

METHOD AND SYSTEM FOR SEPARATING FLUIDS IN A DISTILLATION TOWER

CROSS REFERENCE TO RELATED APPLICATIONS

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[0001] This application claims the priority benefit of United States patent application number 62/022072 filed July 8, 2014 entitled METHOD AND SYSTEM FOR SEPARATING FLUIDS IN A DISTILLATION TOWER, the entirety of which is incorporated by reference herein.

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BACKGROUND

Fields of Disclosure

[0002] The disclosure relates generally to the field of fluid separation. More specifically, the disclosure relates to a method and system for separating fluids in a distillation tower, such as but not limited to during start-up of a distillation tower.

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Description of Related Art

[0003] This section is intended to introduce various aspects of the art, which may be associated with the present disclosure. This discussion is intended to provide a framework to facilitate a better understanding of particular aspects of the present disclosure. Accordingly, it should be understood that this section should be read in this light, and not necessarily as admissions of prior art.

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[0004] The production of natural gas hydrocarbons, such as methane and ethane, from a reservoir oftentimes carries with it the incidental production of non-hydrocarbon gases. Such gases include contaminants, such as at least one of carbon dioxide ("CO₂"), hydrogen sulfide ("H₂S"), carbonyl sulfide, carbon disulfide, and various mercaptans. When a feed stream being produced from a reservoir includes these contaminants mixed with hydrocarbons, the stream is oftentimes referred to as "sour gas."

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[0005] Many natural gas reservoirs have relatively low percentages of hydrocarbons and relatively high percentages of contaminants. Contaminants may act as a diluent and lower the heat content of hydrocarbons. Additionally, in the presence of water some contaminants can become quite corrosive.

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[0006] It is desirable to remove contaminants from a stream containing hydrocarbons to produce sweet and concentrated hydrocarbons. Specifications for pipeline quality natural gas typically call for a maximum of 2 – 4% CO₂ and ¼ grain H₂S per 100 standard cubic feet (scf) (4 parts per million volume (ppmv)) or 5 milligrams per normal meter cubed (mg/Nm³)

H₂S. Specifications for lower temperature processes such as natural gas liquefaction plants or nitrogen rejection units typically require less than 50 parts per million (ppm) CO₂.

[0007] Separating contaminants from hydrocarbons is difficult. Consequently, significant work has been applied to the development of hydrocarbon/contaminant separation methods.

5 These methods can be placed into three general classes: absorption by solvents (physical, chemical, and hybrids), adsorption by solids, and distillation.

[0008] Separation by distillation of some mixtures can be relatively simple and, as such, is widely used in the natural gas industry. However, distillation of mixtures of natural gas hydrocarbons, primarily methane, and one of the most common contaminants in natural gas, carbon dioxide, can present significant difficulties. Conventional distillation principles and conventional distillation equipment are predicated on the presence of only vapor and liquid phases throughout the distillation tower. The separation of CO₂ from methane by distillation involves temperature and pressure conditions that result in solidification of CO₂ if a pipeline or better quality hydrocarbon product is desired. The required temperatures are cold
15 temperatures typically referred to as cryogenic temperatures.

[0009] Certain cryogenic distillations can overcome the above mentioned difficulties. These cryogenic distillations provide the appropriate mechanism to handle the formation and subsequent melting of solids during the separation of solid-forming contaminants from hydrocarbons. The formation of solid contaminants in equilibrium with vapor-liquid
20 mixtures of hydrocarbons and contaminants at particular conditions of temperature and pressure takes place in a controlled freeze zone section of a cryogenic distillation tower.

[0010] During normal operation, the cryogenic distillation tower operates at steady-state temperature conditions. During normal operation, the operating temperature of the top two sections that may be in a distillation tower, *e.g.*, the controlled freeze zone section, and the upper section, are cold. As a result of the cold operating temperatures, the concentration of CO₂ within the upper section and the controlled freeze zone section of the distillation tower is low; the CO₂ is knocked down by cold liquid sprays from a spray assembly within the controlled freeze zone section.

[0011] During abnormal operations, the cryogenic distillation tower does not operate at steady-state temperature conditions. The operating temperatures of the three main sections that may be in a distillation tower are not cold. As a result, the warmer operating temperatures prevent sufficient CO₂ from being knocked out, thereby leading to a higher concentration of CO₂ within the upper section and the controlled freeze zone section of the distillation tower than would be present during normal operation. The increased CO₂
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concentration may result in CO₂ solidifying outside of the middle controlled freeze zone section. The challenge during an abnormal operation is to get from an abnormal operation to a normal operation. Examples of when abnormal operations may occur include but are not limited to during start-up of the distillation tower. During start-up, the distillation tower may
5 be at or near-ambient temperatures.

