaining liquid with its relatively high krypton and xenon content is withdrawn as the liquid concentrate product 16 containing the rare gases. Typically the krypton concentration in concentrate 16 is at least 200 ppm and preferably is at least 400 ppm, and the xenon concentration in concentrate 16 is at least 15 ppm and preferably is at least 30 ppm.

As illustrated in the Figure high pressure nitrogen-rich vapor from an associated double-column air separation plant is employed to carry out the partial vaporization in the reboiling zone. Referring to the Figure, a portion 25 of nitrogen-rich vapor 23 is passed to reboiler condenser 43 wherein it is condensed by indirect heat exchange with partially vaporizing reboiling liquid 61. The resulting condensed nitrogen stream 28 is passed to column 19 as liquid reflux. Conveniently, stream 28 may be combined with liquid nitrogen from main condenser 18 to form combined stream 29 for passage into column 19.

Stripping column 38 operates at a pressure within the range of from 1.03 to 6.9 bar (15 to 100 psia), preferably from 1.03 to 2.1 bar (15 to 30 psia), and serves to strip a significant portion, and preferably substantially all, of the krypton and xenon in vapor 41 into downflowing liquid. The entering downflowing stripping liquid must have a krypton-xenon concentration less than that of vapor 41 and preferably the krypton-xenon concentration in this reflux liquid when it enters the column is less than about 3 ppm. A convenient source for the reflux or stripping liquid is the double column air separation plant. As illustrated in the Figure a liquid stream 35 is taken from above the point where gaseous oxygen product stream 37 is taken. In this way the liquid stream 35 has the low krypton-xenon concentration.

Within column 38 vapor 41 is passed against downflowing liquid 35 and krypton and xenon from vapor 41 are stripped into the downflowing liquid. The resulting richer liquid 39 is passed to reboiling zone 44 to form part of the reboiling liquid 61. The Figure illustrates a convenient arrangement wherein richer liquid 39 is combined with feed liquid 36 to form liquid 40 and this combined liquid is passed to reboiling zone 44 to form reboiling liquid 61.

The lean vapor which results from the stripping operation is withdrawn from column 38 as stream 42 and recovered as gaseous product substantially free of rare gases. The Figure illustrates a convenient arrangement wherein lean vapor 42 is combined with gaseous oxygen product 37 from the air separation process and the resulting combined stream 15 is recovered as gaseous oxygen product.

By passing the feed to the krypton-xenon concentration process directly to the reboiling zone rather than to the stripping column, and by carrying out the stripping process in the defined manner of this invention wherein only the vapor from the reboiling zone is passed through the stripping column, one is able to produce a krypton-xenon concentrate and a gaseous rare gas-free oxygen product employing a stripping column of considerably smaller size than is required for conventional krypton-xenon concentration processes. Typically for this process arrangement, the liquid feeds to the stripping column, i.e. streams 35 and 36, will be about 20 percent of the oxygen product 15 from the plant. Accordingly, the stripping column then handles vapor flow 42 which is about one-fifth that of the conventional rare gas recovery process and thereby requires about one-fifth the cross-sectional flow area of the conventional flow area of a conventional oxygen gas stripping column.

The greater part of the oxygen from the air separation plant bypasses the krypton-xenon process entirely thus reducing markedly the throughput and thus the size requirements of the stripping column. Generally the liquid stream to the reboiling zone contains from about 5 to 40 percent of the oxygen from the air separation plant, and preferably about 20 percent. Another advantage is that the majority of the oxygen gas 37 is maintained at the pressure level of low pressure column 17. The portion of the oxygen product 42 that must be processed in the stripping column can be returned at equivalent pressure by operating the stripping column at a slightly higher pressure level to compensate for the column pressure drops. The higher pressure level can be easily obtained by reducing the elevation of the stripping column and utilizing the hydrostatic liquid height for the two liquid feeds.

A further advantage of this process is that the liquid draw from the lower pressure column sump

serves to avoid buildup of hydrocarbons in that column.

In Table I there are tabulated the results of a computer simulation of the process of this invention carried out in accord with the embodiment illustrated in the Figure. The data is presented for illustrative purposes and is not intended to be limiting. The flow is indicated as measured at ambient temperature (21°C or 70°F) and atmospheric pressure. The purity is defined in mole percent unless parts per million volume (ppm) is specified. The stream numbers correspond to those of the Figure.

