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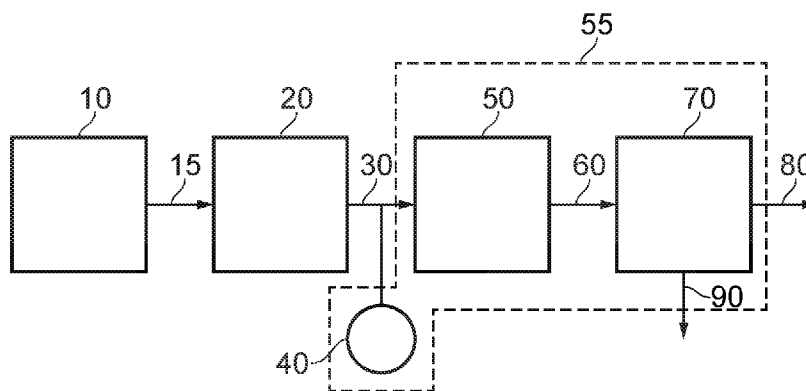


FIG. 1

(57) Abstract: A gas separator and method are disclosed. The gas separator is for separating a target gas component from a gas mixture and comprises: a first cryogenic stage configured in a solidifying phase to receive the gas mixture at a subatmospheric pressure, to solidify a first group of gas components of the gas mixture and to exhaust a second group of gas components of the gas mixture which includes the target gas component; and a second cryogenic stage configured to receive the second group of gas components at a subatmospheric pressure, to solidify the target gas component and to exhaust a third group of gas components of the gas mixture.

GAS SEPARATION

FIELD OF THE INVENTION

The field of the invention relates to a gas separator and method.

5

BACKGROUND

Different techniques exist for separating one or more gas components from a gas mixture. Although these enable gas components to be separated, unexpected consequences can occur. Accordingly, it is desired to provide an improved

10 technique for separating one or more gas components from a gas mixture.

SUMMARY

According to a first aspect, there is provided a gas separator for separating a target gas component from a gas mixture, comprising: a first cryogenic stage
15 configured in a solidifying phase to receive the gas mixture at a subatmospheric pressure, to solidify a first group of gas components of the gas mixture and to exhaust a second group of gas components of the gas mixture which includes the target gas component; and a second cryogenic stage configured to receive the second group of gas components at a subatmospheric pressure, to solidify the
20 target gas component and to exhaust a third group of gas components of the gas mixture.

The first aspect recognizes that a problem with existing techniques is that separating gases with very similar chemical and physical properties with high
25 efficiency is difficult. Accordingly, a separator is provided. The separator may separate one or more target gas components from a gas mixture. The gas mixture may comprise a plurality of gas components. The separator may comprise a first cryogenic stage. The first cryogenic stage may be configured in a solidifying or trapping phase to receive the gas mixture. The gas mixture may
30 be at subatmospheric pressure. The first cryogenic stage may solidify, condense or trap a first group of gas components of the gas mixture. The first cryogenic stage may be configured to exhaust a second group of gas components of the

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gas mixture. The second group of gas components may include the target gas component(s). The separator may comprise a second cryogenic stage. The second cryogenic stage may be configured to receive the second group of gas components. The second group of gas components may be at a subatmospheric pressure. The second cryogenic stage may solidify, condense or trap the target gas component. The second cryogenic stage may exhaust a third group of gas components of the gas mixture. In this way, the gas separator in the first stage traps the first group of gas components, leaving the target gas component and remaining gas components to flow into the second stage. The second stage then traps the target gas component allowing the remaining gases to be exhausted. This provides for efficient, high yield and high purity separation of a target gas component from a complex mixture of other gases.

A solidifying temperature of the first cryogenic stage in the solidifying phase may exceed a solidifying temperature of the second cryogenic stage. Hence, the first cryogenic stage may capture gas components with a higher boiling point than gas components captured in the second stage.

The solidifying temperature of the first cryogenic stage in the solidifying phase may exceed a solidifying temperature of the target gas component. Accordingly, the first cryogenic stage may solidify gas components having a higher boiling point than the target gas component.

The solidifying temperature of the second cryogenic stage may be at or below a solidifying temperature of the target gas component. Hence, at least the target gas component will be solidified by the second cryogenic stage.

The solidifying temperature of the second cryogenic stage may exceed a solidifying temperature of the third group of gas components of the gas mixture. Hence, the solidifying temperature of the second cryogenic stage may be centred around the solidifying temperature of the target gas component, allowing the third group of gas components pass through.

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The second cryogenic stage may be configured in a regeneration phase to evaporate the target gas component and convey the target gas component to a target gas component receptacle. Accordingly, the target gas component(s) may
5 be evaporated in order to be retrieved.

The second cryogenic stage may be configured in the regeneration phase to convey the target gas component to the target gas component receptacle via the first cryogenic stage. Hence, the target gas component(s) may be routed through
10 the first cryogenic stage.

The second cryogenic stage may be configured to operate in the regeneration phase while the first cryogenic stage remains configured to operate in the solidifying phase.
15

An evaporating temperature of the second cryogenic stage in the regeneration phase may exceed the solidifying temperature.

The evaporating temperature of the second cryogenic stage in the regeneration
20 phase may exceed an evaporation temperature of the target gas component.

The first cryogenic stage may be configured in the regeneration phase to evaporate the first group of gas components and to exhaust the first group of gas components.
25

An evaporating temperature of the first cryogenic stage in the regeneration phase may exceed the solidifying temperature.

The evaporating temperature of the first cryogenic stage in the regeneration
30 phase may exceed an evaporation temperature of the first group of gas components.

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The evaporating temperature of the first cryogenic stage in the regeneration phase may exceed the evaporating temperature of the second cryogenic stage.

5 The separator may comprise an analyser configured to analyse a gas component composition of the gas mixture and to determine at least one of the solidifying temperature and the evaporating temperature of the first cryogenic stage and the solidifying temperature and the evaporating temperature of the second cryogenic stage. Hence, an analyser may be used to obtain the composition of the gas mixture and determine the operating conditions of the first and second cryogenic
10 stages.

The analyser may be configured to determine an operating pressure of the first cryogenic stage and the second cryogenic stage in at least one of the solidifying phase and the regeneration phase. It will be appreciated that varying the
15 operating pressure affects the temperature at which gas components solidify and evaporate and can help obtain a suitable temperature window which takes account of temperature variations of the first and second cryogenic stages while still achieving adequate separation.

20 The analyser may be configured to determine the solidifying temperatures, the evaporating temperatures and the operating pressures based on phase diagrams of the gas component composition of the gas mixture.

25 The solidifying temperatures and the operating pressures may be selected to be below a triple-point of gas components to be solidified in that cryogenic stage.

The first cryogenic stage and the second cryogenic stage may comprise a first pair of cryogenic stages and the gas separator may comprise a second pair of cryogenic stages in parallel with the first pair of cryogenic stages, the first pair of
30 cryogenic stages operating in the solidifying phase while the second pair of cryogenic stages operates in the regenerating phase and the first pair of cryogenic stages operating in the regenerating phase while the second pair of

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cryogenic stages operates in the solidifying phase. This helps to provide for continuous operation.

At least one of the first cryogenic stage and the second cryogenic stage may
5 comprise at least one insulated heat exchanger coupled with a cryogenic head.

The insulated heat exchanger may be surrounded by at least one of a vacuum chamber and radiation shield.

10 The insulated heat exchanger may comprise an inlet configured to receive its gas mixture, cooling fins defining a torturous path configured to solidify in the solidifying phase and evaporate in the regeneration phase and an outlet.

The cooling fins may comprise a plurality of coaxial cylinders each defining at
15 least one aperture to support flow between the inlet and the outlet.

The gas mixture may comprise gases exhausted from a semiconductor processing tool.

20 The gas mixture may comprise one of more of: N₂, He, O₂, H₂, CO_x, Kr, Ar, SO_x, NO_x, CF₄, SF₆, NF₃.

The first group of gas components may comprise one or more of: CO_x, SO_x, NO_x, CF₄, SF₆.
25

The second group of gas components may comprise one or more of: N₂, He, O₂, H₂, CO, Kr, Ar, NO, CF₄, NF₃.

The target gas component may comprise at least Kr.

30 The third group of gas components may comprise one or more of: N₂, He, O₂, H₂, CO, Ar.

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The separator may comprise a buffer tank configured to supply the gas mixture.