[0012] A need exists for improved technology that can better facilitate going from an abnormal operation to a normal operation so as to separate fluids in a distillation tower, such as but not limited to during start-up of a distillation tower.

SUMMARY

10 [0013] The present disclosure provides a method and system for separating fluids in a distillation tower.

[0014] A method for separating fluids in a distillation tower may comprise maintaining a rectifier section in the distillation tower; maintaining a controlled freeze zone section in the distillation tower that forms solids from carbon dioxide within the distillation tower; feeding
15 a stream to the distillation tower, wherein the stream comprises the carbon dioxide; reducing a carbon dioxide concentration of the stream received by the rectifier section by: feeding a first cryogenic fluid to the controlled freeze zone section, wherein the first cryogenic fluid comprises a substantially carbon dioxide free fluid; and accumulating rectifier section stream in at least one of a holding vessel and a sump of the rectifier section; and terminating
20 reducing the carbon dioxide concentration when the carbon dioxide concentration of the stream travelling from the controlled freeze zone section to the rectifier section is less than or equal to a maximum carbon dioxide concentration.

[0015] A system for separating fluids in a distillation tower may comprise a rectifier section in the distillation tower; a controlled freeze zone section in the distillation tower that
25 forms solids from carbon dioxide within a stream that enters the distillation tower; a first cryogenic fluid pump outside of the distillation tower that is configured to increase a pressure of a first cryogenic fluid; a first cryogenic heat exchanger outside of the distillation tower that is configured to receive the first cryogenic fluid from the first cryogenic fluid pump and to increase a temperature of the first cryogenic fluid; and a spray assembly configured to spray
30 the first cryogenic fluid into the controlled freeze zone section, wherein the first cryogenic fluid comprises a substantially carbon dioxide free fluid.

[0016] The foregoing has broadly outlined the features of the present disclosure so that the detailed description that follows may be better understood. Additional features will also be described herein.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] These and other features, aspects and advantages of the disclosure will become apparent from the following description, appending claims and the accompanying drawings, which are briefly described below.

5 [0018] Figure 1 is a schematic diagram of a distillation tower with sections within multiple vessels.

[0019] Figure 2 is a schematic diagram of a distillation tower with sections within a single vessel.

10 [0020] Figure 3 is a schematic diagram of a distillation tower with sections within multiple vessels.

[0021] Figure 4 is a schematic diagram of a distillation tower with sections within a single vessel.

[0022] Figure 5 is a schematic diagram of a distillation tower with sections within multiple vessels during startup.

15 [0023] Figure 6 is a schematic diagram of a distillation tower with sections within multiple vessels during startup.

[0024] Figure 7 is a schematic diagram of a distillation tower with sections within a single vessel during startup.

20 [0025] Figure 8 is a schematic diagram of a distillation tower with sections within a single vessel during startup.

[0026] Figure 9 is a schematic diagram of a method for separating fluids in a distillation tower.

25 [0027] It should be noted that the figures are merely examples and no limitations on the scope of the present disclosure are intended thereby. Further, the figures are generally not drawn to scale, but are drafted for purposes of convenience and clarity in illustrating various aspects of the disclosure.

DETAILED DESCRIPTION

30 [0028] For the purpose of promoting an understanding of the principles of the disclosure, reference will now be made to the features illustrated in the drawings and specific language will be used to describe the same. It will nevertheless be understood that no limitation of the scope of the disclosure is thereby intended. Any alterations and further modifications, and any further applications of the principles of the disclosure as described herein are contemplated as would normally occur to one skilled in the art to which the disclosure relates. It will be apparent to those skilled in the relevant art that some features that are not relevant

to the present disclosure may not be shown in the drawings for the sake of clarity.

[0029] At the outset, for ease of reference, certain terms used in this application and their meaning as used in this context are set forth below. To the extent a term used herein is not defined below, it should be given the broadest definition persons in the pertinent art have given that term as reflected in at least one printed publication or issued patent. Further, the present processes are not limited by the usage of the terms shown below, as all equivalents, synonyms, new developments and terms or processes that serve the same or a similar purpose are considered to be within the scope of the present disclosure.