Table 1

Stream No.	37	36	35	39
Flow, m^3/h	22.7	5.2	0.91	0.91
(cfh	800	185	32	32)
Temperature, °K	95	95	95	95
Pressure, bar	1.59	1.59	1.59	1.59
(psia	23	23	23	23)
Purity				
Oxygen, %	99.5	99.6	99.3	99.5
Krypton, ppm	0.3	39.1	2.5	339
Xenon, ppm	–	2.5	–	–
Other, %	0.5	0.4	0.7	0.5

Stream No.	16	42	15	41
Flow, m^3/h	0.48	5.7	28.3	5.7
(cfh	17	200	1000	200)
Temperature, °K	95	95	95	95
Pressure, bar	1.59	1.59	1.59	1.59
(psia	23	23	23	23)
Purity				
Oxygen, %	99.6	99.5	99.5	99.5
Krypton, ppm	427	1	–	55
Xenon, ppm	27	–	–	0.2
Other, %	0.4	0.4	0.5	0.5

As demonstrated by the data in Table I, the process of this invention effectively produces a krypton-xenon concentrate and substantially rare gas-free gaseous oxygen while requiring only a small flowrate for the feed to the concentration process. This significantly reduces both the capital and operating costs of the concentration process.

Claims

1. A process for the production of a krypton-xenon concentrate and the recovery of a gaseous product substantially free of rare gases, comprising:

(1) taking from an air separation plant (17, 19) a feed stream (36) comprising oxygen, krypton and xenon and providing said feed stream to a reboiling zone (44);

(2) partially vaporizing a reboiling liquid (16) to produce a vapor (41), and a liquid krypton-xenon concentrate (16);

(3) recovering krypton-xenon concentrate (16);

(4) introducing into a stripping column (38), reflux liquid (35) having a krypton-xenon concentration less than that in said vapor (41);

(5) passing said vapor (41) against the reflux liquid (35) downflowing in the stripping column (38);

(6) stripping krypton and xenon from the vapor (41) into the reflux liquid (35) to produce a lean vapor (42) and a richer liquid (39);

(7) passing the richer liquid (39) to the reboiling zone (44) to form part of the reboiling liquid (61);

(8) withdrawing lean vapor (42) from the stripping column (38); and

(9) recovering withdrawn lean vapor (42) as gaseous product substantially free of rare gases;

characterized in that said feed stream (36) is taken from said air separation plant (17, 19) as a stream of feed liquid which forms a further part of said reboiling liquid (61) in said reboiling zone (44); and

that the major portion (37) of the gaseous oxygen product (15) is directly recovered from said air separation plant (17, 19).

2. The process of claim 1 wherein said liquid feed stream (36) comprises from about 5 to 40 percent of the oxygen from the air separation plant.

3. The process of claim 1 or 2 wherein the krypton concentration in the liquid feed stream (36) is at least 10 ppm.

4. The process of any one of the preceding claims wherein the richer liquid (39) from the stripping column (38) is combined with feed liquid (36) prior to passage to the reboiling zone (44).

5. The process of any one of the preceding claims wherein the stripping column (38) operates at a pressure in the range of from 1.0 to 6.9 bar (15 to 100 psia).

6. The process of any one of the preceding claims wherein the concentration of krypton in the krypton-xenon concentrate (16) is at least 200 ppm.

7. The process of any one of the preceding claims wherein the feed liquid (36) is taken from the area of heat exchange relation of a double column air separation process.

8. The process of claim 7 wherein the reflux liquid (35) for the stripping column (38) is provided from the lower pressure column (17) of the double column process and is taken from a point above the point from where the feed liquid (36) is taken.

9. The process of claim 7 or 8 wherein said major portion (37) of the gaseous oxygen product (15) is removed from the lower pressure column (17).

10. The process of claim 9 wherein said major portion (37) of the gaseous oxygen product (15) is removed from the lower pressure column (17) at a point between the points from where the feed liquid (36) and the reflux liquid (35) are respectively taken.