- 5 The separator may comprise at least one pre-processing stage configured to reduce at least one of H₂O and HF in the gas mixture.

The subatmospheric pressure may be below around 100mbar, preferably below around 20 mbar and preferably below around 10 mbar.

- 10 The separator may comprise at least one vacuum pump configured to provide the subatmospheric pressure.

- 15 According to a second aspect, there is provided a method of separating a target gas component from a gas mixture, comprising: receiving, in a solidifying phase, the gas mixture at a subatmospheric pressure at first cryogenic stage, solidifying a first group of gas components of the gas mixture and exhausting a second group of gas components of the gas mixture which includes the target gas component; and receiving the second group of gas components at a subatmospheric pressure, solidifying the target gas component and exhausting a
20 third group of gas components of the gas mixture.

A solidifying temperature of the first cryogenic stage in the solidifying phase may exceed a solidifying temperature of the second cryogenic stage.

- 25 The solidifying temperature of the first cryogenic stage in the solidifying phase may exceed a solidifying temperature of the target gas component.

The solidifying temperature of the second cryogenic stage may be at or below a solidifying temperature of the target gas component.

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The method may comprise: in a regeneration phase, evaporating the target gas component and conveying the target gas component to a target gas component receptacle.

- 5 The conveying may comprise: conveying the target gas component to the target gas component receptacle via the first cryogenic stage.

10 The method may comprise: operating the second cryogenic stage in the regeneration phase while the first cryogenic stage remains configured to operate in the solidifying phase.

An evaporating temperature of the second cryogenic stage in the regeneration phase may exceed the solidifying temperature.

- 15 The evaporating temperature of the second cryogenic stage in the regeneration phase may exceed an evaporation temperature of the target gas component.

20 The method may comprise: in the regeneration phase, evaporating the first group of gas components and exhausting the first group of gas components.

An evaporating temperature of the first cryogenic stage in the regeneration phase may exceed the solidifying temperature.

25 The evaporating temperature of the first cryogenic stage in the regeneration phase may exceed an evaporation temperature of the first group of gas components.

30 The evaporating temperature of the first cryogenic stage in the regeneration phase may exceed the evaporating temperature of the second cryogenic stage.

The method may comprise: analysing a gas component composition of the gas mixture and determining at least one of the solidifying temperature and the

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evaporating temperature of the first cryogenic stage and the solidifying temperature and the evaporating temperature of the second cryogenic stage.

5 The determining may comprise determining an operating pressure of the first cryogenic stage and the second cryogenic stage in at least one of the solidifying phase and the regeneration phase.

10 The determining may comprise determining the solidifying temperatures, the evaporating temperatures and the operating pressures based on phase diagrams of the gas component composition of the gas mixture.

15 The method may comprise: selecting the solidifying temperatures and the operating pressures to be below a triple-point of gas components to be solidified in that cryogenic stage.

20 The first cryogenic stage and the second cryogenic stage may comprise a first pair of cryogenic stages and the gas separator comprises a second pair of cryogenic stages in parallel with the first pair of cryogenic stages, the method may comprise: operating first pair of cryogenic stages in the solidifying phase while the second pair of cryogenic stages operates in the regenerating phase and the first pair of cryogenic stages operating in the regenerating phase while the second pair of cryogenic stages operates in the solidifying phase.

25 At least one of the first cryogenic stage and the second cryogenic stage may comprises at least one insulated heat exchanger coupled with a cryogenic head.

The insulated heat exchanger may be surrounded by at least one of a vacuum chamber and radiation shield.

30 The insulated heat exchanger may comprise an inlet configured to receive its gas mixture, cooling fins defining a torturous path configured to solidify in the solidifying phase and evaporate in the regeneration phase and an outlet.

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The cooling fins may comprise a plurality of coaxial cylinders each defining at least one aperture to support flow between the inlet and the outlet.

- 5 The gas mixture may comprise gases exhausted from a semiconductor processing tool.

The gas mixture may comprise one of more of: N₂, He, O₂, H₂, CO_x, Kr, Ar, SO_x, NO_x, CF₄, SF₆, NF₃.

10

The first group of gas components may comprise one or more of: CO_x, SO_x, NO_x, CF₄, SF₆.

The second group of gas components may comprise one or more of: N₂, He, O₂,
15 H₂, CO, Kr, Ar, NO, CF₄, NF₃.

The target gas component may comprise at least Kr.

The third group of gas components may comprise one or more of: N₂, He, O₂, H₂,
20 CO, Ar.

The method may comprise: supplying the gas mixture from a buffer tank.

The method may comprise: pre-processing to reduce at least one of H₂O and HF
25 in the gas mixture.

The subatmospheric pressure may be below around 100mbar, preferably below around 20 mbar and preferably below around 10 mbar.

30 The method may comprise: providing the subatmospheric pressure using at least one vacuum pump.

Further particular and preferred aspects are set out in the accompanying independent and dependent claims. Features of the dependent claims may be combined with features of the independent claims as appropriate, and in combinations other than those explicitly set out in the claims.

5

Where an apparatus feature is described as being operable to provide a function, it will be appreciated that this includes an apparatus feature which provides that function or which is adapted or configured to provide that function.

10 BRIEF DESCRIPTION OF THE DRAWINGS

Embodiments of the present invention will now be described further, with reference to the accompanying drawings, in which:

Figure 1 illustrates schematically processing equipment including a gas separator according to one embodiment;

15

Figure 2 illustrates example vapor pressure curves;

Figure 3 illustrates in more detail a first arrangement of the gas separator in the solidifying or trapping phase;

Figure 4 illustrates in more detail the first arrangement of the gas separator in the regeneration or evaporation phase;

20

Figure 5 illustrates in more detail a second arrangement of the gas separator in the solidifying or trapping phase;

Figure 6 illustrates in more detail the second arrangement of the gas separator in a first part of the regeneration or evaporation phase;

Figure 7 illustrates in more detail the second arrangement of the gas separator in a second part of the regeneration or evaporation phase; and

25

Figure 8 illustrates an example arrangement of a heat exchanger of the gas separator.

DESCRIPTION OF THE EMBODIMENTS

30

Before discussing the embodiments in any more detail, first an overview will be provided. Some embodiments provide an arrangement for capturing or trapping one or more target gas components from a gas stream containing a plurality of

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gas components. The composition of the gas stream is determined (either by analysis or based on information provided about the expected composition). The target gas component(s) to be separated are identified. The gas separator has a plurality of stages. A first stage is configured to operate a desired temperature and pressure which captures those gas components having a higher boiling point than the target gas component(s). Hence, the target gas component(s) and the remainder of the gas components in the gas stream pass to a second stage. The second stage is configured to operate a desired temperature and pressure which captures the target gas component(s) and the remainder of the gas components in the gas stream are exhausted. The captured target gas component(s) can then be released. In some embodiments, phase diagrams are used to identify suitable temperatures and pressures (base typically on an expected operating temperature and/or pressure window achievable by the separator) which capture and release the required gas components in order to separate the target gas component(s) with a suitable purity.

Gas Separator

Figure 1 illustrates schematically processing equipment including a gas separator 55 according to one embodiment. A semiconductor processing tool 10 or other source generates a gas waste stream 15 containing a mixture of gas components. The waste stream 15 is typically supplied to processing equipment 20 which processes or abates the waste stream 15 and generates a processed waste stream 30. Typically, the processing equipment 20 helps to remove highly corrosive material such as, for example, F₂ and HF from the waste stream 15, as well as reduce or remove water present in the processed waste stream 30. The processed waste stream 30 is supplied to the gas separator 55 which separates one or more target gas components 90 from the processed waste stream 30.

The gas separator 55 has a first stage 50 which receives the processed waste stream 30, separates a first group of gas components and exhausts a second group of gas components 60, which are supplied to the second stage 70, which separates the target gas component(s) 90 and exhausts a third group of gas

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components 80. The gas separator 55 typically has a gas analyser 40 which analyses the composition of the processed waste stream 30 supplied to the first stage 50 when that composition is unknown.

