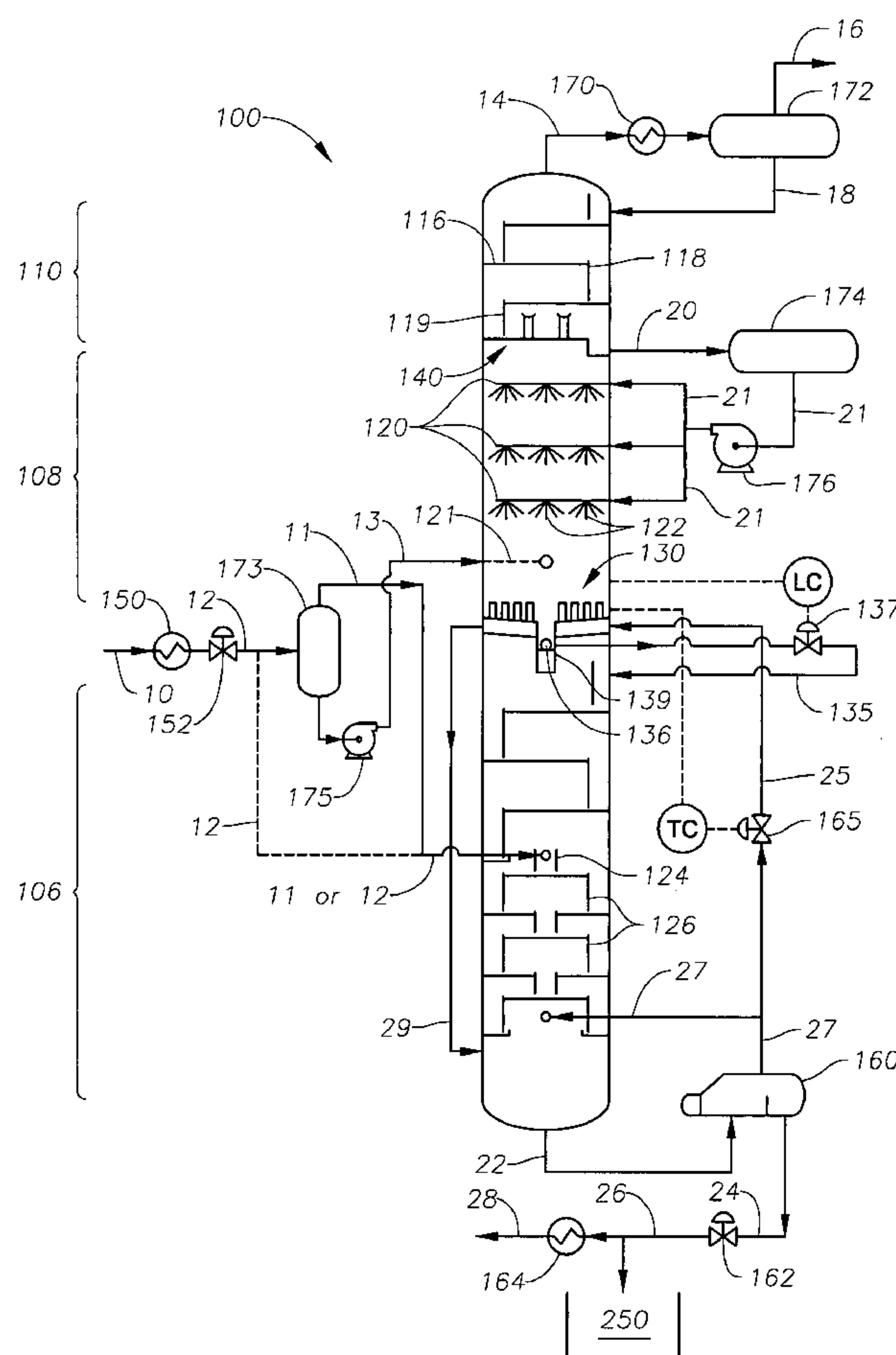


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(19) **United States**(12) **Patent Application Publication**  
**Fieler et al.**(10) **Pub. No.: US 2010/0018248 A1**(43) **Pub. Date: Jan. 28, 2010**(54) **CONTROLLED FREEZE ZONE TOWER****Publication Classification**(76) Inventors: **Eleanor R Fieler**, Humble, TX (US); **Edward J. Grave**, Spring, TX (US); **Paul Scott Northrop**, Spring, TX (US); **Norman K. Yeh**, Houston, TX (US)(51) **Int. Cl.**  
**F25J 3/00** (2006.01)(52) **U.S. Cl.** ..... **62/617**(57) **ABSTRACT**Correspondence Address:  
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Houston, TX 77252-2189 (US)**(21) Appl. No.: **12/518,863**(22) PCT Filed: **Oct. 20, 2007**(86) PCT No.: **PCT/US2007/024216**§ 371 (c)(1),  
(2), (4) Date: **Jun. 11, 2009****Related U.S. Application Data**

(60) Provisional application No. 60/881,395, filed on Jan. 19, 2007.

A cryogenic distillation tower is provided for the separation of a fluid stream containing at least methane and carbon dioxide. The cryogenic distillation tower has a lower stripping section, an upper rectification section, and an intermediate spray section. The intermediate spray section includes a plurality of spray nozzles that inject a liquid freeze zone stream. The nozzles are configured such that substantial liquid coverage is provided across the inner diameter of the intermediate spray section. The liquid freeze zone stream generally includes methane at a temperature and pressure whereby both solid carbon dioxide particles and a methane-enriched vapor stream are formed. The tower may further include one or more baffles below the nozzles to create frictional resistance to the gravitational flow of the liquid freeze zone stream. This aids in the breakout and recovery of methane gas. Additional internal components are provided to improve heat transfer and to facilitate the breakout of methane gas.



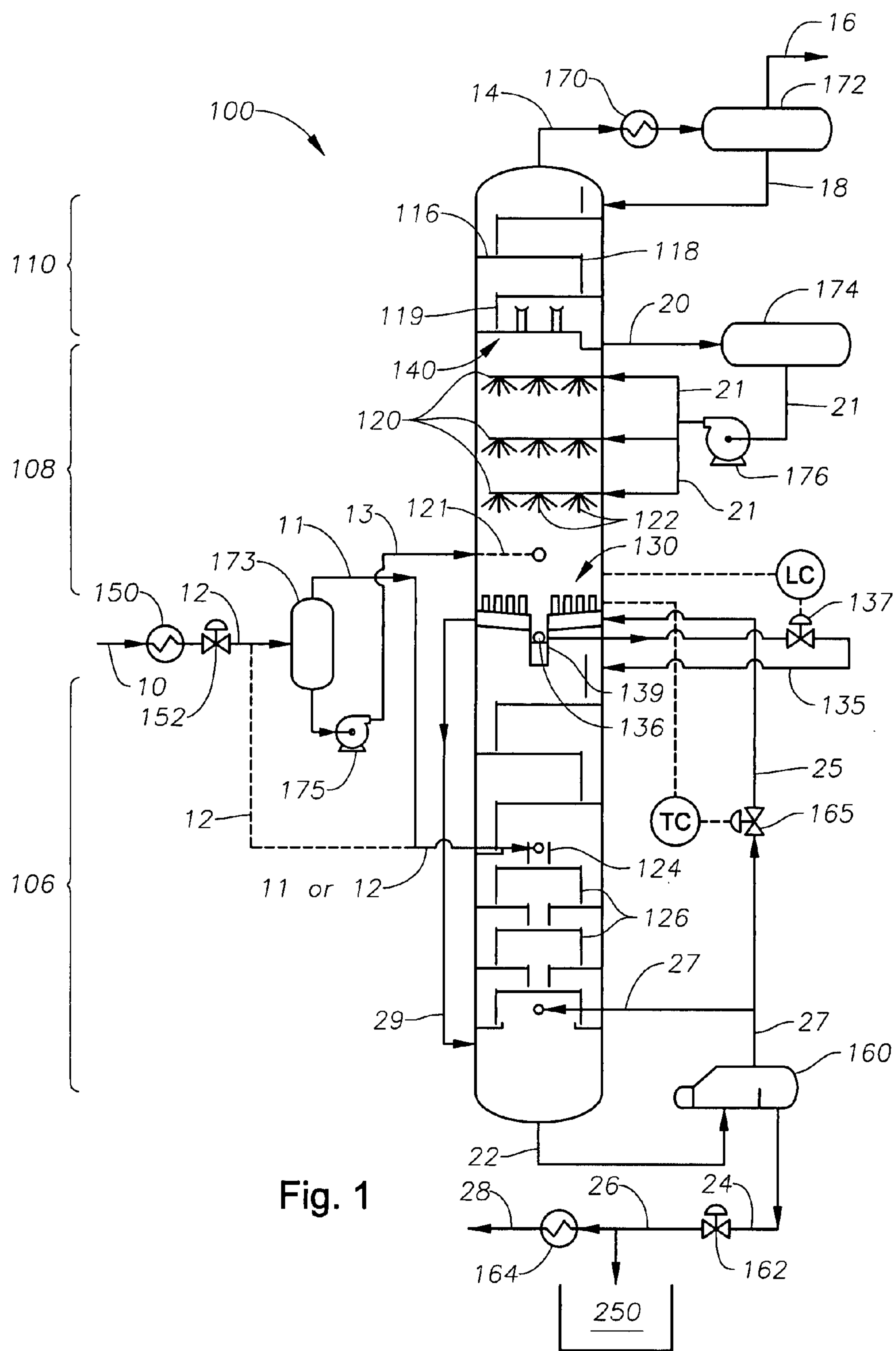
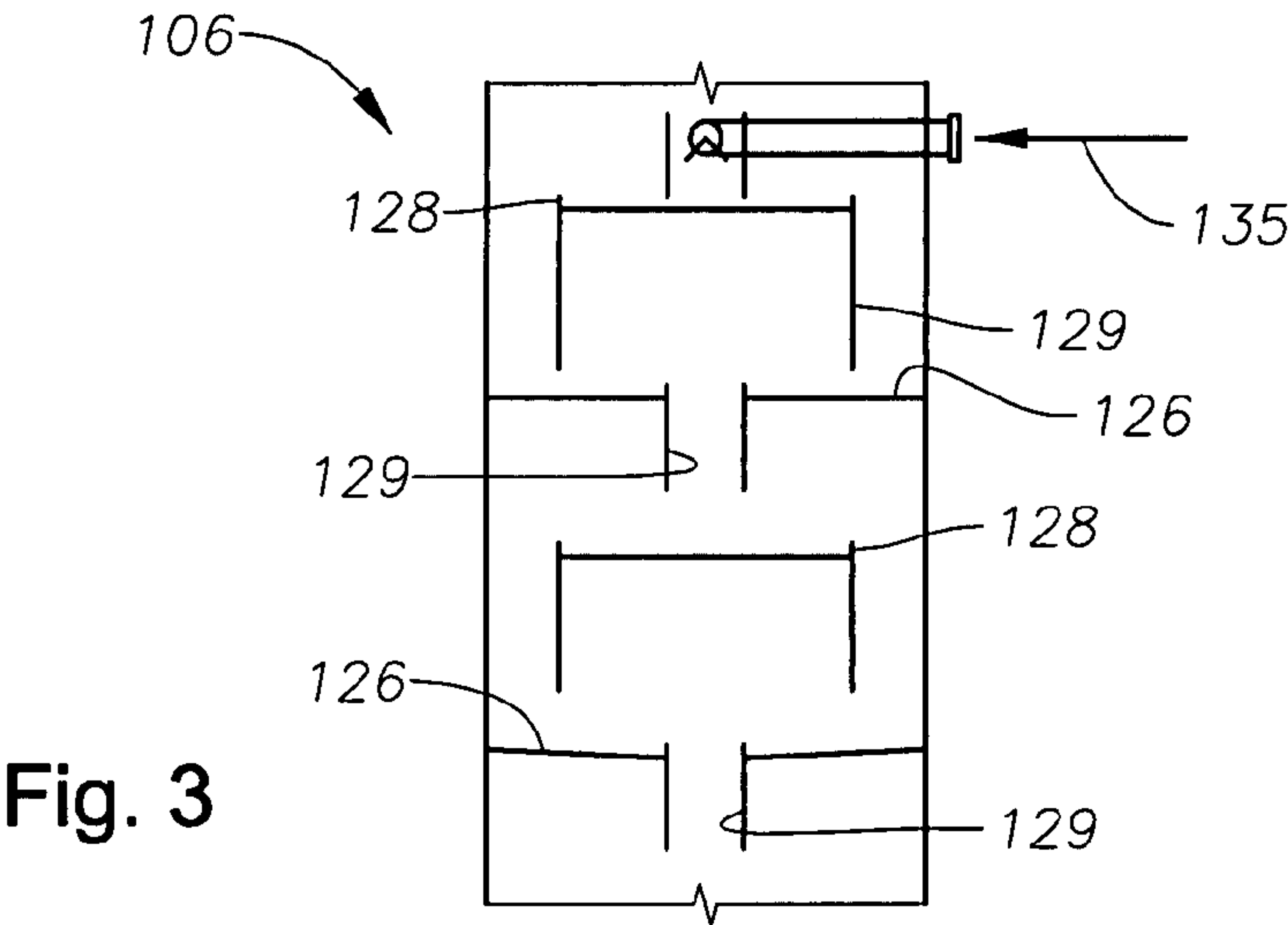
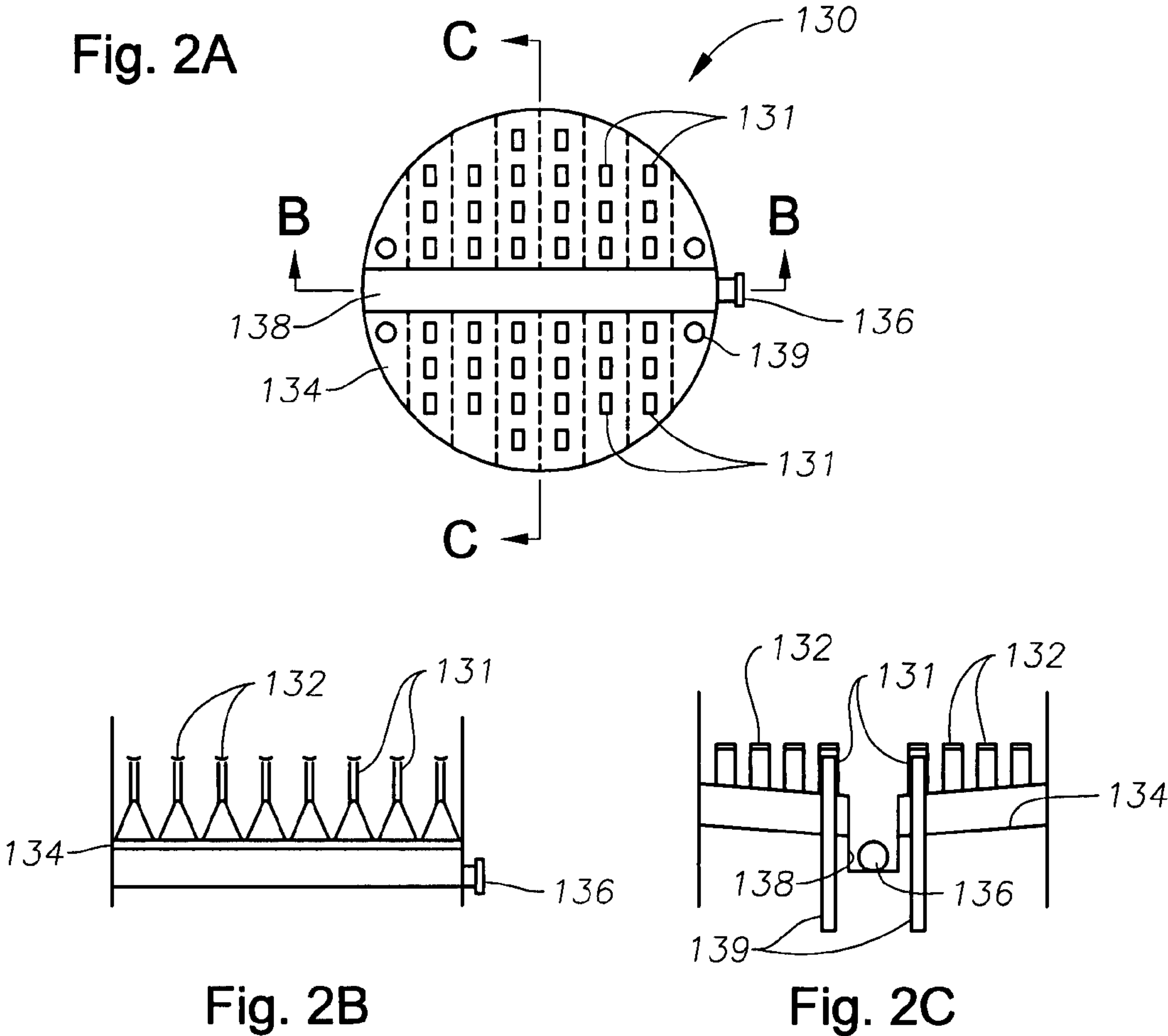