11. The process of any one of claims 1 to 6 wherein a cryogenic air separation plant (17, 19) comprising a higher pressure column (19) and a lower pressure column (17) in heat exchange relation is used; said feed liquid stream (36) is withdrawn from the area of

heat exchange relation of said higher and lower pressure columns (17, 19); the reflux liquid (35) for said stripping column (38) is withdrawn from said lower pressure column (17) at a point above the point where said feed liquid stream (36) is withdrawn; and said major portion (37) of said gaseous oxygen product (15) is withdrawn from said lower pressure column at a point between the points from where said feed liquid stream (36) and said reflux liquid (35) are withdrawn.

12. The process of claim 8 or 11 wherein the reflux liquid (35) is withdrawn from the lower pressure column (17) at least two equilibrium stages above the area of heat exchange relation.

13. The process of any one of claims 7 to 12 wherein the partial vaporization of the reboiling liquid (61) is carried out by indirect heat exchange with condensing nitrogen rich vapor (25) taken from the higher pressure column (19).

14. The process of claim 13 wherein the resulting condensed nitrogen-rich stream (28) is returned to the higher pressure column (19) as liquid reflux.

15. The process of any one of the preceding claims wherein the with-drawn lean vapor (42) and the major portion (37) of the gaseous oxygen product (15) are combined and recovered together.

Patentansprüche

1. Verfahren zur Herstellung eines Krypton-Xenon-Konzentrats und zur Gewinnung eines von Edelgasen im wesentlichen freien gasförmigen Produkts, bei dem:

(1) ein Sauerstoff, Krypton und Xenon enthaltender Einsatzstrom (36) einer Luftzerlegungsanlage (17, 19) entnommen und einer Reboilerzone (44) zugeführt wird;

(2) eine Reboilerflüssigkeit (61) unter Bildung eines Dampfes (41) und eines flüssigen Krypton-Xenon-Konzentrats (16) teilverdampft wird;

(3) Krypton-Xenon-Konzentrat (16) gewonnen wird;

(4) in eine Stripperkolonne (38) Rücklaufflüssigkeit (35) mit einer Krypton-Xenon-Konzentration eingeleitet wird, die kleiner als die in dem Dampf (41) ist;

(5) der Dampf (41) im Gegenstrom zu der Rücklaufflüssigkeit (35) geleitet wird, die in der Stripperkolonne (38) nach unten strömt;

(6) Krypton und Xenon von dem Dampf (41) in die Rücklaufflüssigkeit (35) abgestreift werden, um einen mageren Dampf (42) und eine reichere Flüssigkeit (39) zu erzeugen;

(7) die reichere Flüssigkeit (39) der Reboilerzone (44) zugeleitet wird, um einen Teil der Reboilerflüssigkeit (61) zu bilden;

(8) magerer Dampf (42) aus der Stripperkolonne (38) abgezogen wird; und

(9) abgezogener magerer Dampf (42) als von Edelgasen im wesentlichen freies gasförmiges Produkt gewonnen wird;

dadurch gekennzeichnet, daß der Einsatzstrom (36) der Luftzerlegungsanlage (17, 19) als ein Einsatzflüssigkeitsstrom entnommen wird, der einen weiteren Teil der Reboilerflüssigkeit (61) in der Reboilerzone (44) bildet; und

daß der größere Teil (37) des gasförmigen Sauerstoffprodukts (15) unmittelbar von der Luftzerlegungsanlage (17, 19) gewonnen wird.

2. Verfahren nach Anspruch 1, wobei der flüssige Einsatzstrom (36) etwa 5 bis 40 Prozent des Sauerstoffs von der Luftzerlegungsanlage ausmacht.

3. Verfahren nach Anspruch 1 oder 2, wobei die Kryptonkonzentration in dem flüssigen Einsatzstrom (36) mindestens 10 ppm beträgt.

4. Verfahren nach einem der vorhergehenden Ansprüche, wobei die reichere Flüssigkeit (39) von der Stripperkolonne (38) mit der Einsatzflüssigkeit (36) vor dem Zuleiten zu der Reboilerzone (44) kombiniert wird.

5. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Stripperkolonne (38) bei einem Druck im Bereich von 1,0 bis 6,9 bar (15 bis 100 psia) arbeitet.

6. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Konzentration an Krypton in dem Krypton-Xenon-Konzentrat (16) mindestens 200 ppm beträgt.

7. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Einsatzflüssigkeit (36) aus dem Bereich des Wärmeaustauschs eines Doppelkolonnen-Luftzerlegungsprozesses entnommen wird.

8. Verfahren nach Anspruch 7, wobei die Rücklaufflüssigkeit (35) für die Stripperkolonne (38) von der Niederdruckkolonne (17) des Doppelkolonnenverfahrens angeliefert und von einer Stelle entnommen wird, die über der Entnahmestelle der Einsatzflüssigkeit (36) liegt.

9. Verfahren nach Anspruch 7 oder 8, wobei der größere Teil (37) des gasförmigen Sauerstoffprodukts (15) von der Niederdruckkolonne (17) entnommen wird.

10. Verfahren nach Anspruch 9, wobei der größere Teil (37) des gasförmigen Sauerstoffprodukts (15) von der Niederdruckkolonne (17) an einer Stelle entnommen wird, die zwischen den Entnahmestellen der Einsatzflüssigkeit (36) und der Rücklaufflüssigkeit (35) liegt.

11. Verfahren nach einem der Ansprüche 1 bis 6, wobei eine Tieftemperatur-Luftzerlegungsanlage (17, 19) verwendet wird, die eine Hochdruckkolonne (19) und eine Niederdruckkolonne (17) aufweist, die in Wärmeaustauschbeziehung stehen; der Einsatzflüssigkeitsstrom (36) aus dem Wärmeaustauschbereich der Hoch- und Niederdruckkolonnen (17, 19) abgezogen wird; die Rücklaufflüssigkeit (35) für die Stripperkolonne (38) aus der Niederdruckkolonne (17) an einer Stelle abgezogen wird, die über der Entnahmestelle des Einsatzflüssigkeitsstroms (36) liegt; und der größere Teil (37) des gasförmigen Sauerstoffprodukts (15) aus der Niederdruckkolonne an einer Stelle abgezogen wird, die zwischen den Entnahmestellen für den Einsatzflüssigkeitsstrom (36) und die Rücklaufflüssigkeit (35) liegt.

12. Verfahren nach Anspruch 8 oder 11, wobei die Rücklaufflüssigkeit (35) aus der Niederdruckkolonne (17) mindestens zwei Gleichgewichtsstufen oberhalb des Wärmeaustauschbereichs abgezogen wird.

13. Verfahren nach einem der Ansprüche 7 bis 12, wobei die Teilverdampfung der Reboilerflüssigkeit (61) durch indirekten Wärmeaustausch mit kondensierendem stickstoffreichem Dampf (25) durchgeführt wird, der aus der Hochdruckkolonne (19) entnommen wird.

14. Verfahren nach Anspruch 13, wobei der erhaltene kondensierte stickstoffreiche Strom (28) zu der Hochdruckkolonne (19) als flüssiger Rücklauf zurückgeleitet wird.

15. Verfahren nach einem der vorhergehenden Ansprüche, wobei der abgezogene magere Dampf (42) und der größere Teil (37) des gasförmigen Sauerstoffprodukts (15) kombiniert und zusammen gewonnen werden.

Revendications

1. Procédé de production d'un concentré de krypton-xénon et de séparation d'un produit gazeux pratiquement dépourvu de gaz rares, qui comprend des étapes consistant:

(1) à prélever dans une installation (17, 19) de fractionnement d'air un courant d'alimentation (36) comprenant de l'oxygène, du krypton et du xénon et à introduire ce courant d'alimentation dans une zone de réébullition (44);

(2) à vaporiser partiellement un liquide en réébullition (61) pour produire une vapeur (41) et un concentré liquide krypton-xénon (16);

(3) à recueillir le concentré de krypton-xénon (16);

(4) à introduire dans une colonne de rectification (38) un liquide de reflux (35) ayant une concentration en krypton-xénon plus faible que dans ladite vapeur (41);

(5) à faire descendre la vapeur (41) à contre-courant avec le liquide de reflux (35) dans la colonne de rectification (38);

(6) à entraîner le krypton et le xénon de la vapeur (41) dans le liquide de reflux (35) pour produire une vapeur pauvre (42) et un liquide enrichi (39);