- 5 The waste stream 15 from the semiconductor processing tool 10 or from elsewhere typically contains many components. Recovery of high-value species or components from this waste stream 15 typically requires multiple operations to treat specific components of the mixture. Hence, the waste stream 15 is supplied to the processing equipment 20 which removes typically highly corrosive material
- 10 such as F₂ and HF since these are highly detrimental to the longevity of the equipment. Also, the removal or reduction of water vapor from the processed waste stream 30 helps to reduce the burden on downstream operations and impact on the purity of the target gas component(s) 90. Hence, once the waste stream 15 has been cleaned of corrosive materials and most of the water vapor
- 15 removed then the processed waste stream 30 is supplied to the separator 55.

Typically, in a trapping or solidifying phase, the first stage 50 is operated to capture high boiling-point gas components (such as, for example, CO_x, NO_x and SO_x) forming a first group of gas components and allow the target gas

20 component(s) 90 along with lower boiling-point gas components forming a second group of gas components 60 to pass through and be exhausted from the first stage 50. The second group of gas component 60 is then received by the second stage 70 which is operated, in a trapping or solidifying phase, to capture the target gas component(s) 90 and allow lower boiling-point component to pass

25 through and be exhausted as a third group of gas components 80. In a regeneration phase, the captured target gas component(s) 90 are evaporated by the second stage 70. Also the higher boiling-point components captured by the first stage 50 are evaporated and exhausted typically via the second stage 70.

30 Heat Exchanger

Each stage 50, 70 includes a heat exchanger through which the gases pass, an example of which is illustrated in Figure 8. As can be seen, an inlet 130 is

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provided which receives a gas stream. A plurality of concentric, cylindrical cooling fins 140 provided which define a torturous path through which the gas stream flows before being exhausted via an outlet 150. A cryogenic chiller unit (not shown) is coupled with a chiller head 160 which controls the operating

5 temperature of the cooling fins 140. The heat exchanger comprises a surrounding vacuum chamber 170 and a radiation shield 180 (such as a mylar reflective sheet) which helps to maintain uniform temperature within the heat exchanger and reduce external radiant heating.

10 Pressure/Temperature Selection

To achieve a high capture efficiency, careful choice of the operating temperature and pressure conditions within the first and second stages 50, 70 is important. The temperature and pressure conditions required can vary based on the mixture composition of the processed waste stream 30. This composition may be known

15 or estimated based on the likely composition of the waste stream 15, or may be determined by the analyser 40. Vapor pressure curves for the components within the processed waste stream 30 can then be utilized to determine the operating temperatures and pressures of the first and second stages 50, 70. Also, thermal gradients within the heat exchanger, which are determined by the geometry and

20 material of construction, are taken into account when estimating the correct operating temperatures and pressures. It is desired to minimize the temperature gradient in the heat exchanger so that most of the condensing surface is within a desired operating temperature window. Furthermore, the heat exchanger should provide sufficient condensing surface areas such that the gas stream is cooled to

25 the target temperatures and space is available for the build-up of condensed material without causing blockage.

Calculations have shown that operating the heat exchangers at pressures below 1000 mbar, and particularly below 10 mbar, provide a useful, wide operating

30 window for the heat exchange temperature. Without this wide operating window, the maximum permissible temperature gradient in the heat exchanger becomes difficult to achieve. If parts of the heat exchanger are too cold, then condensation

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of unwanted material will occur. This results in either, for the first stage 50, loss of the target material (that is the target material is captured prematurely by the first stage 50 and sent to the waste stream 80) or, in the second stage 70, this results in capture of material that was supposed to pass through the heat

5 exchanger but will now contaminate the captured target component(s) 90. For example, example applications have shown estimated capture efficiencies for a specific target gas based on partial pressures of the individual species compared to their vapor pressure. When operating at 1000 mbar, the estimated minimum temperature for the target gas is 75K and the target species capture efficiency is 10 94%. Below 75K, unwanted mixture components would be condensed.

However, at 80K, a 5K temperature increase, the capture efficiency of the target gas falls to 83%. Using the same gas mixture, but operating at 1 mbar, the minimum temperature is 47K at a capture efficiency of 99.9%, and this falls to 97.9% at 52K (a 5K temperature increase over the minimum temperature). In 15 other words, lower pressure operation is much less sensitive to temperature increases or temperature gradients in the heat exchanger.

Generally, the operating pressure and temperature are chosen to be below the triple point of the species to be condensed to ensure solid formation versus liquid 20 formation. This reduces the difficulty of handling a mobile cryogenic liquid and reduces the effect of non-ideal behavior of gas-liquid mixtures permitting simplified calculation of the required operating pressure and temperature conditions.

25 Example Operation - Simplified

The gas analyser 40 analyses the processed waste stream 30. In this simplified example, it is assumed that the and processed waste stream 30 is comprised of N₂ (approx. 86%), Kr (approx. 2.2%) and CO₂ (approx. 11.8%). Figure 2 illustrates example vapor pressure curves 100, 110, 120 for N₂, Kr and CO₂, 30 respectively. As can be seen from the vapor pressure curves 100, 110, 120, when operating the first stage 50 at 10 mbar and at 73K (assuming a 5K temperature window within the heat exchanger) in the solidifying or trapping

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phase, the partial pressure of Kr and N₂ is less than the saturation vapor pressure and so Kr and N₂ will pass through the first stage 50 and be exhausted as the first group of gas components 60 while the partial pressure of CO₂ is more than the saturation vapor pressure at 73K and so CO₂ is trapped by being solidified on the cooling fins 40 within the first stage 50. When operating the second stage 70 at 63K (assuming a 5K temperature window within the heat exchanger), the partial pressure of N₂ is less than the saturation vapor pressure at 63K and so N₂ will pass through the second stage 70. However, the partial pressure of Kr is more than the saturation vapor pressure at 63K so Kr is trapped by being solidified on the cooling fins 140 of the second stage 70. In the regeneration phase, the temperature of the second stage 70 is raised to evaporate the Kr from the cooling fins 140 and is collected as the target gas component 90. Likewise, the temperature of the first stage 50 is raised to evaporate the CO₂, which can be exhausted.

Example Operation – Semiconductor Processing Waste Stream – 1st Arrangement

Figure 3 illustrates in more detail a first arrangement of the gas separator in the solidifying or trapping phase. The processed waste stream 30 is collected and stored in a buffer tank 190 and analysed by the gas analyser 40 if its composition is unknown. In this example, the processed waste stream 30 contains main constituents of N₂, He, O₂, H₂, CO₂, Kr, together with minor constituents of Ar, SO₂, NO_x, CO, CF₄, SF₆, NF₃. From the vapor pressure curves for these components it is determined by a controller 320 that in order to separate Kr as the target gas component 90, it is necessary to operate, in the solidifying or trapping phase, the first stage 50 at around 60K-80K at a pressure of around 1 to 10 mbar and the second stage 70 at around 50K-65K at a pressure of around 0.1 to 10 mbar. Temperature sensors 330, 340 and pressure sensors 350, 360 monitor and report the temperatures and pressures in the first and second stages 50, 70 to the controller 320 which controls the operation of the valves, vacuum pumps 300, 310 and the cryogenic chiller units. The processed waste stream 30 is provided to the first stage 50, which in the solidifying or trapping phase, traps

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CO_x, NO_x and SO_x and exhausts Kr, NF₃, CF₄, NO, Ar, CO, H₂, O₂, N₂, He as the second group of gas component 60 which is supplied to the second stage 70.

The second stage 70, in the solidifying or trapping phase, solidifies and traps Kr (together with residual amounts of CF₄, NF₃, and NO) and exhausts Ar, CO, H₂,

5 O₂, N₂, He as the third group of gas components 80.

Calculations have shown that when operating the first stage 50 at 10 mbar and 68K, 100% of CO₂, NO₂, SO₂, SF₆ is captured, 98% of CF₄ is captured and 68% of NO is captured. Likewise, when operating the first stage 50 at 10 mbar and

10 114K, 99% of CO₂, NO₂, SO₂ is captured, zero NO, CF₄ or SF₆ is captured.

Calculations have also shown that when operating the second stage 70 at 8 mbar and 53K, 99.6% of Kr is captured, 0% of N₂ is captured and the target or recovered gas mixture is 95.6% Kr with a 4.4% contamination of NO, CF₄ and

15 Kr is captured, 0% of N₂ is captured and the target or recovered gas mixture is 95.5% Kr with a 4.5% contamination of NO, CF₄ and NF₃.