Fig. 1



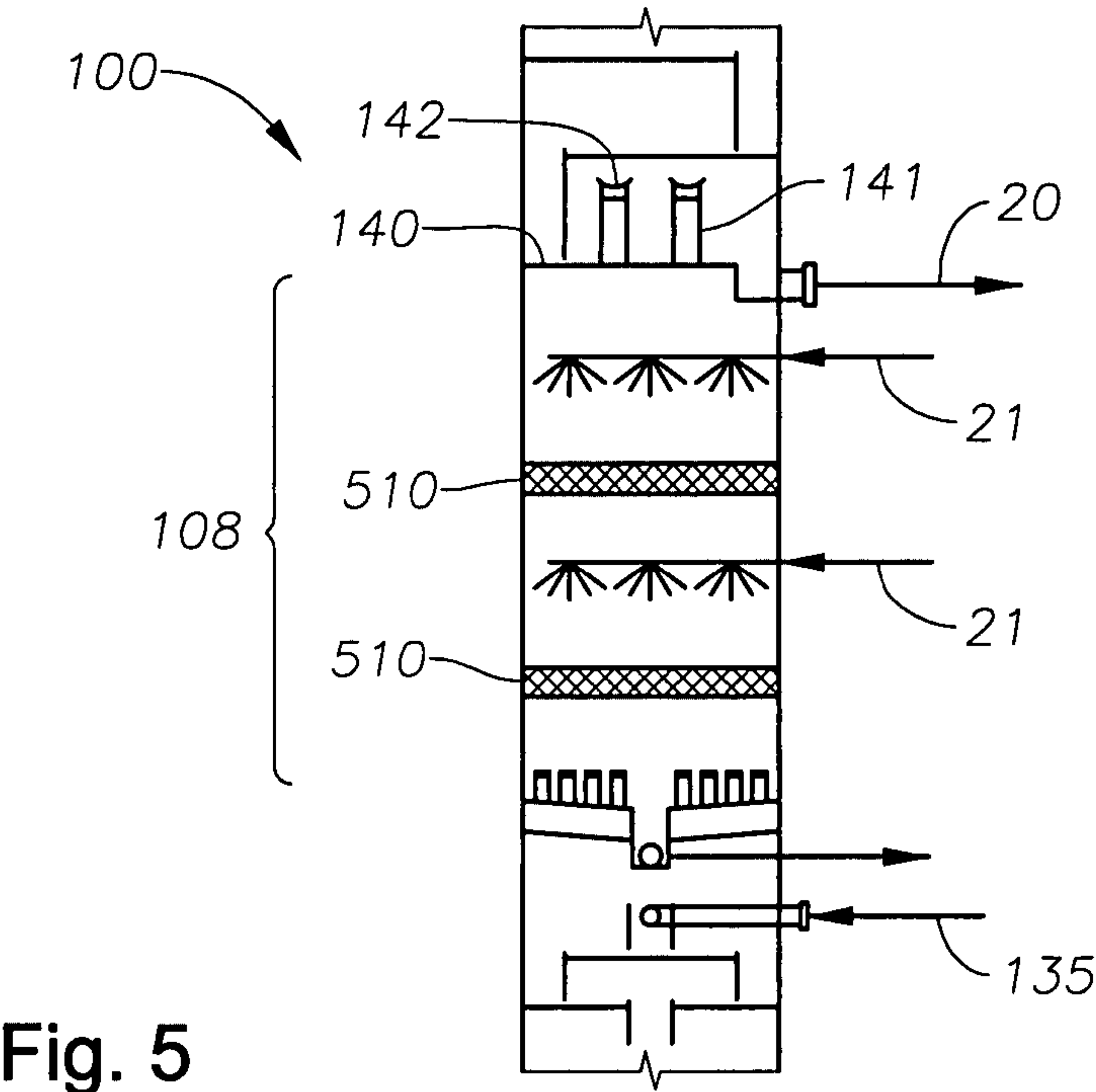
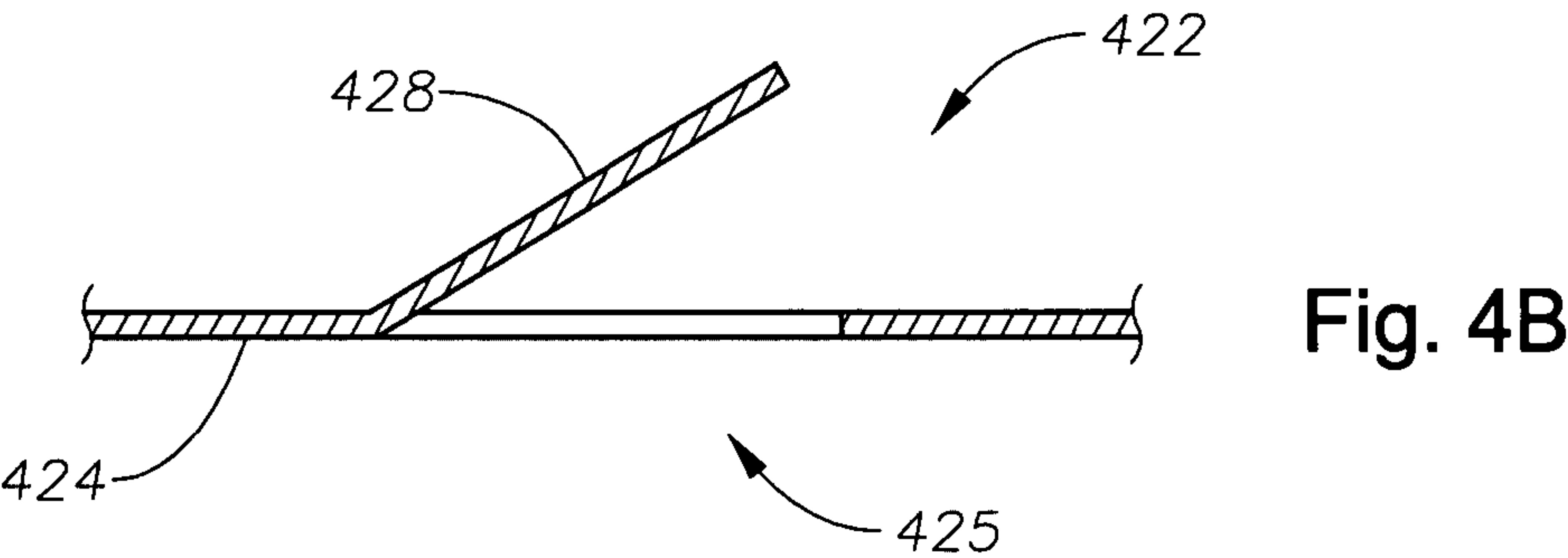
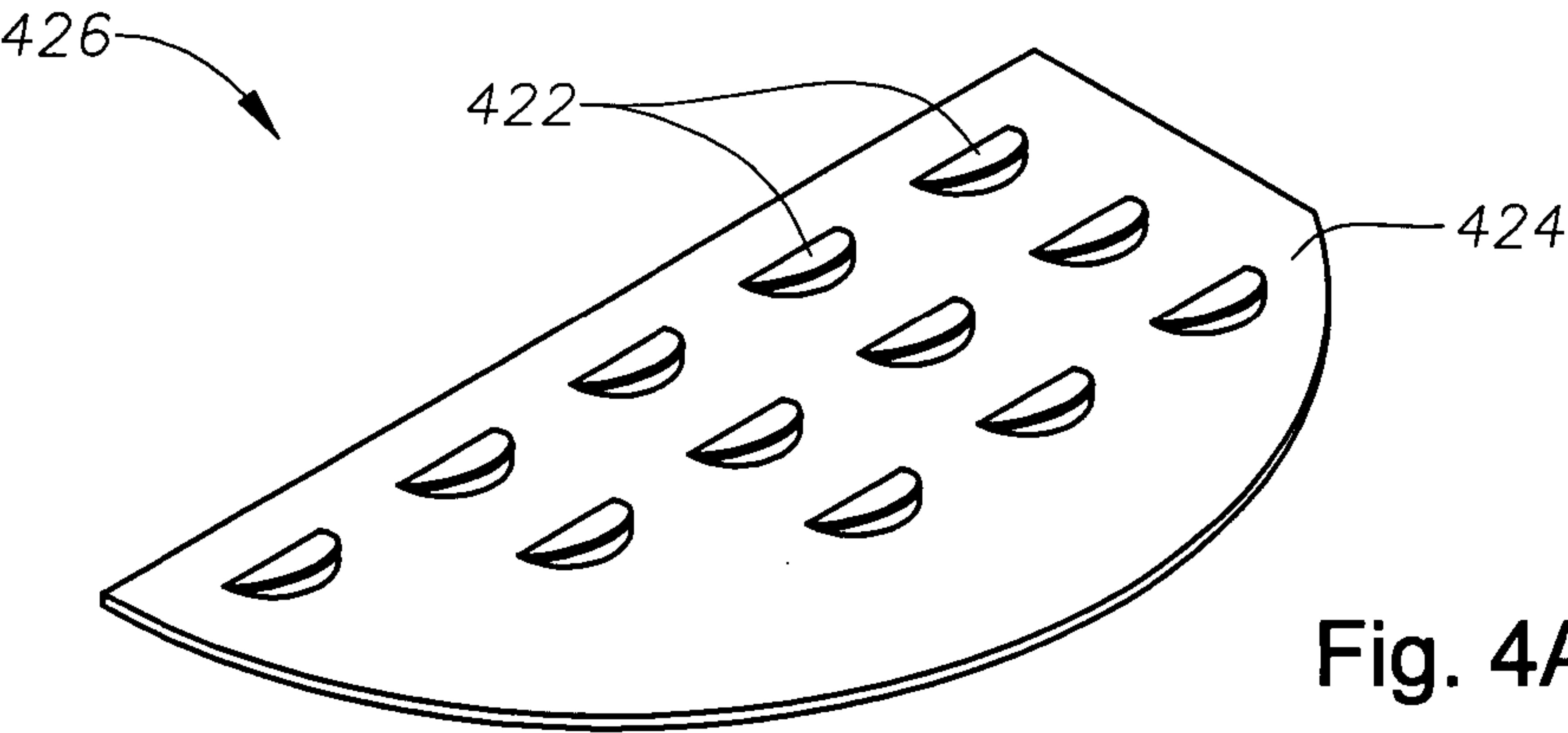