[0030] As used herein, the term “hydrocarbon fluids” refers to a hydrocarbon or mixtures of hydrocarbons that are gases or liquids. For example, hydrocarbon fluids may include a hydrocarbon or mixtures of hydrocarbons that are gases or liquids at formation conditions, at processing conditions or at ambient conditions (25 degrees Celsius ($^{\circ}$ C) and 1 atmosphere (atm) pressure). Hydrocarbon fluids may include, for example, oil, natural gas, coal bed methane, shale oil, pyrolysis oil, pyrolysis gas, a pyrolysis product of coal, and other hydrocarbons that are in a gaseous or liquid state.

[0031] The term “mass transfer device” refers to any object that receives fluids to be contacted, and passes those fluids to other objects, such as through gravitational flow.

[0032] As used herein, the term “fluid” refers to gases, liquids, and combinations of gases and liquids, as well as to combinations of gases and solids, and combinations of liquids and solids.

[0033] As used herein, the term “closed loop refrigeration system” refers to a configuration where the overhead stream of a distillation process having a controlled freeze zone section is chilled and partially condensed with an external working fluid. The external working fluid does not physically mix with the overhead stream. The external working fluid only exchanges heat with the overhead stream. The overhead stream is the stream exiting a top of an upper section of the distillation tower. A closed loop melt tray temperature conditioning system in the distillation process is part of a closed loop refrigeration system.

[0034] As used herein, the term “open loop refrigeration system” refers to a configuration where the overhead stream is used to assist in providing cooling. A compressor may be used to assist in providing the cooling. An open loop melt tray temperature conditioning system in the distillation process is part of an open loop refrigeration system.

[0035] As used herein, the term “normal operation” is when the distillation tower operates at steady-state conditions.

[0036] As used herein, the term “abnormal operation” is when the distillation tower does

not operate at steady-state conditions. An example of an abnormal operation includes but is not limited to during start-up of a distillation tower. Start-up may include the initial start-up of the distillation tower (i.e., when the distillation tower is first used). Start-up may include any time after the initial start-up of the distillation tower (e.g., a time after the distillation tower has been temporarily shut-down).

[0037] As used herein, the term “subsurface” refers to geologic strata occurring below the earth's surface.

[0038] A “reservoir” or “subterranean reservoir” is a subsurface rock or sand formation from which a production fluid or resource can be harvested. The rock formation may include sand, granite, silica, carbonates, clays, and organic matter, such as heavy oil (e.g., bitumen), gas, or coal, among others. Reservoirs can vary in thickness from less than one foot (0.3048 meter (m)) to hundreds of feet (hundreds of meters). The resource is generally a deposit of hydrocarbons.

[0039] The term “steady-state condition” refers to when the key distillation process operating variables, e.g., pressure, temperature, composition, and flows, have not fluctuated substantially for some period of time (e.g., hours). The period of time may range from greater than or equal to 15 minutes. The period of time may be any number within or bounded by the preceding range. When not at steady-state conditions, the distillation process may be at a transient state on its way to normal operation.

[0040] The terms “substantial,” “approximate,” “sufficient” and “about” when used in reference to a quantity or amount of a material, or a specific characteristic thereof, refer to an amount that is sufficient to provide an effect that the material or characteristic was intended to provide. The exact degree of deviation allowable may in some cases depend on the specific context.

[0041] As referenced in this application, the terms “stream,” “gas stream,” “vapor stream,” and “liquid stream” refer to different stages of a feed stream as the feed stream is processed in a distillation tower that separates methane, the primary hydrocarbon in natural gas, from contaminants. Although the phrases “gas stream,” “vapor stream,” and “liquid stream,” refer to situations where a gas, vapor, and liquid is mainly present in the stream, respectively, there may be other phases also present within the stream. For example, a gas may also be present in a “liquid stream.” In some instances, the terms “gas stream” and “vapor stream” may be used interchangeably. The feed stream may interchangeably be referred to as a stream.

[0042] A “flow controlling device” refers to any suitable device that can control pressure

and flow. A flow controlling device being “open” refers to the flow controlling device being anywhere from partially open to completely open. A flow controlling device may be bi-directional whether specified or not as such.

[0043] The articles “the”, “a” and “an” are not necessarily limited to mean only one, but
5 rather are inclusive and open ended so as to include, optionally, multiple such elements.