As shown in Figure 4, during a regeneration or evaporation phase, the flow of the processed waste stream 30 is ceased and the first and second stages 50, 70 are isolated from each other by activating an isolating valve 200. The temperature of the first stage 50 is raised by around 100K to evaporate the CO_x, NO_x, SO_x which is exhausted from the separator. The temperature of the second stage is raised by around 20K which evaporates the Kr (together with residual amounts of CF₄, NF₃, and NO) as the target gas component 90, which is captured and stored.

25

Example Operation – Semiconductor Processing Waste Stream – 2nd

Arrangement

Figure 5 illustrates in more detail a second arrangement of the gas separator in the solidifying or trapping phase. This is similar to the arrangement shown in

30 Figure 4 but omits the isolation valve 200. The processed waste stream 30 is collected and stored in a buffer tank 190 and analysed by the gas analyser 40 if its composition is unknown. In this example, the processed waste stream 30

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contains main constituents of N₂, He, O₂, H₂, CO₂, Kr, together with minor constituents of Ar, SO₂, NO_x, CO, CF₄, SF₆, NF₃. From the vapor pressure curves for these components it is determined by a controller 320 that in order to separate Kr as the target gas component 90, it is necessary to operate, in the

5 solidifying or trapping phase, the first stage 50 at around 60K-80K at a pressure of around 1 to 10 mbar and the second stage 70 at around 50K-65K at a pressure of around 0.1 to 10 mbar. Temperature sensors 330, 340 and pressure sensors 350, 360 monitor and report the temperatures and pressures in the first and second stages 50, 70 to the controller 320 which controls the operation of the

10 valves, vacuum pumps 300, 310 and the cryogenic chiller units. The processed waste stream 30 is provided to the first stage 50, which in the solidifying or trapping phase, traps CO_x, NO_x and SO_x and exhausts Kr, NF₃, CF₄, NO, Ar, CO, H₂, O₂, N₂, He as the second group of gas component 60 which is supplied to the second stage 70. The second stage 70, in the solidifying or trapping phase,

15 solidifies and traps Kr (together with residual amounts of CF₄, NF₃, and NO) and exhausts Ar, CO, H₂, O₂, N₂, He as the third group of gas components 80.

However, as illustrated in Figure 6, during the regeneration or evaporation phase, the supply of the processed waste stream 30 is stopped, the outlet from the

20 second stage 70 is closed by an isolation valve 210, isolation valve is opened and the temperature of the second stage 70 is increased by around 20K to evaporate Kr (together with residual amounts of CF₄, NF₃, and NO) while the temperature of the first stage 50 remains at around 80K. The Kr (together with residual amounts of CF₄, NF₃, and NO) flow through the first stage 50 and are

25 captured as the target gas components 90.

Then, as illustrated in Figure 7, an isolation valve 220 is opened, the isolation valve 230 is closed, the temperature of the second stage 70 is raised further to remove any residual solidified components and the temperature of the first stage

30 50 is raised by around 100K to evaporate CO₂, NO₂, SO₂ and this is exhausted from the gas separator.

In some embodiments, the condensed stream from one stage could provide, after regeneration, the feedstock for another stage for another separation step to recover another species or gas component.

- 5 In some embodiments, the first and second stages 50, 70 are operated other than in series.

In some embodiments, the first and second stages 50, 70 are duplicated in parallel to provide near-continuous operation, with one pair operating in the
10 solidifying or trapping phase, with the other pair operating in the regeneration or evaporating phase and vice-versa.

Although the above arrangements are described with reference to separating gas components from semiconductor waste streams, it will be appreciated that it can
15 equally be used to recover gas components from other gas streams.

Hence, some embodiments provide for separation of typically high-value gas components from a gas mixture which is less energy intensive than techniques suited to large-scale operation. Separating gases with very similar chemical and
20 physical properties with high efficiency is difficult. For example, separation of a simple binary mixture of Krypton from Nitrogen using a porous media with a pressure swing adsorption or vacuum pressure swing adsorption system is inefficient because of the very similar kinetic diameters, 3.60Å and 3.64Å, respectively. The selectivity of the porous media for one species over the other is
25 low and thus high purity of a target species can only be achieved multiple stages and thus with substantial loss (low yield) of target species. In some embodiments, the target species is only a few percent of the total gas flow so high yield, as well as high purity, are critical and the arrangements set out above provide for efficient, high yield and high purity separation of a high-value gas, or
30 gases, from a complex mixture of other gases. These arrangements do not require adsorption onto a porous material and are able to separate gases with very similar physical and chemical properties with high yield and purity. Also,

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these arrangements do not suffer the yield/purity compromise that characterises other methods such as pressure swing adsorption, are more efficient than traditional large-scale liquefaction methods and are tuneable to cope with separation of a broad range of gas mixtures and target species.

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Although illustrative embodiments of the invention have been disclosed in detail herein, with reference to the accompanying drawings, it is understood that the invention is not limited to the precise embodiment and that various changes and modifications can be effected therein by one skilled in the art without departing

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from the scope of the invention as defined by the appended claims and their equivalents.

REFERENCE SIGNS

	Semiconductor Processing Tool	10
	Waste Stream	15
5	Processing Equipment	20
	Processed Waste Stream	30
	Analyzer	40
	First Stage	50
	Gas Separator	55
10	Second Group of Gas Components	60
	Second Stage	70
	Third Group of Gas Components	80
	Target Gas Component(s)	90
	Vapor Pressure Curves	100, 110, 120
15	Inlet	130
	Cooling Fins	140
	Outlet	150
	Chiller Head	160
	Vacuum Chamber	170
20	Radiation Shield	180
	Buffer Tank	190
	Isolation Valve	200, 210, 220, 230
	Vacuum Pumps	300, 310
	Controller	320
25	Temperature Sensors	330, 340
	Pressure Sensors	350, 360

CLAIMS

1. A gas separator for separating a target gas component from a gas mixture, comprising:

5 a first cryogenic stage configured in a solidifying phase to receive said gas mixture at a subatmospheric pressure, to solidify a first group of gas components of said gas mixture and to exhaust a second group of gas components of said gas mixture which includes said target gas component; and

10 a second cryogenic stage configured to receive said second group of gas components at a subatmospheric pressure, to solidify said target gas component and to exhaust a third group of gas components of said gas mixture.

15 2. The gas separator of claim 1, wherein a solidifying temperature of said first cryogenic stage in said solidifying phase exceeds a solidifying temperature of said second cryogenic stage.

20 3. The gas separator of claim 2, wherein said solidifying temperature of said first cryogenic stage in said solidifying phase exceeds a solidifying temperature of said target gas component.

4. The gas separator of claim 2 or 3, wherein said solidifying temperature of said second cryogenic stage is at or below a solidifying temperature of said target gas component.

25 5. The gas separator of any one of claims 2 to 4, wherein said solidifying temperature of said second cryogenic stage exceeds a solidifying temperature of said third group of gas components of said gas mixture.

30 6. The gas separator of any preceding claim, wherein said second cryogenic stage is configured in a regeneration phase to evaporate said target gas component and convey said target gas component to a target gas component receptacle.

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7. The gas separator of claim 6, wherein said second cryogenic stage is configured in said regeneration phase to convey said target gas component to said target gas component receptacle via said first cryogenic stage.

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8. The gas separator of claims 6 or 7, wherein said second cryogenic stage is configured to operate in said regeneration phase while said first cryogenic stage remains configured to operate in said solidifying phase.

10 9. The gas separator of any one of claims 6 to 8, wherein an evaporating temperature of said second cryogenic stage in said regeneration phase exceeds said solidifying temperature.

15 10. The gas separator of claim 9, wherein said evaporating temperature of said second cryogenic stage in said regeneration phase exceeds an evaporation temperature of said target gas component.

20 11. The gas separator of any one of claims 2 to 4, wherein said first cryogenic stage is configured in said regeneration phase to evaporate said first group of gas components and to exhaust said first group of gas components.

12. The gas separator of claim 11, wherein an evaporating temperature of said first cryogenic stage in said regeneration phase exceeds said solidifying temperature.

25

13. The gas separator of claim 12, wherein said evaporating temperature of said first cryogenic stage in said regeneration phase exceeds an evaporation temperature of said first group of gas components, and preferably wherein said evaporating temperature of said first cryogenic stage in said regeneration phase exceeds said evaporating temperature of said second cryogenic stage.