Fig. 6A

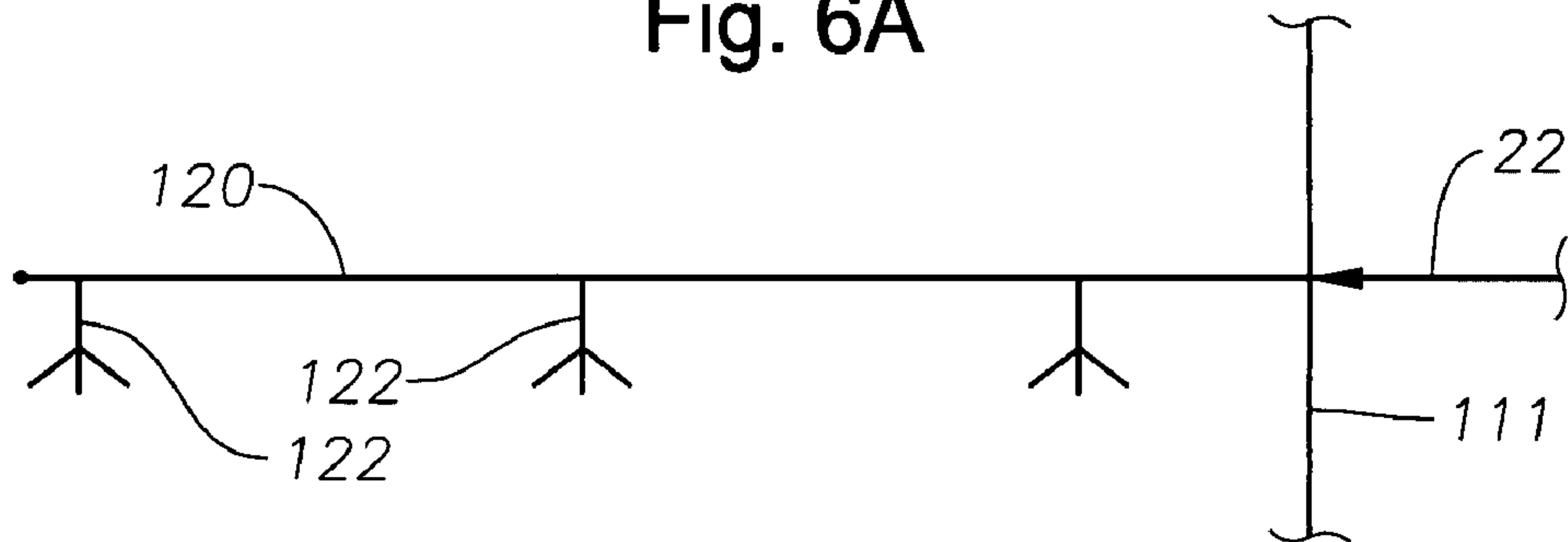
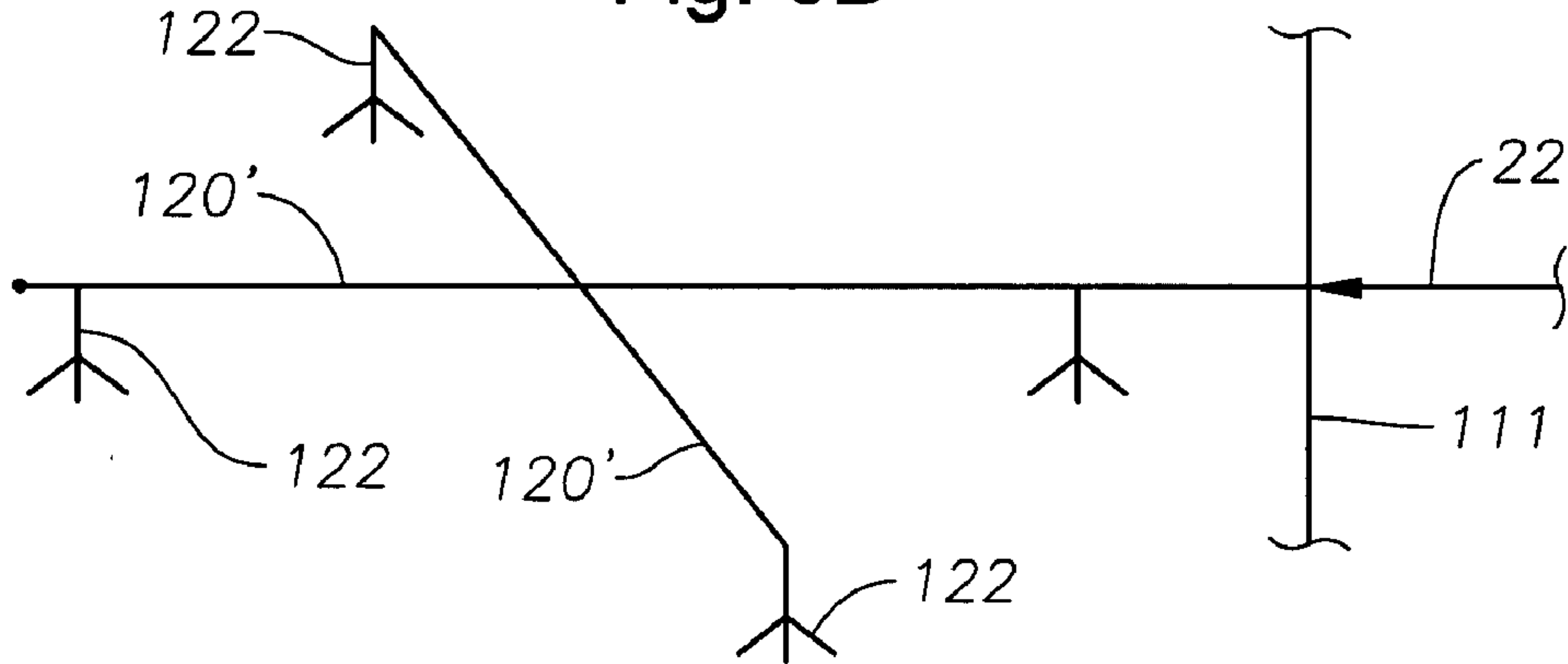


Fig. 6B





## CONTROLLED FREEZE ZONE TOWER

### CROSS REFERENCE TO RELATED APPLICATIONS

**[0001]** This application claims the benefit of U.S. Provisional Application No. 60/881,395, filed 19 Jan. 2007.

**[0002]** This application is related to U.S. Provisional No. 60/881,391, entitled Integrated Controlled Freeze Zone (CFZ) Tower and Dividing Wall (DWC) for Enhanced Hydrocarbon Recovery, filed 19 Jan. 2007, by Vikram Singh, Edward J. Grave, Paul Scott Northrop, and Narasimhan Sundaram.

### BACKGROUND OF THE INVENTION

**[0003]** 1. Field of the Invention

**[0004]** The present invention relates to the field of fluid separation. More specifically, the present invention relates to the cryogenic separation of carbon dioxide from a hydrocarbon fluid stream.

**[0005]** 2. Background of the Invention

**[0006]** The production of hydrocarbons from a reservoir oftentimes carries with it the incidental production of non-hydrocarbon gases. Such gases include contaminants, such as hydrogen sulfide ( $H_2S$ ) and carbon dioxide ( $CO_2$ ). When  $H_2S$  and  $CO_2$  are produced along with a hydrocarbon gas stream (such as methane or ethane), the gas stream is sometimes referred to as “sour gas.”

