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(54) **Process and device for the production of argon by cryogenic separation of air**

(57) A process for producing argon by low temperature air fractionation, comprising the following steps: feeding a rectification column of at least one section (12,14) with said air (10); extracting from said rectification column an argon-containing fraction (18) and feeding it

to a crude argon column (16); extracting from said crude argon column an argon-containing fraction (22) and feeding it to a nitrogen removal column (24); and extracting from said nitrogen removal column an argon-containing fraction (30) and feeding it to a pure argon column (32).

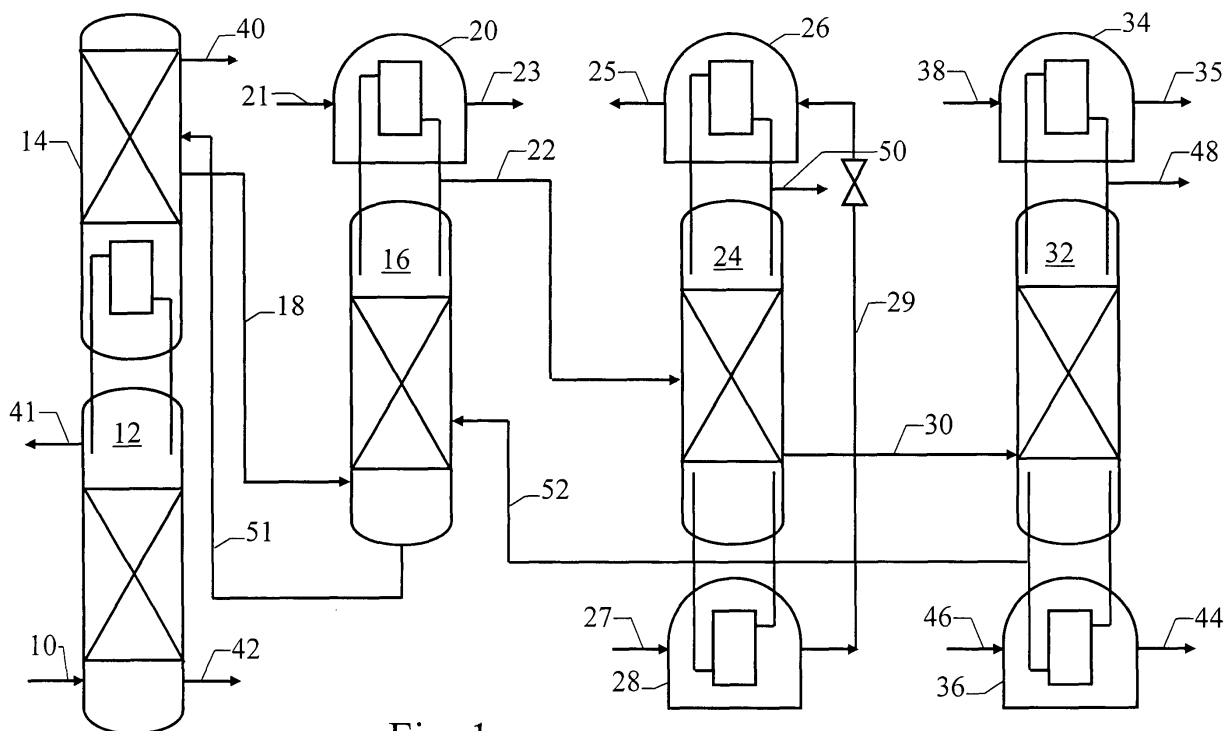


Fig. 1

Description

[0001] The present invention relates to an argon production plant and the relative process, and more particularly to a plant for argon production by low temperature fractionation of air, and the relative process.

[0002] Cryogenic rectification is a known system for separating argon from other atmospheric gases.

[0003] The air is compressed, purified, cooled and fed to a conventional air rectification column preferably divided into two sections.

[0004] The main air separation products, oxygen and nitrogen, can be obtained directly from the rectification column at the required purity level. However argon, the boiling point (87.8 K at 1 bar) of which lies between the boiling point of oxygen (90.2 K at 1 bar) and nitrogen (77.4 K at 1 bar) is consequently enriched within the central region of the upper column section. A fraction with maximum argon enrichment is withdrawn from this region.

[0005] In known plants, the argon-rich fraction is fed to a crude argon column, where oxygen is partially removed. Consequently, the product has still to be purified of the remaining oxygen and nitrogen at the exit of the crude argon column.

[0006] The oxygen can be removed only partially in the crude argon column, because the boiling points of argon and oxygen are extremely close together. The difference in their boiling points is 2.9 K at a pressure of 1 bar.