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14. The gas separator of any preceding claim, comprising an analyser configured to analyse a gas component composition of said gas mixture and to determine at least one of said solidifying temperature and said evaporating temperature of said first cryogenic stage and said solidifying temperature and
5 said evaporating temperature of said second cryogenic stage, and preferably wherein said analyser is configured to determine an operating pressure of said first cryogenic stage and said second cryogenic stage in at least one of said solidifying phase and said regeneration phase.

10 15. The gas separator of claim 14, wherein said analyser is configured to determine said solidifying temperatures, said evaporating temperatures and said operating pressures based on phase diagrams of said gas component composition of said gas mixture, and preferably wherein said solidifying temperatures and said operating pressures are selected to be below a triple-point
15 of gas components to be solidified in that cryogenic stage.

16. The gas separator of any preceding claim, wherein said first cryogenic stage and said second cryogenic stage comprise a first pair of cryogenic stages and said gas separator comprises a second pair of cryogenic stages in parallel
20 with said first pair of cryogenic stages, said first pair of cryogenic stages operating in said solidifying phase while said second pair of cryogenic stages operates in said regenerating phase and said first pair of cryogenic stages operating in said regenerating phase while said second pair of cryogenic stages operates in said solidifying phase.

25 17. The gas separator of any preceding claim, wherein at least one of said first cryogenic stage and said second cryogenic stage comprises at least one insulated heat exchanger coupled with a cryogenic head, and preferably wherein said insulated heat exchanger is surrounded by at least one of a vacuum
30 chamber and radiation shield.

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18. The gas separator of claim 17, wherein said insulated heat exchanger comprises an inlet configured to receive its gas mixture, cooling fins defining a torturous path configured to solidify in said solidifying phase and evaporate in said regeneration phase and an outlet, and preferably wherein said cooling fins
5 comprise a plurality of coaxial cylinders each defining at least one aperture to support flow between said inlet and said outlet.

19. The gas separator of any preceding claim, wherein said gas mixture comprises one of more of: N₂, He, O₂, H₂, CO_x, Kr, Ar, SO_x, NO_x, CF₄, SF₆, NF₃.

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20. The gas separator of any preceding claim, wherein said first group of gas components comprise one or more of: CO_x, SO_x, NO_x, CF₄, SF₆.

21. The gas separator of any preceding claim, wherein said second group of
15 gas components comprise one or more of: N₂, He, O₂, H₂, CO, Kr, Ar, NO, CF₄, NF₃.

22. The gas separator of any preceding claim, wherein said target gas component comprises at least Kr.

20

23. The gas separator of any preceding claim, wherein said third group of gas components comprise one or more of: N₂, He, O₂, H₂, CO, Ar.

24. The gas separator of any preceding claim, wherein said subatmospheric
25 pressure is below around 100mbar, preferably below around 20 mbar and preferably below around 10 mbar.

25. A method of separating a target gas component from a gas mixture, comprising:

30 receiving, in a solidifying phase, said gas mixture at a subatmospheric pressure at first cryogenic stage, solidifying a first group of gas components of

- 25 -

said gas mixture and exhausting a second group of gas components of said gas mixture which includes said target gas component; and

receiving said second group of gas components at a subatmospheric pressure, solidifying said target gas component and exhausting a third group of

5 gas components of said gas mixture.

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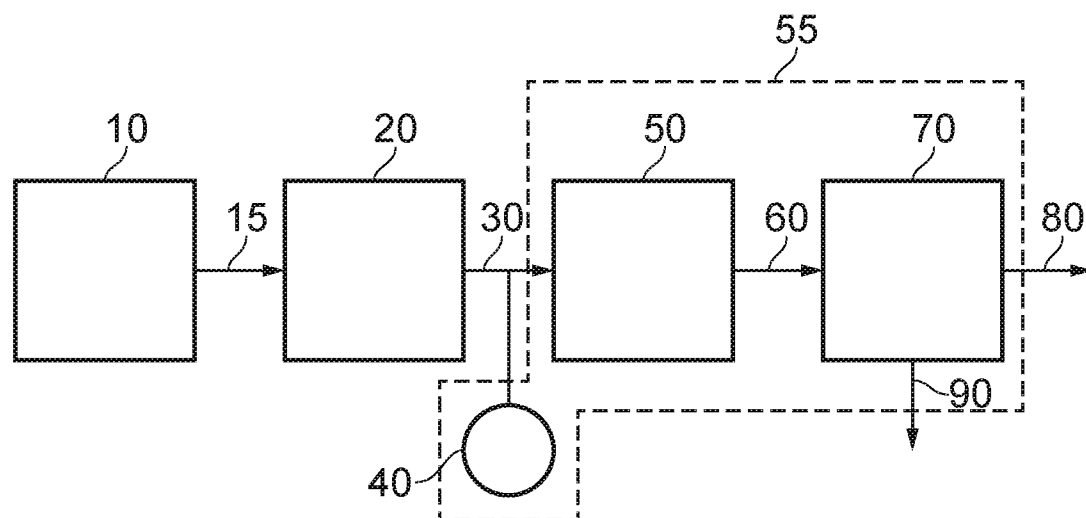