**[0007]** Sour gas is usually treated to remove  $CO_2$ ,  $H_2S$ , and other contaminants before it is sent downstream for further processing or sale. The separation process creates an issue as to the disposal of the separated contaminants. In some cases, the concentrated acid gas (consisting primarily of  $H_2S$  and  $CO_2$ ) is sent to a sulfur recovery unit (“SRU”). The SRU converts the  $H_2S$  into benign elemental sulfur. However, in some areas (such as the Caspian Sea region), additional elemental sulfur production is undesirable because there is a limited market. Consequently, millions of tons of sulfur have been stored in large, above-ground blocks in some areas of the world, most notably Canada and Kazakhstan.

**[0008]** As for the carbon dioxide gas, it is oftentimes vented to the atmosphere. However, the practice of venting  $CO_2$  is coming under greater scrutiny, particularly in countries that have ratified the Kyoto protocol which requires the reduction of  $CO_2$  emissions. Further,  $CO_2$  gas can and is being sold as a miscible flood agent for enhanced oil recovery. That is, it may not be a disposal prospect, but is potentially a business opportunity to get additional revenue from  $CO_2$  sales and/or additional production from its use.

**[0009]** For these reasons, acid gas injection (“AGI”) may be a preferred alternative to sulfur recovery and  $CO_2$  venting. AGI means that unwanted sour gases are reinjected into a subterranean formation and sequestered for potential later use.

**[0010]** For “highly sour” streams, that is, production streams containing greater than about 15% to about 20%  $CO_2$  and  $H_2S$ , it can be particularly challenging to design, construct, and operate a process that can economically separate these contaminants from the desired hydrocarbons. Cryogenic gas processing (i.e., distillation) is a known procedure for sour gas separation. Cryogenic gas separation avoids the use of solvents, minimizes acid gas removal equipment, and generates a cooled and liquefied acid gas stream at moderate pressures (e.g., 350-500 pounds per square inch gauge

(psig)). The cold, liquefied stream is particularly suitable for injection into a subterranean reservoir through AGI. Because the liquefied acid gas has a relatively high density, a hydrostatic head can be used in an AGI well. That is, the energy required to pump the liquefied acid gas is lower than the energy required to compress low-pressure acid gases to reservoir pressure. In addition, there are fewer stages involving compressors and pumps, so there is less equipment utilized for the acid gas stream.

**[0011]** One problem which has been encountered in the practice of cryogenic gas processing relates to the relatively low temperature at which  $CO_2$  phase changes into a solid. If  $CO_2$  is present at concentrations greater than about 5% in the gas to be processed, it freezes out as a solid in a standard cryogenic distillation unit. The formation of  $CO_2$  as a solid interrupts the cryogenic distillation process. To circumvent this problem, a “Controlled Freeze Zone” (CFZ) process was developed to anticipate the formation of solid  $CO_2$  and captures the frozen  $CO_2$  particles at the bottom of a specially-designed distillation tower. As a result, a clean methane stream (along with any nitrogen or helium present in the raw gas) is generated overhead, while a liquid  $CO_2/H_2S$  stream at 30° to 40° F. (Fahrenheit) is generated at the bottom of the tower. The liquid sour gas stream is thus synergetic with AGI. Certain aspects of the CFZ process, associated equipment and technology are described in U.S. Pat. No. 4,533,372; U.S. Pat. No. 4,923,493; U.S. Pat. No. 5,062,270; U.S. Pat. No. 5,120,338; and U.S. Pat. No. 6,053,007. Each of these patents is incorporated herein by reference in its respective entirety.

**[0012]** The cryogenic distillation column employing the CFZ technology provides a spray zone in the middle of the column. The spray zone (or CFZ section) serves as a freezing section that causes gaseous  $CO_2$  to freeze out of methane (or other light hydrocarbons) within the column, and gravitationally fall. A cold liquid (primarily methane) is injected into the intermediate spray section to provide a liquid spray. The cold liquid within the spray zone contacts gas as it rises in the column, causing the  $CO_2$  to solidify and fall down.

**[0013]** Below the spray zone is a stripping section of the distillation column. The stripping section receives the raw feed stream (gas and liquid) for initial stripping of  $CO_2$  from the gas stream. The bottom of the stripping section is warmer than the feed temperature. Upon entering the column, the liquid portion of the feed stream falls through the stripping section. As it moves downward, dissolved methane vaporizes and rises through the spray section. In the spray zone, entrained  $CO_2$  vapor contacts the cold liquid spray, which causes the  $CO_2$  to freeze out of the gas phase. In some embodiments, the feed stream can be introduced directly into the intermediate spray section.

**[0014]** Above the spray zone is a rectification section of the column. In the rectification section, an overhead gas is captured and made available for fuel or piped away for sales. The overhead gas may be partially liquefied by cooling, and the liquid returned to the column as “reflux.” The reflux liquid is injected as the cold spray into the spray section.

**[0015]** There is a need for improved internal features and components of the CFZ distillation column. More specifically, there is a need for improvements to the CFZ cryogenic distillation tower to facilitate heat and mass transfer within the column. Further, there is a need for a cryogenic distillation tower offering improved internal features above and below the controlled freeze zone section to facilitate the extraction of  $CO_2$  from a gas stream. Still further, there is a need for an



improved sour gas removal chamber producing a liquefied acid gas stream, thereby facilitating the injection and sequestration of  $H_2S$  and  $CO_2$  at low wellhead pressure.

#### SUMMARY OF THE INVENTION

**[0016]** A cryogenic distillation tower is provided for the separation of sour gasses in a fluid stream. The fluid stream contains at least methane and carbon dioxide. The distillation tower has an inner diameter that houses internal components that facilitate the separation process. The tower generally includes a lower stripping section, an intermediate spray section, and an upper rectification section.

**[0017]** The lower stripping section comprises a melt tray, and at least one stripping tray or packed bed below the melt tray. The stripping section is configured to operate at a temperature and pressure at which substantially no carbon dioxide solids are formed, but methane vapor is released.

**[0018]** The intermediate spray section comprises one or more spray nozzles configured to inject a liquid freeze zone stream. The nozzles are uniquely configured such that substantial liquid coverage of the intermediate spray section across the inner diameter is provided. The liquid sprayed into the freeze zone substantially comprises methane at a temperature and pressure whereby both solid carbon dioxide particles and a methane-enriched vapor stream are formed upon injection.

**[0019]** The upper rectification section comprises a rectification tray and, preferably, one or more additional trays, or packing. The upper rectification section is configured to receive vaporized methane from the lower stripping section and the methane-enriched vapor stream from the intermediate spray section. The rectification section may also receive a return liquefied stream of condensed vapor for further capture of residual  $CO_2$  and  $H_2S$ . Unvaporized fluids are collected in the bottom rectification tray, released from the rectification section, optionally further chilled, and then reinjected into the spray section as the liquid freeze zone stream.

**[0020]** As noted, the nozzles in the intermediate spray section are uniquely configured such that substantial liquid coverage of the spray section across the inner diameter is provided. Preferably, the spray nozzles are disposed within one or more spray heads. In one aspect, the one or more spray heads define one or more spray nozzles, each having one or more nozzles and each being positioned at different levels in the spray section. In another aspect, a first spray header forms a substantially linear arrangement of spray nozzles, and a second spray header forms a substantially linear arrangement of spray nozzles transverse to and below the first spray header. In yet another aspect, the spray nozzles are positioned in (i) a first substantially linear spray head having a plurality of nozzles, (ii) a second substantially linear spray header below the first spray header also having a plurality of nozzles, and (iii) a third substantially linear spray head below the second spray header also having a plurality of nozzles, with the first, second and third spray headers being offset from one another.

**[0021]** Preferably, the melt tray in the lower stripping section includes a base, and a plurality of chimneys extending upward from the base. The melt tray preferably also includes a cap over each chimney. In one aspect, the base defines a generally radial base extending across the inner diameter of the distillation tower, with the base having first and second opposite sides sloped inwardly towards an intermediate portion of the base. A downcomer is then disposed within the intermediate portion of the base to receive liquids collected

by the melt tray. In another aspect, the base has a generally sinusoidal or other corrugated profile to increase its surface area.

**[0022]** In similar fashion, the rectification tray preferably includes a base and a plurality of chimneys extending upward from the base. Preferably again, a cap is disposed over each chimney.

**[0023]** Any commercially-available mass transfer device may be employed in either the stripping section or the rectification section. Such mass transfer devices may be fabricated from an anti-sticking material, such as Teflon, or from a fouling-resistant material.

**[0024]** In another embodiment, the spray section further includes one or more baffles or grid-type packing. In one aspect, a baffle is disposed below at least one of the spray headers for creating a fluid path for liquids moving down the spray section.

**[0025]** A method for producing hydrocarbon gases is also provided. In one embodiment, the method includes the steps of receiving a fluid stream containing at least methane and carbon dioxide, and then chilling the fluid stream to substantially liquefied phase. The fluid stream is introduced into a cryogenic distillation tower, such as any of the towers described above. Low molecular weight gases are recovered from the upper rectification section.

#### BRIEF DESCRIPTION OF THE DRAWINGS

**[0026]** So that the manner in which the features of the present invention can be better understood, certain drawings, charts and flow charts are appended hereto. It is to be noted, however, that the drawings illustrate only selected embodiments of the inventions and are therefore not to be considered limiting of scope, for the inventions may admit to other equally effective embodiments and applications.

**[0027]** FIG. 1 is a side view of a controlled freeze zone distillation tower of the present invention, in one embodiment. The initial feed stream is seen being injected into the intermediate spray section of the tower.

**[0028]** FIG. 2A is a plan view of the melt tray, in one embodiment.

**[0029]** FIG. 2B is a cross-sectional view of the melt tray of FIG. 2A taken across line 2B-2B.

**[0030]** FIG. 2C is a cross-sectional view of the melt tray of FIG. 2A taken across line 2C-2C.

**[0031]** FIG. 3 is an enlarged side view of the stripping trays in the stripping section of the distillation tower, in one embodiment.

**[0032]** FIG. 4A is perspective view of a jet tray as may be used in either the stripping section or rectification section of the distillation tower, in one embodiment.

**[0033]** FIG. 4B is a side view of one of the openings in the jet tray of FIG. 4A.

**[0034]** FIG. 5 is a side view of the intermediate spray section of the distillation tower of FIG. 1. In this view, two baffles have been added to the spray section.

**[0035]** FIGS. 6A and 6B are nozzle arrangements, in other embodiments.

**[0036]** FIG. 6A presents a substantially linear spray head supporting three spray nozzles. The spray head may be used in the intermediate spray section of the distillation tower, either to inject the initial feed stream or to inject the liquid freeze zone stream, or both. More than one such spray head arrangement may be placed at different levels in the spray section.



[0037] FIG. 6B presents a pair of transverse, substantially linear spray heads supporting multiple spray nozzles. The spray heads may be used in the intermediate spray section of the distillation tower to inject the initial feed stream. More than one such spray head arrangement may be placed at different levels in the spray section.

#### DETAILED DESCRIPTION OF CERTAIN EMBODIMENTS

##### Definitions

[0038] As used herein, the term “mass transfer device” refers to any object that receives fluid and passes it to another object, such as through gravitational flow. One non-limiting example is a tray for stripping out certain fluids. A grid packing is another example.

[0039] The term “baffle” means any object that interferes with the direct gravitational flow of liquid.

##### Description of Specific Embodiments

[0040] FIG. 1 presents a schematic view of a cryogenic distillation tower of the present invention, in one embodiment. The cryogenic distillation tower is referred to generally as 100. The tower 100 may be interchangeably referred to herein as a “cryogenic distillation tower,” a “column,” a “CFZ column,” or a “splitter tower.”