[0007] The remainder of the oxygen is normally removed by a deoxo unit in which the product is mixed with hydrogen, the oxygen then being burnt with the formation of water.

[0008] A deoxo unit is a costly apparatus with high running costs because of its considerable hydrogen consumption.

[0009] An object of the present invention is to provide an argon production plant and relative process, which is of lower installation and operating cost than those of the known art.

[0010] Another object is to enable already existing plants to be easily and economically converted to obtain plants of equal or superior performance with lower running and maintenance costs.

[0011] These objects are attained according to the present invention by a process for producing argon by low temperature air fractionation, comprising the following steps: feeding a rectification column of at least one section with said air; extracting from said rectification column an argon-containing fraction and feeding it to a crude argon column; extracting from said crude argon column an argon-containing fraction and feeding it to a nitrogen removal column; and extracting from said nitrogen removal column an argon-containing fraction and feeding it to a pure argon column.

[0012] These objects are also attained by a plant for producing argon by low temperature air fractionation,

comprising a rectification column of at least one section fed with said air; a crude argon column fed with an argon-containing fraction withdrawn from said rectification column; a nitrogen removal column fed with an argon-containing fraction withdrawn from said crude argon column; and a pure argon column fed with an argon-containing fraction withdrawn from said nitrogen removal column.

[0013] Further characteristics of the invention are described in the dependent claims.

[0014] The characteristics and advantages of the present invention will be apparent from the ensuing detailed description of one embodiment thereof, illustrated by way of non-limiting example in the accompanying Figure 1, which shows a schematic view of a plant according to the present invention.

[0015] In the accompanying figure, the argon production plant consists of a conventional rectification column with two portions or sections, comprising a high pressure column 12 and a low pressure column 14.

[0016] After being cleaned, cooled and compressed, the air, comprising mainly oxygen, nitrogen and argon, is fed into the column 12 through the pipe 10.

[0017] The column 12 has between 30 and 50 theoretical stages, the column 14 having between 50 and 90 theoretical stages.

[0018] A fraction of the product is withdrawn from a suitable region of the column 14 (between stage 20 and stage 40) where the argon concentration is a maximum and the nitrogen concentration is low, and is fed to the bottom of a crude argon column 16 via a pipe 18.

[0019] In the top of the column 16 a condenser heat exchanger 20 is present, fed via the pipe 21 with oxygen-rich liquid, i.e. with about 30-45% of oxygen (O₂), about 55-70% of nitrogen (N₂) and about 1-2% of argon (Ar), originating from the pipe 42, or with crude liquid argon originating from the bottom of the column 32. Through the pipe 23, the condenser heat exchanger 20 discharges vapour at the same composition as that entering through the pipe 42, and returns it either to the column 14 in a region between the exits of the pipes 18 and 40 at a point where the oxygen-rich mixture has an equal or similar oxygen (O₂) percentage, or to the column 32 if in the form of crude argon in the gaseous state. The stream 51 leaving the bottom of the column 16, and containing essentially oxygen (O₂) and argon (Ar), is returned to the upper part 14 of the air fractionation column in a position above the exit 18.

[0020] The argon withdrawn from the crude argon column 16 via the pipe 22 has a concentration between 96 and 99.9%, and is fed to an intermediate point of a nitrogen removal column 24.

[0021] The nitrogen removal column 24 has between 30 and 60 theoretical stages.

[0022] In the top of the column 24 a condenser heat exchanger 26 is present, fed with liquid nitrogen via the pipe 29, at a pressure between 1.1 and 2 bar, which enables the argon and oxygen fraction present in the column 24 to condense. whereas the nitrogen fraction

(0.1-4%) which does not condense (because the nitrogen condensation temperature is lower than the condenser temperature), is discharged to atmosphere through the pipe 50.

[0023] The liquid nitrogen, fed to the condenser heat exchanger 26 through the pipe 29, vaporizes and is discharged via the pipe 25 and fed to the main heat exchanger (not shown, the purpose of which is to cool the air entering through the pipe 10).

[0024] In the bottom of the column 24 a reboiler heat exchanger 28 is present, fed via the pipe 27 with gaseous nitrogen at a pressure between 3 and 7 bar and a temperature close to its liquefaction temperature.

[0025] This nitrogen is liquefied by the argon mixture fraction present on the other side of the reboiler heat exchanger 28, and is fed to the condenser heat exchanger 26 via the pipe 29.

[0026] Argon collects in the bottom of the column 24 with a nitrogen content less than 10 ppm and with a residual oxygen percentage corresponding to the feed fraction, and hence preferably between 10 ppm and 5%, as it depends on the number of stages present in the crude argon column 16. Typically the number of theoretical stages of the crude argon column 16 is between 40 and 145.