[0044] “At least one,” in reference to a list of one or more entities should be understood to mean at least one entity selected from any one or more of the entity in the list of entities, but not necessarily including at least one of each and every entity specifically listed within the list of entities and not excluding any combinations of entities in the list of entities. This
10 definition also allows that entities may optionally be present other than the entities specifically identified within the list of entities to which the phrase “at least one” refers, whether related or unrelated to those entities specifically identified. Thus, as a non-limiting example, “at least one of A and B” (or, equivalently, “at least one of A or B,” or, equivalently “at least one of A and/or B”) may refer, to at least one, optionally including more than one,
15 A, with no B present (and optionally including entities other than B); to at least one, optionally including more than one, B, with no A present (and optionally including entities other than A); to at least one, optionally including more than one, A, and at least one, optionally including more than one, B (and optionally including other entities). In other words, the phrases “at least one,” “one or more,” and “and/or” are open-ended expressions
20 that are both conjunctive and disjunctive in operation. For example, each of the expressions “at least one of A, B and C,” “at least one of A, B, or C,” “one or more of A, B, and C,” “one or more of A, B, or C” and “A, B, and/or C” may mean A alone, B alone, C alone, A and B together, A and C together, B and C together, A, B and C together, and optionally any of the above in combination with at least one other entity.

25 [0045] The disclosure relates to a system and method for separating fluids within a distillation tower such as but not limited to during abnormal operations. Figures 1-9 of the disclosure display various aspects of the system and method. The system may comprise a closed loop refrigeration system or an open loop refrigeration system.

[0046] The system and method may separate a feed stream, having hydrocarbons (e.g.,
30 methane) and contaminants, in a distillation tower **104, 204**. The system may comprise the distillation tower **104, 204** (Figures 1-8). The distillation tower **104, 204** may separate the contaminants from the hydrocarbons.

[0047] The distillation tower **104, 204** may be separated into three functional sections: a lower section **106**, a middle controlled freeze zone section **108** and an upper section **110**. The

distillation tower **104, 204** may incorporate three functional sections when the upper section **110** is needed and/or desired. The lower section **106** may interchangeably be referred to as a stripper section. The middle controlled freeze zone section **108** may interchangeably be referred to as a controlled freeze zone section. The upper section **110** may interchangeably be referred to as a rectifier section.

[0048] The distillation tower **104, 204** may incorporate only two functional sections when the upper section **110** is not needed and/or desired. When the distillation tower does not include an upper section **110**, a portion of vapor leaving the middle controlled freeze zone section **108** may be condensed in a condenser **122** and returned to the middle controlled freeze zone section **108** as a liquid stream via a spray assembly **129**. Line **41** may be eliminated. The stream in line **14**, now taking the vapors leaving the middle controlled freeze section **108**, directs these vapors to the condenser **122**.

[0049] The sections of the distillation tower **104** may be housed within a single vessel (Figures 2, 4 and 7-8). For example, the lower section **106**, the middle controlled freeze zone section **108**, and the upper section **110** may be housed within a single vessel **164**.

[0050] The sections of the distillation tower **204** may be housed within a plurality of vessels to form a split-tower configuration (Figures 1, 3 and 5-6). Each of the vessels may be separate from the other vessels. A line **341** and/or another suitable mechanism may connect one vessel to another vessel so that a vapor stream may travel from the middle controlled freeze zone section **108** to the upper section **110**. Before one vessel is connected to another vessel, the vapor stream travelling from the middle controlled freeze zone section **108** may exit the middle controlled freeze zone section **108** and travel away from the upper section **110** via line **343**. The vapor stream travelling from the middle controlled freeze zone section **108** may travel away from the upper section **110** via line **343** during abnormal operation. To prevent the vapor stream from travelling from the middle controlled freeze zone section **108** to the upper section **110**, before one vessel is connected to another vessel, line **341** may include a flow controlling device **342**. The flow controlling device **342** may comprise, but is not limited to, a valve.

[0051] In the split-tower configuration, the lower section **106**, middle controlled freeze zone section **108** and upper section **110** may be housed within two or more vessels. For example, the upper section **110** may be housed within a single vessel **254** and the lower and middle controlled freeze zone sections **106, 108** may be housed within a single vessel **264**. When this is the case, a liquid stream exiting the upper section **110**, may exit through a liquid outlet bottom **260**. The liquid outlet bottom **260** is at the bottom of the upper section **110**.

Although not shown, each of the sections may be housed within its own separate vessel, or one or more sections may be housed within separate vessels, or the upper and middle controlled freeze zone sections may be housed within a single vessel and the lower section may be housed within a single vessel, etc. When sections of the distillation tower are housed
5 within vessels, the vessels may be side-by-side along a horizontal line and/or above each other along a vertical line.