[0041] The cryogenic distillation tower 100 of FIG. 1 receives an initial fluid stream 10. The fluid stream 10 is comprised of production gasses. Typically, the fluid stream represents a dried gas stream from a wellhead (not shown), and contains about 65% to about 95% methane. Some ethane may be present as a light hydrocarbon, along with certain contaminants, such as CO<sub>2</sub> and H<sub>2</sub>S. Not uncommonly, helium and nitrogen gasses may also be present. The initial fluid stream 10 is at a post-production pressure of approximately 600 pounds per square inch (psi). In some instances, the pressure of initial fluid stream 10 may be up to 1,000 psi.

[0042] The fluid stream 10 is typically chilled before entering the distillation tower 100. A refrigeration unit 150, such as a propane refrigerator, is provided for the initial fluid stream 10, which may be a gas feed stream. The refrigeration unit 150 is used to bring the temperature of the initial fluid stream 10 down to about -30° to about -40° F. The chilled fluid stream may then be moved through an expansion device 152, such as a Joule-Thompson (“J-T”) valve. The expansion device 152 serves as an expander to obtain subcooling and preferably, partial liquification of the feed gas. A J-T valve is preferred for gas feed streams that are prone to forming solids. The expansion device 152 is preferably mounted close to the cryogenic distillation tower 100 to minimize heat loss in the piping.

[0043] As an alternative to a J-T valve, the expander device 152 may be a turbo expander. A turbo expander provides greater cooling and creates a source of shaft work for processes like the refrigeration unit 150. In this manner, the operator may minimize the overall energy requirements for the distillation process. However, because the cryogenic distillation tower 100 is normally used in fields where the operator anticipates a high CO<sub>2</sub> stream, such that solids formation is likely, solids formation may become a matter of concern within not only the cryogenic distillation tower 100, but also within a turbo expander. Thus, the J-T valve is likely the preferred expander device 152.

[0044] In either instance, the refrigeration unit 150 and expander device 152 convert the raw gas in the initial fluid stream 10 into a chilled fluid stream 12 comprised primarily of liquids. Preferably, the temperature of the chilled fluid stream 12 is around -30° to -70° F. In one aspect, the cryogenic distillation tower 100 is operated at a pressure of 550 psi, and the chilled fluid stream 12 is at approximately -62° F. At these conditions, the chilled fluid stream 12 is in a substantially liquid phase, although some vapor phase may inevitably be entrained into the chilled fluid stream 12. Most likely, no solids formation has arisen from the presence of CO<sub>2</sub>.

[0045] The cryogenic distillation tower 100 is divided into three primary sections. These are a stripping section 106, an intermediate spray section or “freeze zone” 108, and a rectification section 110. In the tower arrangement of FIG. 1, the chilled fluid stream 12 is introduced into the distillation tower 100 near the top of the stripping section 106. However, the chilled fluid stream 12 may alternatively be introduced into the spray section 108.

[0046] It is noted in the arrangement of FIG. 1 that the stripping section 106, the intermediate spray section 108, the rectification section 110, and all the components are housed within a single vessel 100. This is believed to be the most economical design. However, for offshore applications in which height of the tower 100 and motion considerations may need to be considered, or for remote locations in which transportation limitations are an issue, the tower 110 may optionally be split into two separate pressure vessels (not shown). For example, the rectification section 110 and the freeze zone 108 may be located in one vessel, while the stripping section 106 is in another section. External piping would then be used to interconnect the two vessels portions.

[0047] In either embodiment, the temperature of the stripping section 106 is higher than the feed temperature of the chilled fluid stream 12. The temperature of the stripping section 106 is designed to be above the vapor point of the methane in the chilled fluid stream 12. In this manner, methane is preferentially stripped from the heavier hydrocarbon and acid liquid components. Of course, those of ordinary skill in the art will understand that the liquid within the distillation tower 100 is a mixture, meaning that the liquid boils at some intermediate temperature between methane and CO<sub>2</sub>. Further, in the event that there are heavier hydrocarbons present in the mixture, this increases the boiling temperature of the mixture. These factors become design considerations for the operating temperatures within the distillation tower 100.

[0048] In the stripping section 106, the CO<sub>2</sub> and any other liquid-phase fluids gravitationally fall towards the bottom of the cryogenic distillation tower 100. At the same time, methane and other vapor-phase fluids break out and rise upwards towards the top of the tower 100. This separation is accomplished primarily through the density differential between the gas and liquid phases. However, the separation process is aided by internal components within the distillation tower 100. As described below, these include a melt tray 130, a plurality of advantageously-configured mass transfer devices 126, and an optional heater line 25.

[0049] Referring again to FIG. 1, the chilled fluid stream 12 may be introduced into the column 100 near the top section of the stripping section 106. Alternatively, it may be desirable to introduce the feed stream 12 into the intermediate spray section 108 above the melt tray 130. Again, the point of injection of the chilled fluid stream 12 is a design issue dictated by the composition of the initial fluid stream 10.



[0050] As with most distillation columns, the feed stream 12 is put onto a tray or region of the column 100 that has similar composition to the feed composition. Thus, where the CO<sub>2</sub> content of the feed stream 12 is high enough (such as greater than 60%) such that solids are not expected, it may be preferable to inject the feed stream 12 directly into the stripping section 106 through a two phase flashbox type device (or vapor distributor) 124 in the column 100. In this case, the expander device 152 could be a turbo expander or a J-T valve or other device. A turbo expander would be preferred as it would extract greater thermal energy from the feed stream 12, as well as recover some work for use elsewhere in the process. If the likelihood of solids formation is significant, the expander device 152 of choice would be a J-T valve. In this case, stream 12 is separated in a two phase vessel 173 to minimize the possibility of solids plugging the inlet line and internals. The vapor leaving the inlet separator 11 is introduced into the column 100 through the vapor distributor 124. The liquid/solid slurry 13 is discharged through an inlet distributor 121 such that it would not be prone to plugging by solids. Inlet feed 13 can be fed to the tower by gravity or by pump 175. Thus, where the initial fluid stream 10 has a sufficiently high CO<sub>2</sub> content such that early solids formation is contemplated, it may be desirable to separate the chilled fluid stream 10 in a separator instead of discharging directly into the lower portion of spray section 108 to avoid vapor maldistribution into the freeze zone 108.

[0051] In the arrangement of FIG. 1, the chilled fluid stream 12 may be distributed directly into the cryogenic distillation tower 100 through one or more two-phase flashboxes 124, as long as significant solids or hydrates are not expected. The feed distributor is slotted such that the two-phase fluid impinges against baffles in the flashbox 124. The use of a flashbox 124 serves to separate the two-phase vapor-liquid mixture in the chilled fluid stream 12. If solids are anticipated, the feed may need to be separated in a vessel 173 prior to feeding the column 100 as described above.

[0052] The chilled fluid stream 12 (or 11) exits the flashbox 124. The location of the feed inlet in section 106 is dependent on feed composition. The liquid leaving the flashbox travels down the stripping trays. Because of the temperature and composition of the chilled fluid stream 12, carbon dioxide primarily exists in liquid form. The vapor leaving the flashbox 124 travels through one or more trays, depending on the composition to maximize mass transfer. The vapor proceeds to travel through the risers of the melt tray 130 and into the freeze zone 108. The melt tray risers act as a vapor distributor for uniform distribution through this zone 108. The vapor contacts the cold liquid from spray headers 120 to “freeze out” the CO<sub>2</sub>. Stated another way, the solid CO<sub>2</sub> “snow” onto the melt tray 130 and then gravitationally flow in liquid form down the melt tray 130 and through the stripping section 106 there below. As will be discussed more fully below, the intermediate spray section 108 is an intermediate freeze zone of the cryogenic distillation tower 100. With the alternate configuration in which feed stream 12 is separated in vessel 173 prior to entering the tower 100, the separated cooled fluid stream 13 is introduced into the tower 100 above the melt tray 130. Thus, a liquid-solid mixture of sour gas and heavier hydrocarbon components flows from the distributor 121 with liquids falling down onto the melt tray 130.

[0053] The melt tray 130 is configured to gravitationally receive liquid and solid materials, primarily CO<sub>2</sub> and H<sub>2</sub>S, from the intermediate spray section 108. The melt tray 130 is

referred to as a “melt tray” as it serves to warm the liquid and solid materials and direct them downward through the stripping section 106 in liquid form for further purification. The melt tray 130 collects and warms the solid-liquid mixture from the intermediate spray section 108 in a pool of liquid. The melt tray 130 is designed to release vapor flow back to the intermediate spray section 108, to provide adequate heat transfer to melt the solid CO<sub>2</sub>, and to facilitate liquid/slurry drainage to the lower distillation or stripping section 106 of the column 100 below the melt tray 130.

[0054] FIG. 2A provides a plan view of the melt tray 130, in one embodiment. FIG. 2B provides a cross-sectional view of the melt tray 130, taken across line 2B-2B of FIG. 2A. FIG. 2C shows a cross-sectional view of the melt tray 130, taken across line 2C-2C. The melt tray 130 will be described with reference to these three drawings collectively.

[0055] First, the melt tray 130 includes a base 134. The base may be a substantially planar body. However, in the preferred embodiment shown in FIGS. 2A, 2B and 2C, the base 134 employs a substantially non-planar profile. The non-planar base configuration provides an increased surface area for contacting liquids and solids landing on the melt tray 130 from the intermediate spray section 108. This serves to increase heat transfer from the vapors passing up from the stripping section 106 of the column 100 to the liquids and thawing solids. In one embodiment, the base 134 is corrugated. In another embodiment, the base 134 is substantially sinusoidal. The corrugated embodiment of the tray design is shown in FIG. 2B. It is understood that other non-planar geometries may be used to increase the heat transfer area of the melt tray 130.

[0056] The melt tray base 134 is preferably inclined. The incline is demonstrated in the side view of FIG. 2C. Although most solids should be melted, the incline serves to ensure that any unmelted solids in the liquid mixture drain off of the melt tray 130 and into the stripping section 106 there below.

[0057] In the view of FIG. 2C, a sump or “downcomer” 138 is seen central to the melt tray 130. The melt tray base 134 slopes inwardly towards the downcomer 138 to deliver the solid-liquid mixture. The base 134 may be sloped in any manner to facilitate liquid draw-off.

[0058] As described in U.S. Pat. No. 4,533,372, the melt tray was referred to as a “chimney tray.” This was due to the presence of a single venting chimney. The chimney provided an opening through which vapors may move upward through the chimney tray. However, the presence of a single chimney meant that all gasses moving upward through the chimney tray had to egress through the single opening. On the other hand, in the melt tray 130 of FIGS. 2A, 2B and 2C, a plurality of chimneys 131 (or “risers”) is provided. The use of multiple chimneys 131 provides improved vapor distribution. This contributes to better heat/mass transfer in the intermediate spray section 108.

[0059] Referring again to FIG. 1, the chimneys 131 may be of any profile. For instance, the chimneys 131 may be round, rectangular, or any other shape that allows vapor to pass through the melt tray 130. The chimneys 131 may also be narrow and extend upwards into the intermediate spray section 108. This enables a beneficial pressure drop to distribute the vapor evenly as it rises into the CFZ intermediate spray section 108. The chimneys 131 are preferably located on peaks of the corrugated base 134 to provide additional heat transfer area.



[0060] Optionally, a heater may be placed below or just above the melt tray base 134 to facilitate thawing of the solid. A heater line 25 is shown and will be discussed further below. The heater line 25 utilizes thermal energy already available from a bottom reboiler 160.