(7) à transférer le liquide enrichi (39) dans la zone de réébullition (44) pour former une partie du liquide de réébullition (61);

(8) à évacuer de la vapeur pauvre (42) de la colonne de rectification (38); et

(9) à recueillir la vapeur pauvre (42) évacuée comme produit gazeux pratiquement dépourvu de gaz rares; caractérisé en ce que ledit courant d'alimentation (36) est prélevé dans l'installation (17, 19) de fractionnement d'air comme courant de liquide d'alimentation qui forme une autre partie du liquide de réébullition (61) dans ladite zone de réébullition (44); et en ce que

la majeure partie (37) de l'oxygène gazeux constituant le produit (15) est directement recueillie à la sortie de ladite installation (17, 19) de fractionnement d'air.

2. Procédé suivant la revendication 1, dans lequel le courant liquide d'alimentation (36) comprend environ 5 à 40% de l'oxygène venant de l'installation de fractionnement d'air.

3. Procédé suivant la revendication 1 ou 2, dans lequel la concentration en krypton dans le courant liquide d'alimentation (36) est d'au moins 10 ppm.

4. Procédé suivant l'une quelconque des revendications précédentes, dans lequel le liquide enrichi (39) sortant de la colonne de rectification (38) rejoint le liquide d'alimentation (36) avant le transfert dans la zone de réébullition (44).

5. Procédé suivant l'une quelconque des revendications précédentes, dans lequel la colonne de rectification (38) fonctionne à une pression absolue comprise dans l'intervalle de 1,0 à 6,9 bars (15 à 100 lb/in^2).

6. Procédé suivant l'une quelconque des revendications précédentes, dans lequel la concentration du krypton dans le concentré de krypton-xénon (16) est d'au moins 200 ppm.

7. Procédé suivant l'une quelconque des revendications précédentes, dans lequel le liquide d'alimentation (36) est prélevé dans la zone de relation d'échange de chaleur d'un procédé de fractionnement d'air à colonne double.

8. Procédé suivant la revendication 7, dans lequel le liquide de reflux (35) destiné à la colonne d'entraînement (38) provient de la colonne (17) à basse pression du procédé à colonne double et est prélevé en un point situé au-dessus du point de prélèvement du liquide d'alimentation (36).

9. Procédé suivant la revendication 7 ou 8, dans lequel la portion principale (37) du produit (15) constitué d'oxygène gazeux est déchargée de la colonne (17) à basse pression.

10. Procédé suivant la revendication 9, dans lequel la portion principale (37) du produit (15) constitué d'oxygène gazeux est déchargée de la colonne (17) à basse pression en un point situé entre les points où le liquide d'alimentation (36) et le liquide de reflux (35) sont respectivement prélevés.

11. Procédé suivant l'une quelconque des revendications 1 à 6, dans lequel une installation cryogénique (17, 19) de fractionnement d'air comprenant une colonne à haute pression (19) et une colonne à basse pression (17) en relation d'échange de chaleur est utilisée; ledit courant liquide d'alimentation (36) es soutiré de la zone de relation d'échange de chaleur desdites colonnes à haute et basse pressions (17, 19); le liquide de reflux (35) destiné à la colonne de rectification (38) est soutiré de la colonne (17) à basse pression en un point situé au-dessus du point de soutirage du courant liquide d'alimentation (36); et la portion principale (37) du produit (15) constitué d'oxygène gazeux est soutirée de la colonne à basse pression en un point situé entre les points de soutirage du courant liquide d'alimentation (36) et du liquide de reflux (35).