FIG. 1

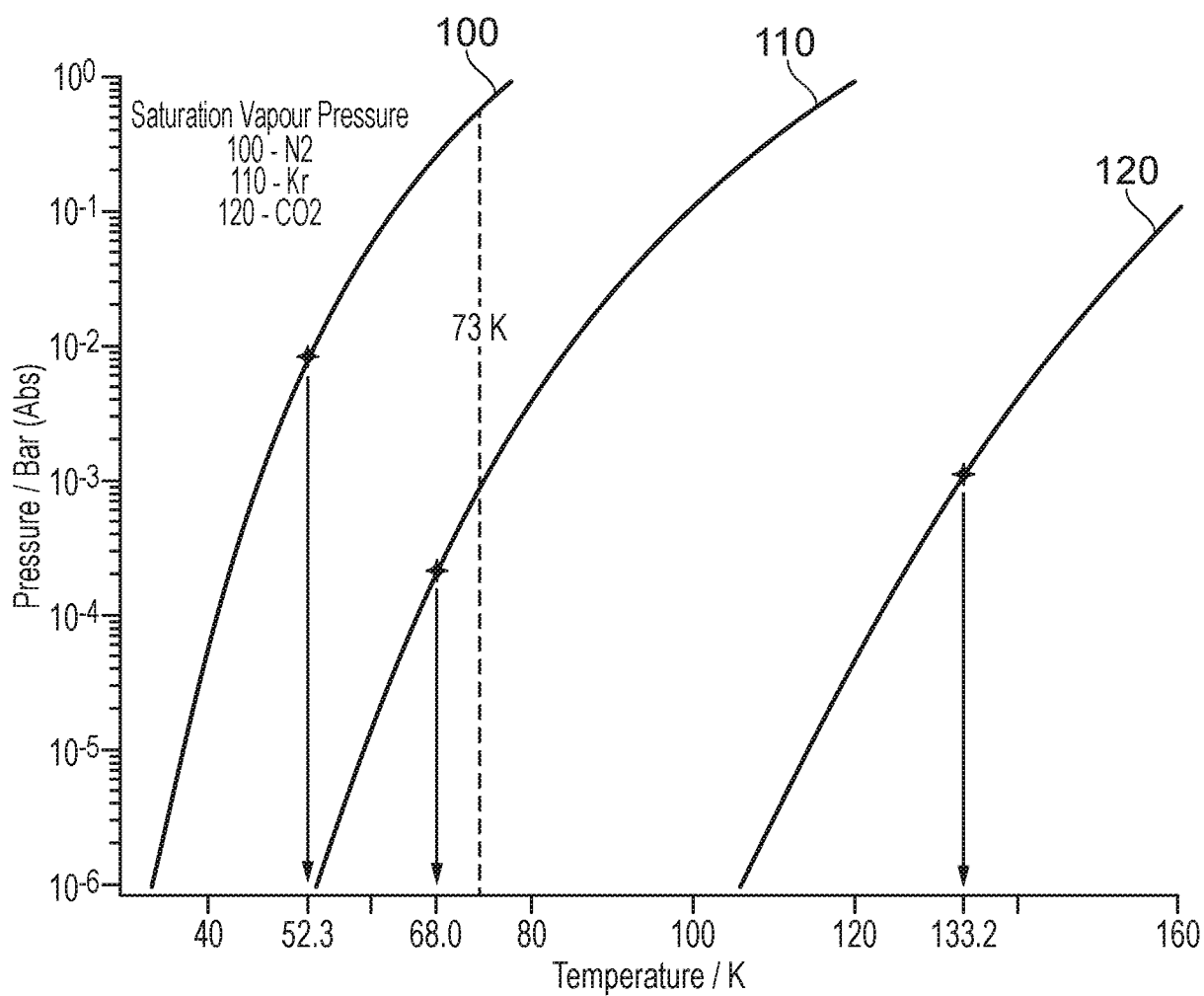


FIG. 2

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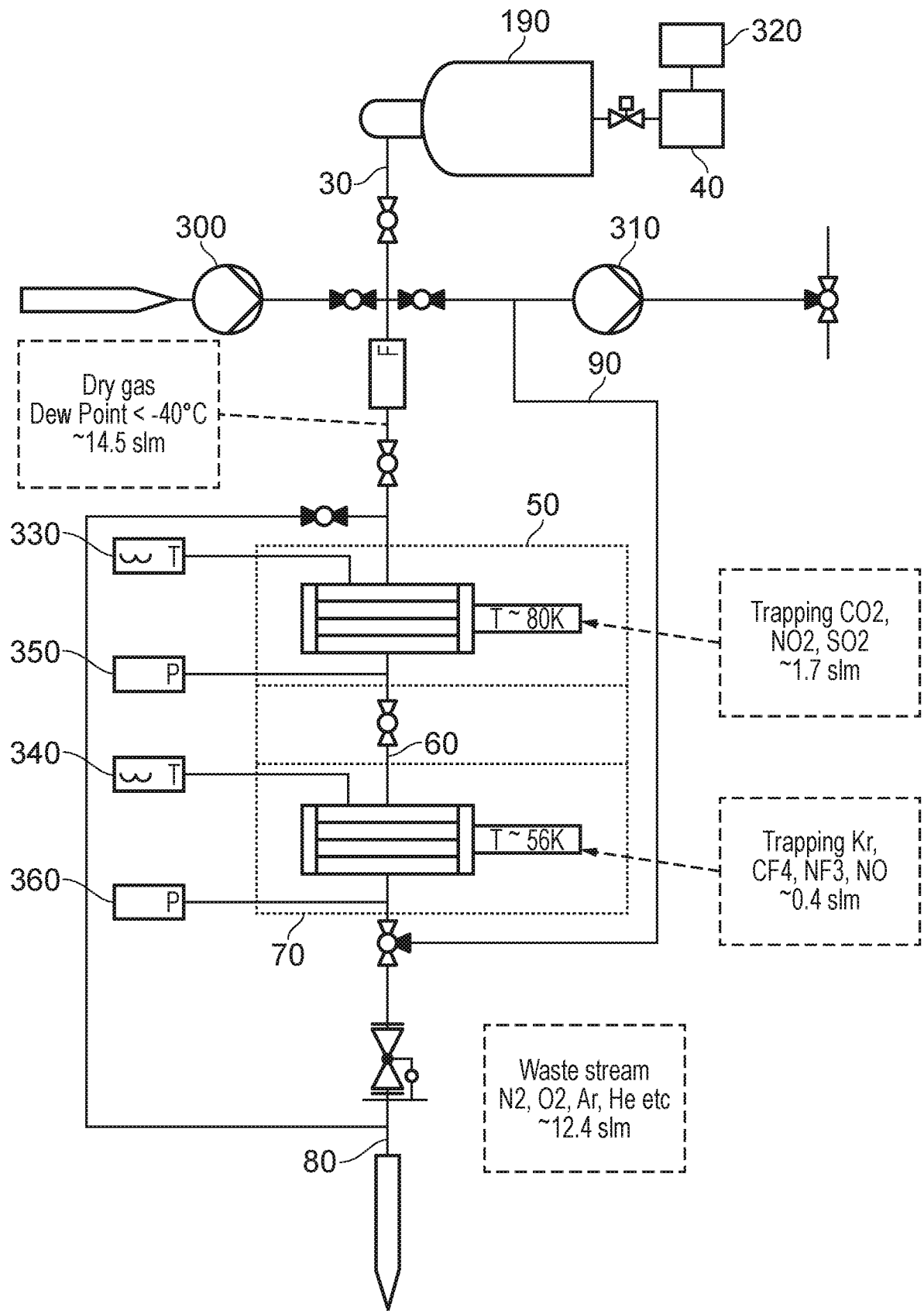


FIG. 3

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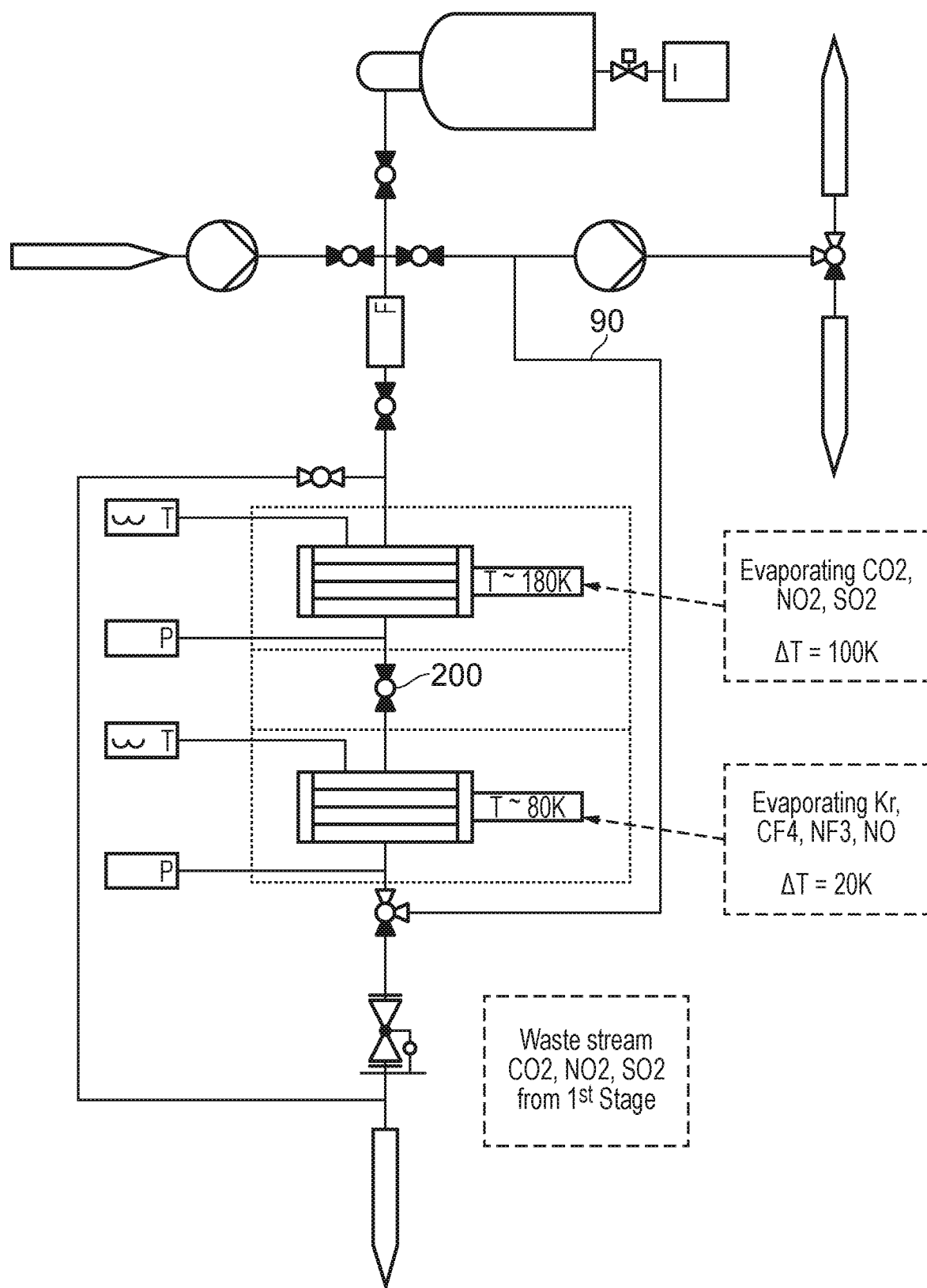