[0061] The top openings of the chimneys 131 are preferably covered with hats or caps 132. This minimizes interaction between the vapor flowing upwards out of the chimneys 131 and the liquid/solid mixture falling onto the melt tray 130. In FIGS. 2A, 2B and 2C, caps 132 are seen above each of the chimneys 131.

[0062] The melt tray 130 may also be designed with bubble caps. The bubble caps define convex indentations in the base 134 rising from underneath the melt tray 130. The bubble caps further increase surface area in the melt tray 130 to provide additional heat transfer to the CO<sub>2</sub>-rich liquid. With this design, a suitable liquid draw off, such as an increase incline angle, should be provided to ensure clear liquid is directed to the stripping trays 126 below.

[0063] The melt tray 130 may also be designed with an internal reflux system. The reflux system serves to ensure that all liquid is substantially free of solids and that sufficient heat transfer has been provided. The reflux system first includes a draw-off nozzle 136. In one embodiment, the draw-off nozzle 136 resides within the draw-off sump 138. Fluids collected in draw-off sump 136 are delivered to a reflux line 135. Flow through reflux line 135 is controlled by a control valve 137 and a level controller "LC." Fluids are returned to the stripping section 106 via the reflux line 135. If the liquid level is too high, the control valve 137 opens; if the level is too low, the control valve 137 closes. If the operator chooses not to employ the reflux system in the stripping section 106, then the control valve 137 is closed and fluids are directed immediately to the mass transfer devices, or "stripping trays" 126 below the melt tray 130 for stripping via an overflow downcomer 139.

[0064] Whether or not an internal reflux system is used, solid CO<sub>2</sub> is warmed on the melt tray 130 and converted to a CO<sub>2</sub>-rich liquid. The melt tray 130 is heated from below by vapors from the stripping section 106. Supplemental heat may optionally be added to the melt tray 130 by various means such as heater line 25. The CO<sub>2</sub>-rich liquid is drawn off from the melt tray 130 under liquid level control and gravitationally introduced to the stripping section 106.

[0065] As noted, a plurality of stripping trays 126 are provided in the stripping section 106 below the melt tray 130. The stripping trays 126 are preferably in a substantially parallel relation, one above the other. Each of the stripping trays 126 may optionally be positioned at a very slight incline, with a weir such that a liquid level is maintained on the tray. Fluids gravitationally flow along each tray, over the weir, and then flow down onto the next tray via a downcomer.

[0066] The stripping trays 126 may be in a variety of arrangements. The stripping trays 126 may be arranged in generally horizontal relation to form a sinusoidal, cascading liquid flow. However, it is preferred that the stripping trays 126 be arranged to create a cascading liquid flow that is divided by separate stripping trays substantially along the same horizontal plane. This is shown in the arrangement of FIG. 3, where the liquid flow is split at least once so that liquid falls over two opposing downcomers 129.

[0067] FIG. 3 provides a side view of a stripping tray 126 arrangement, in one embodiment. Each of the stripping trays 126 receives and collects fluids from above. Each stripping

tray 126 preferably has a weir 128 that serves as a dam to enable the collection of a small pool of fluid on each of the stripping trays 126. The fluid is contacted with upcoming vapor rich in lighter hydrocarbons that strip out the methane from the cross flowing liquid in this "contact area" of the trays 126. The weirs 128 serve to dynamically seal the downcomers 129 to prevent vapor bypassing through the downcomers 129 and to further facilitate the breakout of hydrocarbon gasses.

[0068] The percent of methane becomes increasingly small as the fluids move downward in the stripping section 106. In the upper part of the stripping section 106, the methane content of the liquid may be as high as 25 mol %, while at the bottom stripping tray the methane content may be as low as 0.04 mol %. The methane content flashes out quickly along the stripping trays or packing 126. The number of mass transfer devices 126 used in the stripping section 106 is a matter of composition. However, only a few levels of stripping trays 126 may be utilized to remove methane to a desired level of 1% or less in the liquefied acid gas, for example. The level of purity depends on the number of equilibrium stages in the stripping section.

[0069] Various individual stripping tray 126 configurations that facilitate methane breakout may be employed. The stripping tray 126 may simply be a panel with sieve holes, valves or bubble caps. Sieve holes, valves and bubble caps can be a variety of sizes and open area, depending on hydraulics. However, to provide further heat transfer to the fluid and to prevent unwanted blockage due solids, so called "jet trays" may be employed below the melt tray. In lieu of trays, random or structured packing may also be employed.

[0070] FIG. 4A provides a plan view of an illustrative jet tray 426, in one embodiment. FIG. 4B provides a cross-sectional view of a jet tab 422 from the jet tray 426. As shown, each jet tray 426 has a body 424, with a plurality of jet tabs 422 formed within the body 424. Each jet tab 422 includes an inclined tab member 428 covering an opening 425. Thus, a jet tray 426 has a plurality of small openings 425.

[0071] In operation, one or more jet trays 426 may be located in the stripping 106 or rectification 110 sections of the tower 100 where solids accumulation is possible. The trays 426 may be arranged with multiple passes such as the pattern of stripping trays 126 in FIG. 3. However, any tray or packing arrangement may be utilized that facilitates the breakout of methane gas. Fluid cascades down upon each jet tray 426. The fluids then flow along the body 424. The fluid is then contacted with the vapor exiting the openings 425. The tabs 422 are optimally oriented to move the fluid quickly and efficiently across the tray 426. An adjoined downcomer (not shown) is provided to move the liquid to the subsequent tray 426. The openings 425 also permit gas vapors released during the fluid movement process in the stripping section 106 to travel upwards more efficiently to the melt tray 130 and through the chimneys 131.

[0072] In one aspect, the trays 126 or 426 may be fabricated from fouling-resistant materials, that is, materials that prevent solids-buildup. Fouling-resistant materials are utilized in some processing equipment to prevent the buildup of corrosive metal particles, polymers, salts, hydrates, catalyst fines, or other chemical solids compounds. In the case of the cryogenic distillation tower 100, fouling resistant materials may be used in the trays 126 or 426 to limit sticking of CO<sub>2</sub> solids. For example, a Teflon coating may be applied to the surface of the trays 126 or 426.



[0073] Alternatively, a physical design may be provided to ensure that the CO<sub>2</sub> does not start to build up in solid form along the inner diameter of the column 100. In this respect, the jet tabs 422 may be oriented to push liquid along the wall of the column 100, thereby preventing solids accumulation along the wall of the column 100 and ensuring good vapor liquid contact.

[0074] In any of the tray arrangements, as the fluids hit the stripping trays 126, separation of materials occurs. Methane gasses break out of solution and move upward in vapor form. The CO<sub>2</sub>, however, is cold enough and in high enough concentration that it remains in its liquid form and travels down to the bottom of the stripping section 106. The liquid is then moved out of the cryogenic distillation tower 100 in an exit line as an exit fluid stream 22.

[0075] Upon exiting the distillation tower 100, the exit fluid stream 22 enters a reboiler 160. In FIG. 1, the reboiler 160 is a kettle type that provides reboiled vapor to the bottom of the stripping trays. A heater line 25 from the reboiler vapor stream may be used to provide supplemental heat to the melt tray 130. The supplemental heat is controlled through a valve 165 and temperature controller TC. Alternately, a thermosyphon heat exchanger may be used for the initial fluid stream 10 to economize energy. In this respect, the liquids entering the reboiler 160 remain at a relatively low temperature, for example, about 30° to 40° F. By heat integrating with the initial fluid stream 10, the operator may warm the cold exit fluid stream 22 from the distillation tower 100 while cooling the production fluid stream 10. For this case, the fluid providing supplemental heat through line 25 is a mixed phase return from the reboiler 160.

[0076] It is contemplated that under some conditions, the melt tray 130 may operate without a heater. In these instances, the melt tray 130 may be designed with a heating feature. The heater may be electric. The heating medium may reside external to the cryogenic distillation tower 100. However, it is preferred that a heater be offered that employs the heat energy available in exit fluid stream 22. The vapor stream 25 captured from exit fluid stream 22 after exiting reboiler 160 exists in one aspect at 30° to 40° F., so they contain relative heat energy. Thus, in FIG. 1, vapor stream 25 is shown being directed to the melt tray 130 through a heating coil on the melt tray 130 (not shown). As the supplemental heat stream passes through and warms the melt tray 130, it is return to the tower 100 bottom liquid pool through line 29.

[0077] In operation, reboiled vapor stream 27 is introduced at the bottom of the column, above the bottom liquid level and below the last stripping tray 126. As the reboiled vapor passes through each tray, residual methane is stripped out of the liquid. This vapor cools off as it travels up the tower. By the time the stripping vapors of stream 27 reach the corrugated melt tray 130, the temperature may have dropped to about 0° F. to -20° F. However, this remains quite warm compared to the melting solid on the melt tray 130, which may be around -50° F. to -80° F. The vapor still has enough enthalpy to melt the solids CO<sub>2</sub> as it comes in contact with the melt tray 130 to warm the melt tray 130.

[0078] Referring back to reboiler 160, fluids in a bottom stream 24 that remain in liquid form may optionally enter a valve 162. The valve 162 reduces the pressure of the bottom liquid product, effectively providing a refrigeration effect. Thus, a chilled bottom stream 26 is provided. The bottom stream 24 may be maintained at pressure for direct feed to a pump, then to an acid gas injection (AGI) well.

[0079] The chilled bottom stream 26 may be reinjected in its liquid phase into a geologic reservoir through an AGI well (seen schematically at 250 in FIG. 1). The liquid exiting the reboiler 160 is pumped downhole. In some situations, the liquid CO<sub>2</sub> may be pumped into a partially recovered oil reservoir as part of an enhanced oil recovery process. Thus, the CO<sub>2</sub> could be a miscible injectant. As an alternative, the CO<sub>2</sub> may be used as a miscible flood agent for enhanced oil recovery.

[0080] Some or all of the chilled bottom stream 26 may optionally be moved through a heat exchanger 164. The heat exchanger 164 recovers additional cold from the liquid stream before the CO<sub>2</sub> is warmed into its gas phase for energy recovery. This final stage is shown at stream 28. However, this step is optional, and it is preferred that the CO<sub>2</sub> be reinjected through AGI well 250 in its liquid phase to avoid CO<sub>2</sub> venting.

[0081] Referring again to the stripping section 106 of the column 100, gas moves up through the stripping section 106, through the chimneys 131 in the melt tray 130, and into the intermediate spray section 108. The spray section 108 is an open chamber having a plurality of spray nozzles 122. As the vapor moves through the spray section 108, the temperature in the intermediate spray section 108 becomes colder. As the vapor moves upward, the light gas products are contacted by liquid methane coming from the spray nozzles 122. This liquid methane is colder than the vaporized methane moving upward, having been chilled through a refrigerator 170. In one arrangement, the liquid methane exits from spray nozzles 122 at a temperature of about -120° F. to -130° F.

[0082] As the methane vapors move further up the cryogenic distillation tower 100, they leave the intermediate spray section 108 and enter the rectification section 110. The vapors continue to move upward along with other light gasses broken out from the original chilled fluid stream 12. The combined hydrocarbon vapors move out of the top of the cryogenic distillation tower 100, becoming a condenser exit stream 14, or "condenser feed."

[0083] The hydrocarbon gas in condenser exit stream 14 is moved into the external refrigeration unit 170. In one aspect, the refrigeration unit 170 uses an ethylene refrigerant or other refrigerant capable of chilling the condenser exit stream 14 down to about -138° to -142° F. This serves to at least partially liquefy the condenser exit stream 14. The liquefied condenser exit stream 14 is then moved to a separation chamber 172.