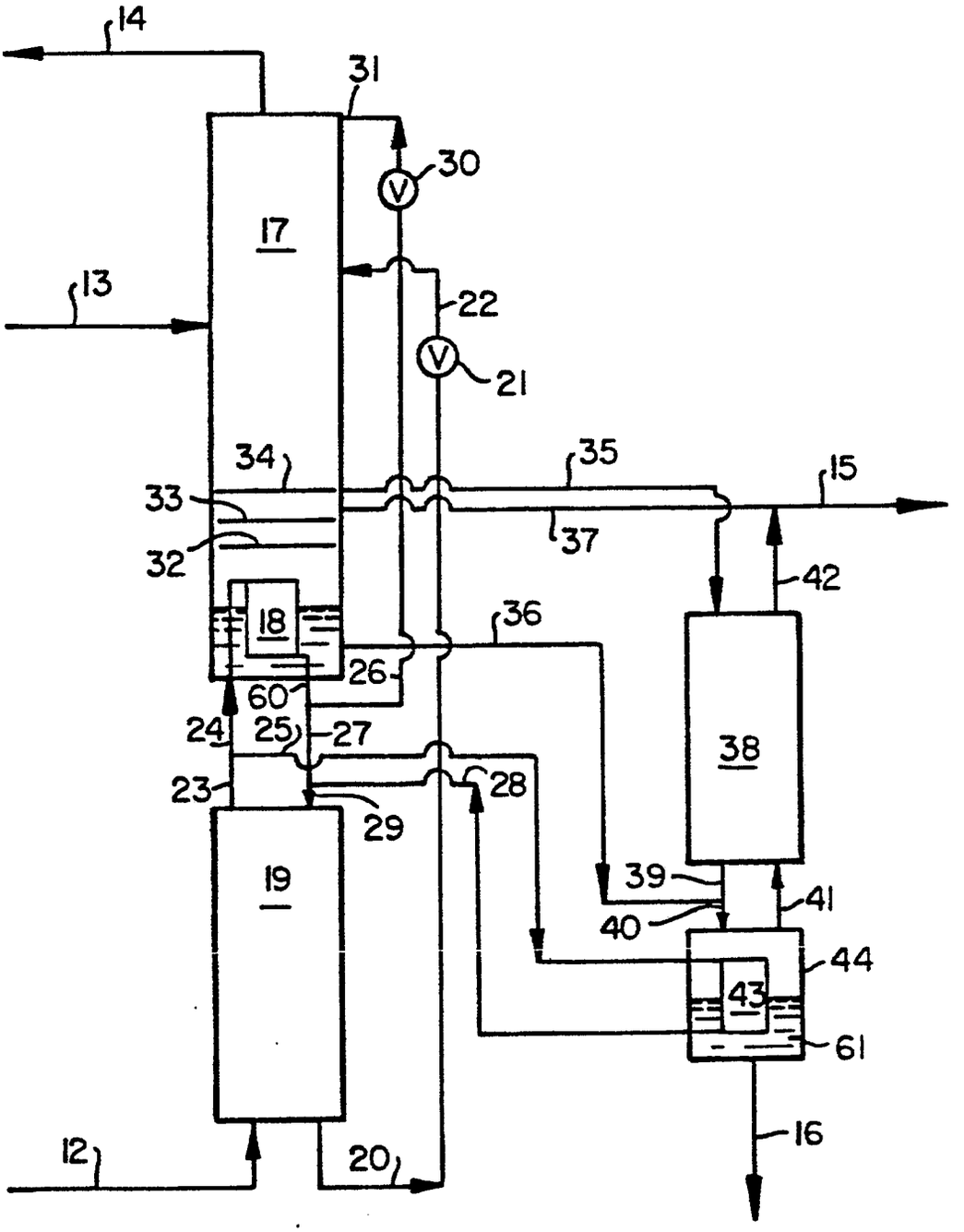
12. Procédé suivant la revendication 8 ou 11, dans lequel le liquide de reflux (35) est soutiré de la colonne (17) à basse pression au moins deux étages d'équilibre au-dessus de la zone de relation d'échange de chaleur.

13. Procédé suivant l'une quelconque des revendications 7 à 12, dans lequel la vaporisation partielle du liquide de réébullition (61) est effectuée par échange indirect de chaleur avec de la vapeur riche en azote (25) qui se condense, cette vapeur provenant de la colonne (19) à haute pression.

14. Procédé suivant la revendication 13, dans lequel le courant condensé (28) résultant riche en

azote est recyclé à la colonne à haute pression (19) comme reflux liquide.

15. Procédé suivant l'une quelconque des revendications précédentes, dans lequel la vapeur pauvre (42) évacuée et la portion principale (37) du produit (15) constitué d'oxygène gazeux sont réunies et recueillies ensemble.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