FIG. 4

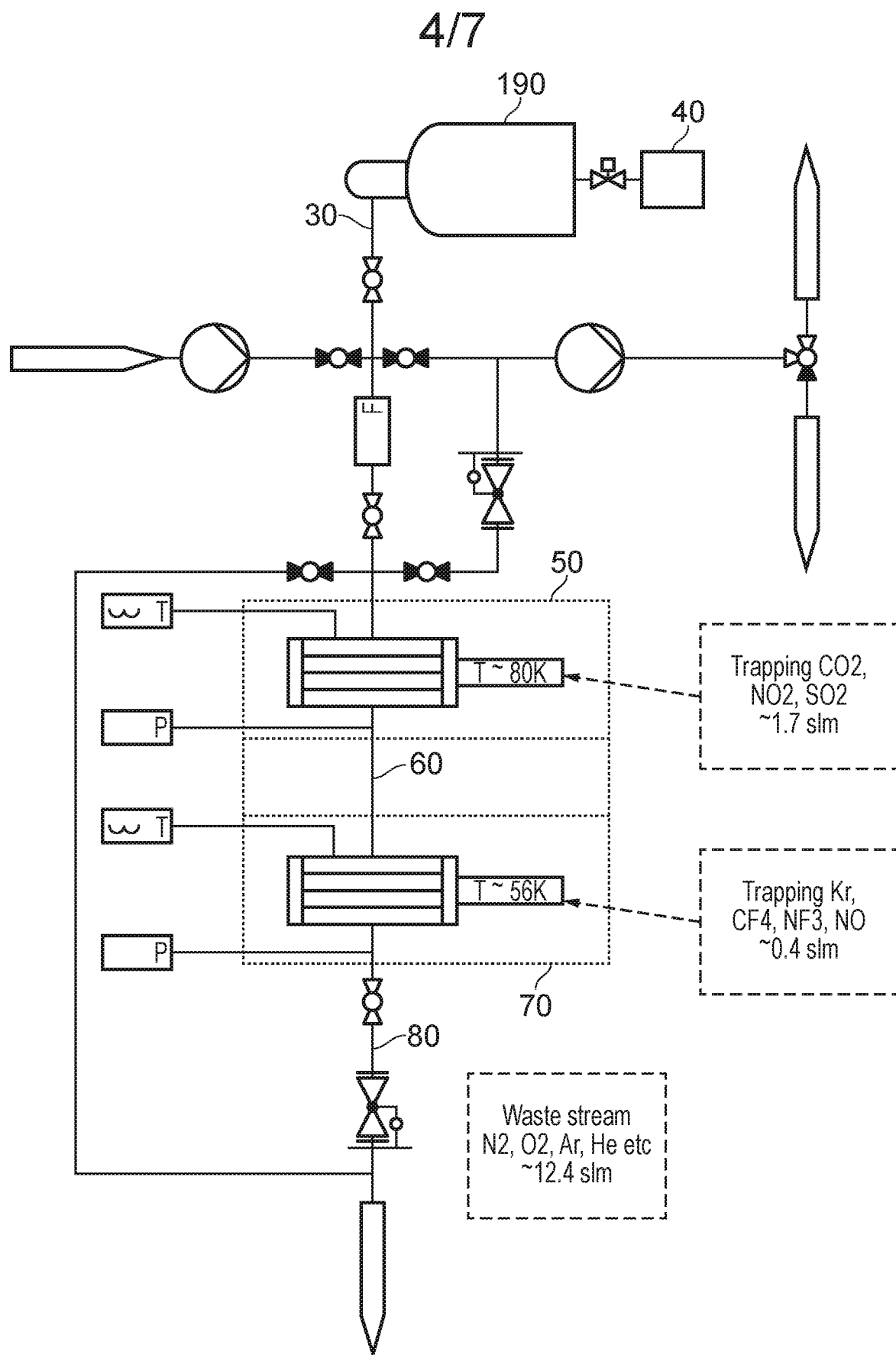


FIG. 5

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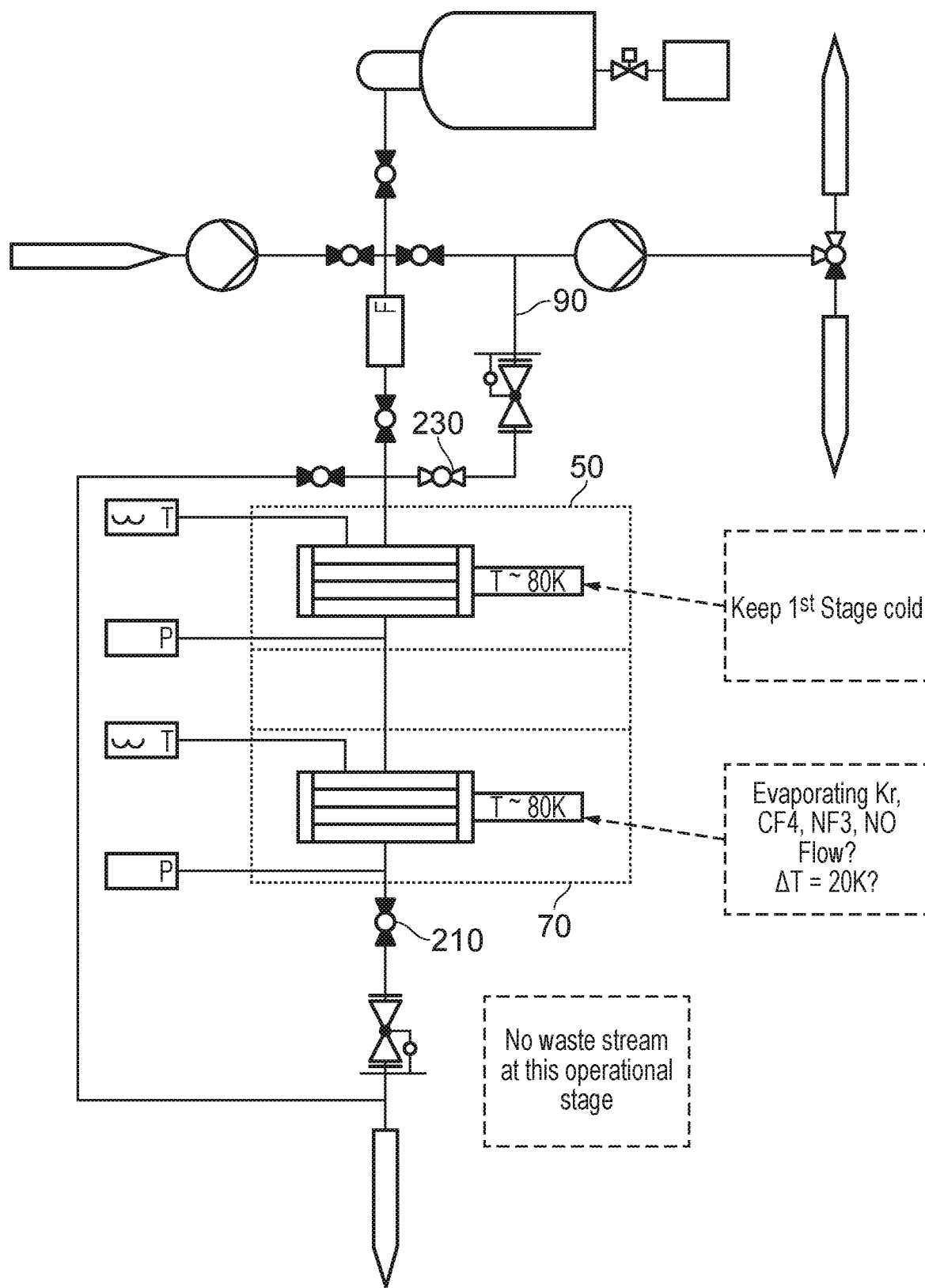