[0084] In another embodiment, the condenser exit stream 14 is taken through an open-loop refrigeration system (not shown). In this alternative arrangement, the condenser exit stream 14 is actually used as a refrigerant. The condenser exit stream 14 is pressurized to about 1,000 psi to 1,400 psi, then cooled using ambient air, and possibly an external propane refrigerant. The chilled and pressurized gas stream is then directed through an expander for further cooling. At this point, a turbo expander could be used to recover even more liquid as well as some shaft work. It is understood here that the present inventions are not limited by the cooling method for the condenser exit stream 14.

[0085] It is also noted that the degree of cooling between refrigerator 170 and the refrigeration unit 150 may be varied. In some instances, it may be desirable to operate the refrigeration unit 150 at a higher temperature, but then be more aggressive with cooling the overhead stream 14 in refrigerator 170. Again, the present inventions are not limited to these types of design choices.



[0086] As noted above, the chilled gas stream exiting refrigerator 170 is directed to a reflux vessel or separation chamber 172. Separation chamber 172 is used to separate gas from liquid. A pump may be used to move liquid back into the column 100 if the separation chamber 172 is not mounted above the column 100 to provide gravity feed of liquid. A portion of the condenser exit stream 14 entering the refrigerator 170 is not condensed; instead, this portion remains in the vapor phase. This portion represents the lighter hydrocarbon gasses, primarily methane. The methane is, of course, the “product” ultimately sought to be captured and sold commercially, along with any ethane. The methane and, perhaps, some ethane, is captured as represented in FIG. 1 by product stream 16. Nitrogen and helium may also be present in product stream 16.

[0087] The remaining liquefied gasses primarily represent residual sour gasses dissolved in methane. The sour gases are liquefied by refrigerator 170, and are directed back to the cryogenic distillation tower 100 for further processing. Optionally, additional heat exchanging may take place to capture cold energy from the condenser exit stream 14.

[0088] It should be noted here that the refrigerator 170 may be referred to as a “reflux condenser.” A reflux condenser is a heat exchanger that causes condensation. This means that a portion of the condenser exit stream 14 coming from the top of the rectification section 110 is condensed, and then returned to the rectification section 110. The condensed gas, or “reflux,” is represented as reflux fluid stream 18. Most of the reflux fluid stream 18 is methane, typically 95% or more, with traces of nitrogen, carbon dioxide and hydrogen sulfide. Sufficient reflux fluid is generated such that the desired CO<sub>2</sub> or H<sub>2</sub>S specification is met at the top of the column 100.

[0089] The reflux fluid stream 18 is returned into the rectification section 110. The reflux fluid stream 18 is then gravitationally carried through one or more mass transfer devices 116 in the rectification section 110. In one embodiment, the mass transfer devices 116 are rectification trays that provide a cascading series of weirs 118 and downcomers 119.

[0090] As fluids from reflux fluid stream 18 move downward through the rectification trays 116, additional methane vaporizes out of the rectification section 110. The methane gasses rejoin the condenser exit stream 14 to become product stream 16. However, the remaining liquid phase of reflux fluid stream 18 falls onto a collector tray 140. As it does so, the reflux fluid stream 18 unavoidably picks up a small percentage of hydrocarbon and residual acid gasses moving upward from the stripping section 106. The liquid mixture of methane and carbon dioxide is collected at collector tray 140.

[0091] The collector tray 140 preferably defines a substantially planar body for collecting liquids. However, as with melt tray 130, collector tray 140 also has one, and preferably a plurality of chimneys for venting gasses coming up from the spray section 108. A chimney and cap arrangement such as that presented by components 131 and 132 in FIG. 2B may be used. Chimneys 141 and caps 142 for collector tray 140 are shown in the enlarged view of FIG. 5, discussed further below.

[0092] It is noted here that in the rectification section 110, any H<sub>2</sub>S present has a slight preference towards being a liquid versus the gas at the processing temperature. In this respect, the H<sub>2</sub>S has a comparatively low relative volatility. By contacting the remaining vapor with more liquid, the cryogenic distillation tower 100 drives the H<sub>2</sub>S concentration down to within the desired parts-per-million (ppm) limit, such as a 4

ppm specification. As fluid moves through the mass transfer devices 116, the H<sub>2</sub>S contacts the liquid methane and is pulled out of the vapor phase and becomes a part of a chamber liquid stream 20. From there, the H<sub>2</sub>S moves in liquid form downward through the stripping section 106 and ultimately exits the cryogenic distillation tower 100 as the liquefied acid gas stream 22.

[0093] In cryogenic distillation tower 100, the liquid captured at collector tray 140 is drawn off of the rectification section 110 as a liquid stream 20. The liquid stream 20 is comprised primarily of methane. In one aspect, the liquid stream 20 is comprised of about 93% methane, 3% CO<sub>2</sub>, 0.5% H<sub>2</sub>S, and 3.5% N<sub>2</sub>. At this point, liquid stream 20 is at about -125° F. to -130° F. This is only slightly warmer than the reflux fluid stream 18. The liquid stream 20 is directed into a reflux drum 174. The purpose of the reflux drum 174 is to provide surge capacity for a pump 176. Upon exiting the reflux drum 174, a spray stream 21 is created. Spray stream 21 is pressurized in a pump 176 for a second reintroduction into the cryogenic distillation tower 100. In this instance, the spray stream 21 is pumped into the intermediate spray section 108 and emitted through nozzles 122.

[0094] Some portion of the spray stream 21, particularly the methane, vaporizes upon exiting the nozzles 122. From there, the methane rises through the intermediate spray section 108, through the chimneys in the collector tray 140, through the rectification mass transfer devices 116 in the rectification section 110, and ultimately become commercial product in product stream 16. However, another portion of the liquid from nozzles 122 vaporizes as it cools upflowing feed gas, causing carbon dioxide to desublime from the gas phase. The CO<sub>2</sub> “snow” falls upon the melt tray 130, and melts into the liquid phase. From there, the CO<sub>2</sub>-rich liquid cascades down mass transfer devices 126 in the stripping section 106 along with liquid CO<sub>2</sub> from the chilled fluid stream 12 described above. At that point, any remaining hydrocarbons from the spray stream 21 of the nozzles 122 should quickly break out into vapor. These vapors move upwards in the cryogenic distillation tower 100 and re-enter the rectification section 110.

[0095] In accordance with one of the many aspects of the present invention, it has been determined that it is desirable to provide some additional form of resistance to fluid flow in the spray section 108 below the nozzles 122. The fluid flow resistance interferes with the flow of the liquid as it moves down the intermediate spray section 108 and onto the melt tray 130. Thus, the cryogenic distillation tower 100 may optionally include baffles or grid packing.

[0096] FIG. 5 provides an enlarged view of the cryogenic distillation tower 100. In this view, the intermediate spray section 108 is primarily seen. Two sets of nozzles 122 are also seen. Further, a pair of illustrative baffles or grid packing 510 is shown disposed within the spray section 108. The baffles or grid packing 510 preferably traverse the diameter of the column 100. In the arrangement of FIG. 5, one of the baffles or grid packing 510 is placed below each of the respective two sets of nozzles 122. As methane-rich liquid is injected from the nozzles 122 into the spray section 108, the fluid contacts the baffles or grid packing 510. This, in turn, facilitates the breakout of methane from solution.

[0097] The baffles or grid packing 510 may take one of any number of shapes and forms. The baffles or grid packing 510 are configured to create a frictional flow path for liquids and snow as the material gravitationally travels down through the



intermediate spray section **108**. As such, the baffles or grid packing **510** may offer angles, grids, perforations or other types of diversion surfaces. In general, the baffles or grid packing **510** should be made to be resistant to fouling as well.

[0098] The use of baffles or grid packing **510** in the intermediate spray section **108** improves the efficiency of fluid separation and heat transfer from the spray droplets to the upflowing gas. Baffles or grid packing **510** further reduce back-mixing of CO<sub>2</sub>. In this respect, the frictional flow path created by the baffles or grid packing **510** prevent the fine, low mass CO<sub>2</sub> particles from moving back up the column **100** and re-entering the rectification section **110**. These particles would undesirably remix with methane and re-enter the condenser exit stream **14**, only to be recycled again.

[0099] Another feature of the column **100** that can improve the efficiency of fluid separation in the intermediate spray section **108** pertains to the configuration of the nozzles **122**. Rather than employing a single spray source at one or more levels in a reflux fluid stream, the present disclosure offers spray headers optionally designed with multiple spray nozzles **122** incorporated therein.

[0100] The configuration of the spray nozzles **122** has an impact on the mass transfer taking place within the intermediate spray section **108**. During the separation process, a chilled liquid stream is injected through the nozzles **122** and into the intermediate spray section **108**. As the liquid enters the intermediate spray section **108** and begins to fall within the cryogenic distillation tower **100**, liquefied methane is evaporated. Some CO<sub>2</sub> momentarily enters the gas phase and moves upward with the methane. However, because of the cold temperature within the intermediate spray section **108**, the vaporized carbon dioxide quickly turns into a solid phase and begins to “snow.” This phenomenon is referred to as desublimation. In this way, some CO<sub>2</sub> never re-enters the liquid phase until it hits the melt tray **130**. The CO<sub>2</sub> particles move down the column **100** in snow form.

[0101] FIGS. 6A and 6B demonstrate various nozzle **122** arrangements in perspective views. In each of FIGS. 6A and 6B, a spray head incorporating multiple spray nozzles **122** is provided. In each Figure, injection line, which is the exit fluid stream **22**, is shown introducing the injectant fluid through a wall **111** of the column **100**.

[0102] FIG. 6A is an enlarged view of the nozzle arrangement from FIG. 1. Here, a spray header **120** supports three nozzles **122** in series. In FIG. 1, two reflux spray headers are provided, each supporting three nozzles **122**. Thus, a 3×2 array is formed. Preferably, each nozzle is capable of providing 70° to 140° spray distribution. These multiple spray headers can be optimized by using a variety of nozzle sizes for turndown operation.

[0103] FIG. 6B presents an alternate spray header **120'** in which two transverse lines support nozzles **122**. Alternatively, three spray headers **120** at different levels in the intermediate spray section **108** may be disposed at 120° (or other) relative angles. Alternatively still, the spray nozzles **122** may be supported by a first substantially linear spray header having a plurality of nozzles **122**, and a second substantially linear spray header transverse to and below the first spray header, also having a plurality of nozzles. These arrangements are not specifically shown but are readily understood from the views of FIG. 1 and FIG. 6B, together. Other spray patterns are possible.

[0104] It is desirable to have chilled liquid contacting as much of the gas that is moving up the column as possible. If

vapor bypasses the sprays, higher levels of CO<sub>2</sub> could reach the rectification section **110** of the tower **100**. The unique spray header arrangements **120**, **120'** (and others described above) avoid dry areas where vapor could bypass the spray. It is preferred that the individual nozzles **122** provide a more limited spray distribution, e.g., 100° to 120°, but that multiple nozzles **122** be used to achieve full distribution. The use of multiple spray nozzles **122** at a level of the intermediate spray section **108** provides fuller, overlapping liquid coverage of the intermediate spray section **108**. This serves to ensure 360° coverage within the spray section **108** and provide good vapor-liquid contact and heat/mass transfer. This, in turn, more effectively chills any gaseous carbon dioxide moving upward through the cryogenic distillation tower **100**.

[0105] The use of an overlapping nozzle **122** arrangement for complete coverage minimizes back-mixing as well. In this respect, complete coverage prevents the fine, low mass CO<sub>2</sub> particles from moving back up the column and re-entering the rectification section **110**. These particles would then remix with methane and re-enter the condenser exit stream **14**, only to be recycled again.