[0027] In the case of existing argon production plants which are to be modified by removing the deoxo unit, this percentage is normally between 1% and 2% (with about 40 theoretical stages).

[0028] The argon withdrawn from the column 24 is fed via the pipe 30 to a pure argon column 32.

[0029] The number of theoretical stages of the pure argon column 32 is between 40 and 120 and depends on the residual oxygen fraction present in the feed; the lower the oxygen fraction, the smaller the number of theoretical stages required.

[0030] A reflux condenser 36 is present in the top of the pure argon column 32, the bottom of which contains a reboiler heat exchanger 36.

[0031] Through the pipe 38 the condenser heat exchanger 34 is fed either with a liquid argon fraction withdrawn from the column 14 at low pressure and hence via the pipe 40, or with a liquid argon fraction withdrawn from the column 12 at high pressure and hence via the pipe 41, or with an oxygen-rich liquid fraction withdrawn from the column 12 at high pressure and hence via the pipe 42, or with fluid withdrawn from the reboiler heat exchanger 36 via the pipe 44. The condenser heat exchanger 34 discharges through the pipe 35, either into the main plant heat exchanger if in the form of nitrogen in the gaseous state, or into the column 14 in a region between the exits of the pipes 18 and 40 at a point where the oxygen-rich mixture has an equal or similar oxygen (O₂) percentage.

[0032] Through the pipe 46, the reboiler heat exchanger 36 is fed either with compressed air or with compressed nitrogen, or with crude argon vapour leaving the top of the crude argon column 16, in which case it becomes the reboiler heat exchanger for the pure argon

column 32 and the condenser for the crude argon column 16.

[0033] Argon which is practically oxygen-free (less than 10 ppm) and nitrogen-free (as this was eliminated in the previous column) can be withdrawn from the top of the pure argon column 32 through the pipe 48.

[0034] The oxygen-rich liquid stream 52 is withdrawn from the bottom of the column 32 and fed to the lower part of the crude argon column 16.

[0035] The columns 12, 14, 16, 24 and 32 can comprise either plates or structured fillings or random fillings, either individually or combined.

[0036] In already existing plants in which the deoxo unit is to be eliminated in accordance with the present invention, the conversion is very simple. A large part of the existing plant can be retained rather than replaced, and is hence economical.

[0037] Such plants are normally formed from a two-section rectification column followed by a deoxo unit and a nitrogen removal column in succession.

[0038] For the conversion, the deoxo unit is removed, and the connection which connected the two-section rectification column to the deoxo unit is now made directly to the nitrogen removal column. A pure argon column is installed and positioned in series after the nitrogen removal column. A plant in accordance with the present invention is obtained in this manner.

[0039] The operating temperatures and pressures, the connections and all other items necessary for the operation of the plant of the present invention which are not specifically stated form part of the knowledge of the expert of the art, and represents information obtainable from suitable technical documentation.

Claims

1. A process for producing argon by low temperature air fractionation, comprising the following steps:

feeding a rectification column (12, 14) of at least one section with said air (10);
extracting from said rectification column (12, 14) an argon-containing fraction (18) and feeding it to a crude argon column (16);
extracting from said crude argon column (16) an argon-containing fraction (22) and feeding it to a nitrogen removal column (24);
extracting from said nitrogen removal column (24) an argon-containing fraction (30) and feeding it to a pure argon column (32).

2. A process as claimed in claim 1, **characterised by** comprising the step of cooling the top of said pure argon column (32) by means of a heat exchanger (34) fed with a liquid nitrogen fraction (38) withdrawn from the low pressure section (40) or from the high pressure section (41) of said rectification column (12,

14).

3. A process as claimed in claim 1, **characterised by** comprising the step of cooling the top of said pure argon column (32) by means of a heat exchanger (34) fed (38) with a fraction (42) withdrawn from the high pressure section of said rectification column (12, 14). 5

5. A process as claimed in claim 1, **characterised by** comprising the step of using in the bottom of said pure argon column (32) a heat exchanger (36) fed (46) with a pressurized fluid. 10

6. A process as claimed in claim 1, **characterised by** comprising the step of using in the bottom of said pure argon column (32) a heat exchanger (36) fed (46) with pressurized argon or air which, when liquefied (44), feeds (38) a heat exchanger (34) positioned in the top of said pure argon column (32). 15 20

7. A process as claimed in claim 1, **characterised by** comprising the step of using in the bottom of said pure argon column (32) a heat exchanger (36) fed (46) with argon vapour extracted from the top of said crude argon column (16). 25