FIG. 6

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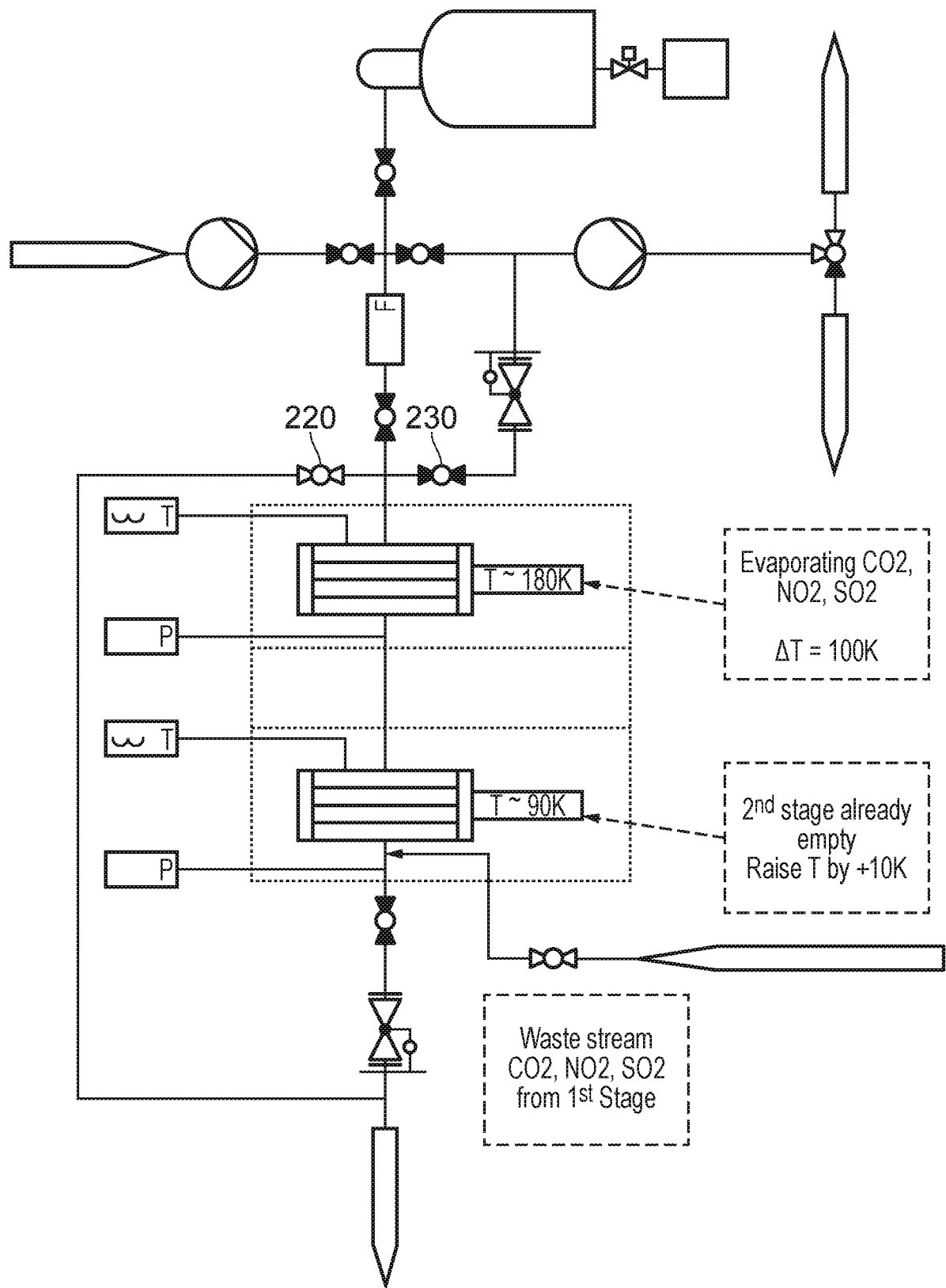


FIG. 7

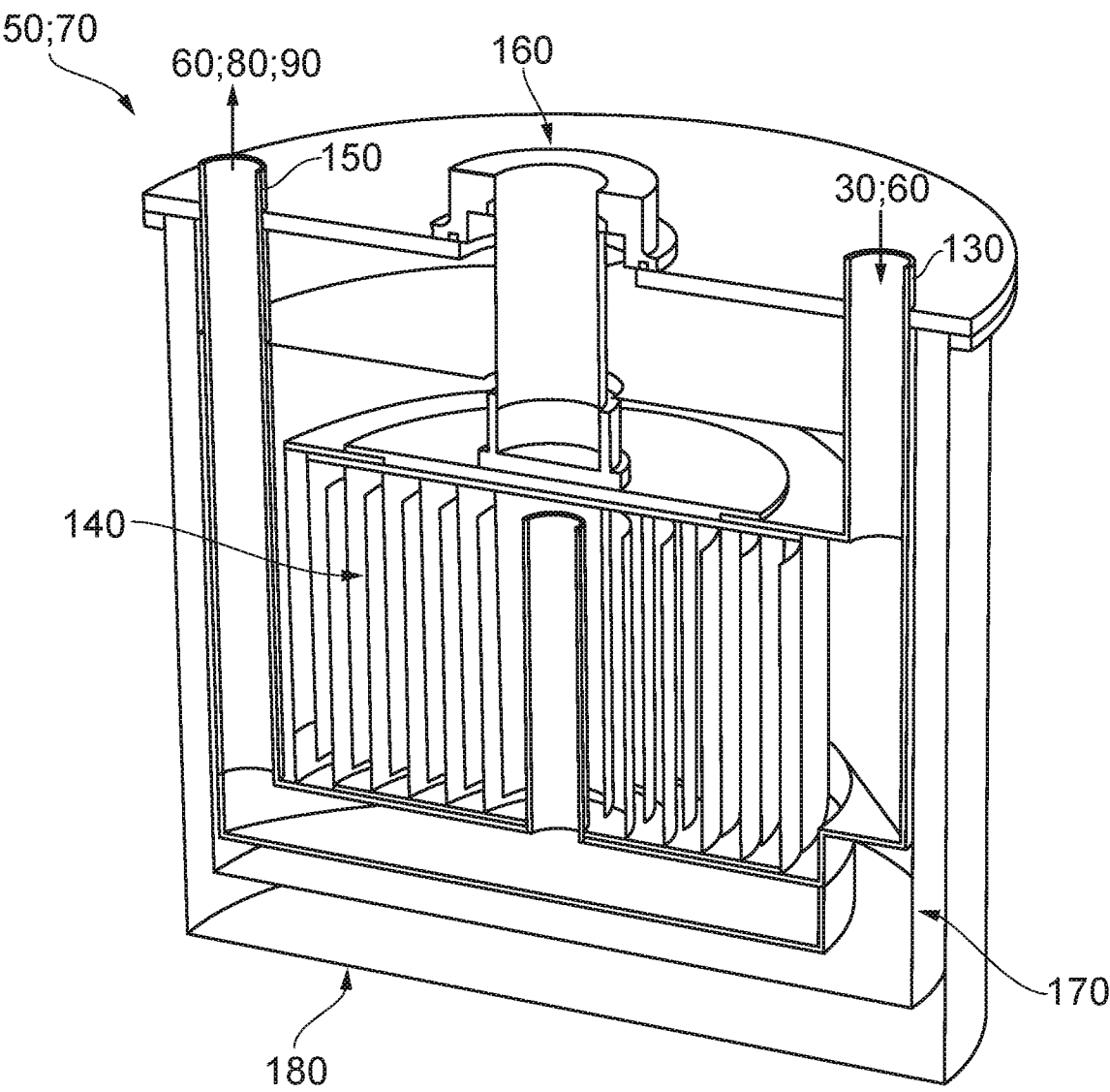


FIG. 8

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2024/051446

A. CLASSIFICATION OF SUBJECT MATTER

INV. B01D53/00 F25J3/06

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B01D F25J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	CN 114 593 560 A (SUZHOU JINMACRO GAS CO LTD) 7 June 2022 (2022-06-07) abstract; figure 1 -----	1, 17, 19-21, 25 2-5, 11-13, 22
X A	US 5 626 035 A (POZVONKOV FELIX [RU]) 6 May 1997 (1997-05-06) claims 9,10; figure 1 column 3, lines 45-50 column 6, lines 16-26, 57-65 -----	1, 6-10, 14-19, 21, 23-25 2-5, 11-13, 22
A	US 4 283 367 A (KOEPPPE WERNER ET AL) 11 August 1981 (1981-08-11) claim 1; figure 1 column 4, lines 11-14 ----- - / - -	22



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance;; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance;; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

7 August 2024

Date of mailing of the international search report

13/09/2024

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INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2024/051446

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	JP S61 140778 A (KOBE STEEL LTD) 27 June 1986 (1986-06-27) abstract; figure 1 -----	1 - 25

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Information on patent family members

International application No

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