[0106] In one aspect, individual nozzles **122** may be configured to offer a particularly fine or atomized spray distribution. Finer distribution aids hydrocarbons in breaking out of solution as the liquid travels towards the stripping section **106**.

[0107] In yet an additional embodiment, separate spray heads **120** may be used to spray reflux liquid at different temperatures. In this instance, the reflux stream fed into the uppermost spray head is chilled to a temperature below that of the reflux stream fed into the lowermost spray head.

[0108] It can be seen that the process of cycling vapors through the cryogenic distillation tower **100** ultimately produces a hydrocarbon product comprised of methane and ethane gas in product stream **16**. The product is sent down a pipeline or processed to LNG. At the same time, sour gasses are removed through exit fluid stream **22**.

[0109] The above-described internal features are expected to improve mass transfer, heat transfer, and solids handling in the cryogenic distillation tower **100**. They are also expected to improve column operation during upset conditions, when CO<sub>2</sub> levels are not yet stabilized in the cryogenic distillation tower **100**. While it will be apparent that the invention herein described is well calculated to achieve the benefits and advantages set forth above, it will be appreciated that the invention is susceptible to modification, variation and change without departing from the spirit thereof. For example, adjustments may be made to the operational temperatures within the tower **100** to maximize distillation and fluid separation. The temperature ranges disclosed herein are merely exemplary, and it is understood that temperatures could fall outside of these ranges such as during transients or upsets.

What is claimed is:

1. A cryogenic distillation tower for the separation of a fluid stream containing at least methane and carbon dioxide, the cryogenic distillation tower defining an inner diameter that houses internal components, comprising:

a lower stripping section comprising a melt tray and at least one mass transfer device below the melt tray, the lower stripping section being configured to operate at a temperature and pressure at which substantially no carbon dioxide solids are formed, and vaporized methane is released;



- an intermediate spray section comprising a plurality of spray nozzles configured to inject a liquid freeze zone stream such that substantial liquid coverage of the intermediate spray section across the inner diameter is provided, the liquid freeze zone stream substantially comprising methane at a temperature and pressure whereby both solid carbon dioxide particles and a methane-enriched vapor stream are formed upon injection; and
- an upper rectification section comprising a collector tray, and at least one mass transfer device above the collector tray, the upper rectification section being configured to receive the vaporized methane from the lower stripping section and the methane-enriched vapor stream from the intermediate spray section.
2. The cryogenic distillation tower of claim 1, wherein the plurality of spray nozzles is disposed within at least one spray header.
3. The cryogenic distillation tower of claim 2, wherein the plurality of spray nozzles is positioned in a first spray header having a first plurality of nozzles, and a second spray header below the first spray head also having a second plurality of nozzles.
4. The cryogenic distillation tower of claim 1, wherein the plurality of nozzles are positioned at different levels within the upper rectification section.
5. The cryogenic distillation tower of claim 5, wherein the plurality of spray nozzles in the first spray header forms a substantially linear arrangement, and the plurality of spray nozzles in the second spray header forms a substantially linear arrangement essentially transverse to and below the first spray header.
6. The cryogenic distillation tower of claim 5, wherein the plurality of spray nozzles is positioned in:
- a first substantially linear spray header having a first plurality of nozzles,
  - a second substantially linear spray header below the first spray header also having a second plurality of nozzles, and
  - a third substantially linear spray header below the second spray header also having a third plurality of nozzles, with the first spray header, second spray head and third spray header being offset relative to one another.
7. The cryogenic distillation tower of claim 1, wherein the melt tray comprises:
- a base; and
  - a plurality of chimneys extending upward from the base.
8. The cryogenic distillation tower of claim 7, wherein the melt tray further comprises a cap over each of the plurality of chimneys.
9. The cryogenic distillation tower of claim 7, wherein:
- the base of the melt tray defines a generally radial base extending across the inner diameter of the cryogenic distillation tower, the base having a first side, and a second side opposite the first side, wherein the first side and second side are sloped inwardly towards an intermediate portion of the base; and
  - the base further comprises a sump disposed within the intermediate portion of the base to receive and direct liquids.
10. The cryogenic distillation tower of claim 9, wherein the base further comprises a draw-off nozzle for receiving internal reflux fluids collected in the sump.
11. The cryogenic distillation tower of claim 7, wherein the base of the melt tray has a generally sinusoidal profile.

12. The cryogenic distillation tower of claim 7, wherein the base of the melt tray has a generally corrugated profile.
13. The cryogenic distillation tower of claim 7, wherein the at least one mass transfer device below the melt tray comprises:
- a base;
  - a plurality of openings in the base; and
  - a plurality of tabs, wherein each of the plurality of tabs extend over one of the plurality of openings in the base to substantially interfere with the gravitational flow of liquids downward through the one of the plurality of openings.
14. The cryogenic distillation tower of claim 7, wherein the at least one mass transfer device below the melt tray comprises:
- a packing that creates a plurality of channels through which fluids gravitationally flow.
15. The cryogenic distillation tower of claim 1, wherein the collector tray comprises:
- a base; and
  - a plurality of chimneys extending upward from the base.
16. The cryogenic distillation tower of claim 15, wherein the collector tray further comprises a cap over each of the plurality of chimneys.
17. The cryogenic distillation tower of claim 1, wherein the at least one mass transfer devices in the lower stripping section is fabricated from or coated with a fouling-resistant material.
18. The cryogenic distillation tower of claim 1, wherein the at least one mass transfer device above the collector tray comprises:
- a base;
  - a plurality of openings in the base; and
  - a plurality of tabs, wherein each of the plurality of tabs extend over one of the plurality of openings in the base to substantially interfere with the gravitational flow of liquids downward through the one of the plurality of openings.
19. The cryogenic distillation tower of claim 1, wherein the at least one mass transfer device above the collector tray comprises:
- a packing that creates a plurality of channels through which fluids gravitationally flow.
20. The cryogenic distillation tower of claim 1, wherein the at least one mass transfer device in the lower stripping section or in the upper rectification section above the rectification tray is fabricated from or coated with a fouling-resistant material.
21. The cryogenic distillation tower of claim 4, further comprising a baffle or grid packing below at least one of the plurality of spray headers for creating a frictional fluid path for liquids moving down the intermediate spray section.
22. The cryogenic distillation tower of claim 1, wherein the at least one stripping tray comprises a plurality of stripping trays arranged to create a cascading liquid flow.
23. The cryogenic distillation tower of claim 1, further comprising:
- an exit line which receives an exit fluid stream from the lower stripping section comprised primarily of carbon dioxide;
  - a reboiler wherein the exit fluids are warmed; and
  - a heater line for capturing gas vaporized by the reboiler, the vapor line carrying warmed vapor to the melt tray to heat the melt tray.



**24.** The cryogenic distillation tower of claim **1**, wherein the melt tray comprises a plurality of bubble caps to increase the surface area.

**25.** The cryogenic distillation tower of claim **2**, wherein the plurality of spray nozzles is arranged in:

- a first spray header to distribute a portion of the liquid freeze zone stream at a first temperature; and
- a second spray header positioned at a different level in the intermediate spray section to distribute a portion of the liquid freeze zone stream at a second temperature that is higher than the first temperature.

**26.** The cryogenic distillation tower of claim **25**, wherein the first spray header is positioned above the second spray header.

**27.** The cryogenic distillation tower of claim **1**, further comprising an internal downcomer on the melt tray to provide reflux to the stripping section.

**28.** The cryogenic distillation tower of claim **1**, wherein the fluid stream is injected into the cryogenic distillation tower through at least one spray header having a plurality of nozzles.

**29.** The cryogenic distillation tower of claim **28**, wherein the at least one spray header comprises a first spray header, and a second spray header below the first spray header.

**30.** A method for producing hydrocarbon gases, comprising:

- receiving an initial fluid stream containing at least methane and carbon dioxide;
- chilling the initial fluid stream to a substantially liquefied phase;

introducing the liquefied fluid stream into a cryogenic distillation tower to separate the carbon dioxide from the methane, the distillation tower defining an inner diameter which houses internal components comprising:

- a lower stripping section comprising a melt tray and at least one mass transfer device below the melt tray, the lower stripping section being configured to operate at a temperature and pressure at which substantially no carbon dioxide solids are formed and vaporized methane is released;

- an intermediate spray section comprising a plurality of spray nozzles configured to inject a spray stream such that substantial liquid coverage of the intermediate spray section across the inner diameter is provided, the spray stream substantially comprising methane at a temperature and pressure whereby both solid carbon dioxide particles and a methane-enriched vapor stream are formed upon injection; and

- an upper rectification section comprising a collector tray, the upper rectification section being configured to receive vaporized methane from the lower stripping section and the methane-enriched vapor stream from the intermediate spray section; and

recovering vaporized methane from the upper rectification section.

**31.** The method of claim **30**, wherein the plurality of spray nozzles is disposed within at least one spray header.

**32.** The method of claim **31**, wherein the cryogenic distillation tower further comprises a baffle below at least one of the spray headers for creating a fluid path for liquids moving down the intermediate spray section.

**33.** The method of claim **30**, wherein the plurality of spray nozzles is positioned in a first spray head having a first plu-

rality of nozzles, and a second spray header below the first spray header also having a second plurality of nozzles.

**34.** The method of claim **33**, wherein the plurality of spray nozzles in the first spray header forms a substantially linear arrangement, and the plurality of spray nozzles in the second spray header forms a substantially linear arrangement essentially transverse to and below the first spray header.

**35.** The method of claim **33**, wherein the plurality of spray nozzles is positioned in:

- a first substantially linear spray header having a first plurality of nozzles,
- a second substantially linear spray header below the first spray header also having a second plurality of nozzles, and
- a third substantially linear spray header below the second spray header also having a third plurality of nozzles, with the first spray header, second spray header and third spray header being offset relative to one another.

**36.** The method of claim **30**, wherein the spray nozzles are each capable of providing about a 70° to 140° spray distribution.

**37.** The method of claim **30**, wherein the spray header defines at least two intersecting fluid lines supporting nozzles.

**38.** The method of claim **30**, wherein the melt tray comprises:

- a base; and
- a plurality of chimneys extending upward from the base.

**39.** The method of claim **38** wherein the melt tray further comprises a cap over each chimney.

**40.** The method of claim **30**, wherein the at least one mass transfer device below the melt tray comprises:

- a base;
- a plurality of openings in the base; and
- tabs extending over the respective openings in the base to substantially interfere with the gravitational flow of liquids downward through the openings.

**41.** The method of claim **40**, wherein the melt tray is fabricated from or coated with a fouling-resistant material.

**42.** The method of claim **30**, wherein the at least one mass transfer device below the melt tray comprises a plurality of stripping trays arranged to create a cascading liquid flow.

**43.** The method of claim **30**, wherein the cryogenic distillation tower further comprises:

- an exit line which receives exit fluids from the stripping section comprised primarily of carbon dioxide; and
- a reboiler wherein exit fluids are warmed.

**44.** The method of claim **43**, wherein the cryogenic distillation tower further comprises

- a heater line for capturing gas vaporized by the reboiler, the vapor line carrying warmed vapor to the melt tray to heat the melt tray.

**45.** The method of claim **43**, further comprising: collecting the carbon dioxide for use as a miscible agent in an enhanced oil recovery operation.

**46.** The method claim **30**, wherein the melt tray comprises a plurality of bubble caps to increase the surface area.

**47.** The method of claim **30**, wherein the spray nozzles are arranged in:

- a first spray header to distribute a portion of the liquid stream at a first temperature; and
- a second spray header positioned at a different level in the intermediate spray section to distribute a portion of the liquid stream at a second temperature that is higher than the first temperature.

**48.** The method of claim **47**, wherein the first spray header is positioned above the second spray header.