8. A process as claimed in claim 1, **characterised by** comprising the step of using in the top of said crude argon column (16) a heat exchanger (20) fed (21) with oxygen-rich liquid (42) or with liquid argon. 30

9. A plant for producing argon by low temperature air fractionation (10), comprising: 35

- a rectification column (12, 14) of at least one section fed with said air (10);
- a crude argon column (16) fed with an argon-containing fraction (18) withdrawn from said rectification column (12, 14); 40
- a nitrogen removal column (24) fed with an argon-containing fraction (22) withdrawn from said crude argon column (16);
- a pure argon column (32) fed with an argon-containing fraction (30) withdrawn from said nitrogen removal column (24). 45

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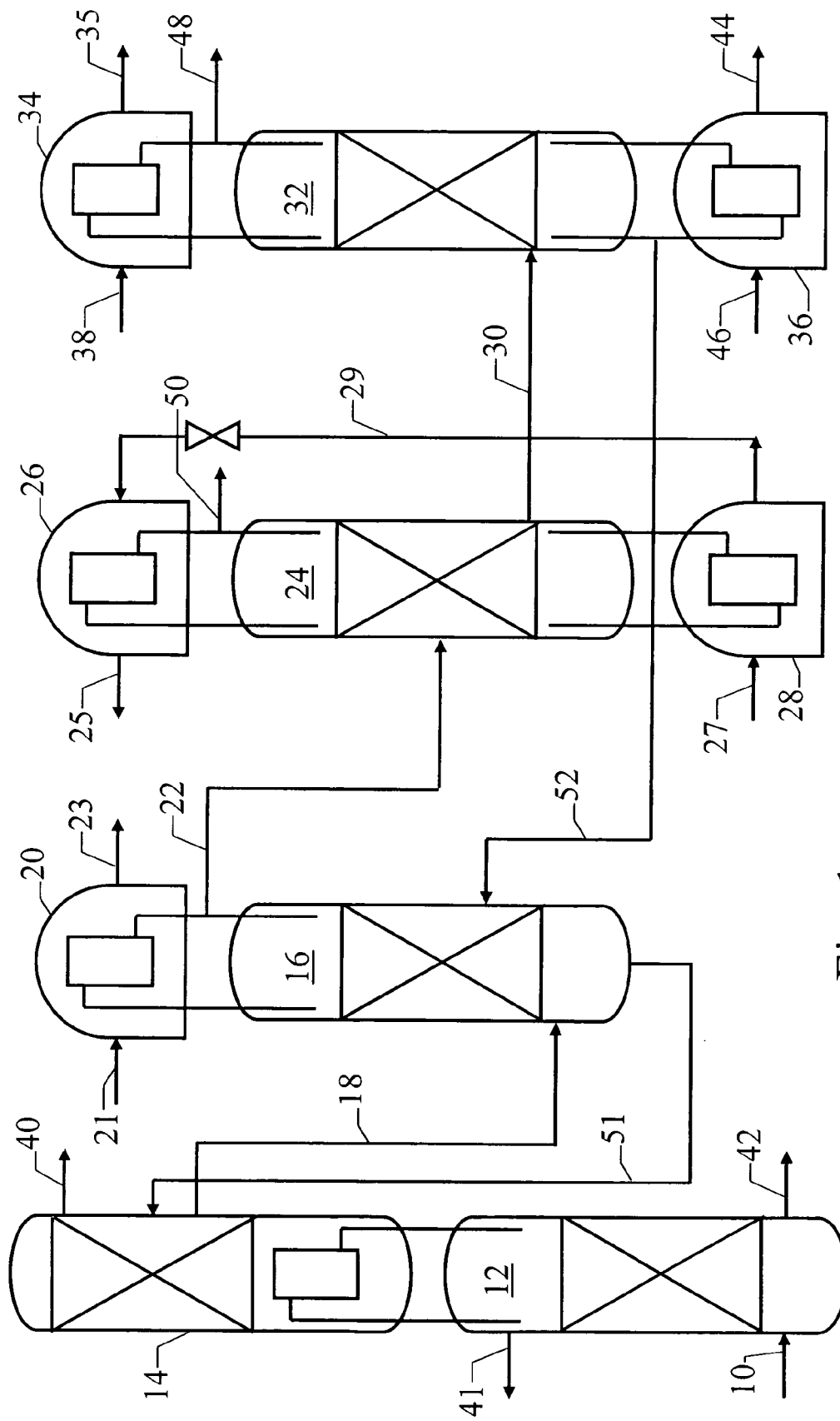


Fig. 1



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 05 42 5609

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 27 January 2006	Examiner Görizt, D
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
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EP 05 42 5609

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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