[0052] The split-tower configuration may be beneficial in situations where the height of the distillation tower, motion considerations, and/or transportation issues, such as for remote locations, need to be considered. This split-tower configuration allows for the independent
10 operation of one or more sections. For example, when the upper section is housed within a single vessel and the lower and middle controlled freeze zone sections are housed within a single vessel, independent generation of reflux using a substantially contaminant-free, largely hydrocarbon stream from a packed gas pipeline or an adjacent hydrocarbon line, may occur in the upper section. And the reflux may be used to cool the upper section, establish an
15 appropriate temperature profile in the upper section, and/or build up liquid inventory at the bottom of the upper section to serve as an initial source of spray liquids for the middle controlled freeze zone section. Moreover, the middle controlled freeze zone and lower sections may be independently prepared by chilling the feed stream, feeding it to the optimal location be that in the lower section or in the middle controlled freeze zone section, generating liquids for the lower and the middle controlled freeze zone sections, and disposing
20 the vapors off the middle controlled freeze zone section while they are off specification with too high a contaminant content. Also, liquid from the upper section may be intermittently or continuously sprayed, building up liquid level in the bottom of the middle controlled freeze zone section and bringing the contaminant content in the middle controlled freeze zone section down and near steady state level so that the two vessels may be connected to send the
25 vapor stream from the middle controlled freeze zone section to the upper section, continuously spraying liquid from the bottom of the upper section into the middle controlled freeze zone section and stabilizing operations into operating at a steady-state temperature. The split tower configuration may utilize a sump 331 of the upper section as a liquid receiver.
30 The sump 331 may be the liquid receiver for a pump 128. When the split tower configuration utilizes the sump 331, the distillation tower 204 may or may not utilize a holding vessel 126 (Figures 1-8).

[0053] The system may include a heat exchanger 100 (Figures 1-8). A feed stream 10 may enter the heat exchanger 100 before entering the distillation tower 104, 204. The feed

stream **10** may interchangeably be referred to as a stream. The stream **10** may be cooled within the heat exchanger **100**. The heat exchanger **100** helps drop the temperature of the stream **10** to a level suitable for introduction into the distillation tower **104, 204**.

[0054] The system may include an expander device **102** (Figures 1-8). The stream **10** may enter the expander device **102** before entering the distillation tower **104, 204**. The stream **10** may be expanded in the expander device **102** after exiting the heat exchanger **100**. The expander device **102** helps drop the temperature of the stream **10** to a level suitable for introduction into the distillation tower **104, 204**. The expander device **102** may be any suitable device, such as a valve. If the expander device **102** is a valve, the valve may be any suitable valve that may aid in cooling the stream **10** before it enters the distillation tower **104, 204**. For example, the valve **102** may comprise a Joule-Thompson (J-T) valve.

[0055] The system may include a feed separator **103** (Figures 3-4). The stream may enter the feed separator **103** before entering the distillation tower **104, 204**. The feed separator **103** may separate a stream **10** having a mixed liquid and vapor stream into a liquid stream and a vapor stream. Lines **12** may extend from the feed separator **103** to the distillation tower **104, 204**. One of the lines **12** may receive the vapor stream from the feed separator **103**. Another one of the lines **12** may receive the liquid stream from the feed separator **103**. Each of the lines **12** may extend to the same and/or different sections (i.e. middle controlled freeze zone, and lower sections) of the distillation tower **104, 204**.

[0056] If the system includes a feed separator, the expander device **102** may or may not be downstream of the feed separator **103**. In embodiments where the expander device **102** is downstream of the feed separator **103**, the expander device **102** may comprise a plurality of expander devices **102** such that each line **12** has an expander device **102**.

[0057] The system may include a dehydration unit **261** (Figures 1-8). The feed stream **10** may enter the dehydration unit **261** before entering the distillation tower **104, 204**. The feed stream **10** enters the dehydration unit **261** before entering the heat exchanger **100** and/or the expander device **102**. The dehydration unit **261** removes water from the feed stream **10** to prevent water from later presenting a problem in the heat exchanger **100**, expander device **102**, feed separator **103**, or distillation tower **104, 204**. The water can present a problem by forming a separate water phase (i.e., ice and/or hydrate) that plugs lines, equipment or negatively affects the distillation process. The dehydration unit **261** dehydrates the feed stream to a dew point sufficiently low to ensure a separate water phase will not form at any point downstream during the rest of the process. The dehydration unit may be any suitable dehydration unit, such as a molecular sieve or a glycol dehydration unit.

[0058] The system may include a filtering unit (not shown). The feed stream **10** may enter the filtering unit before entering the distillation tower **104, 204**. The filtering unit may remove undesirable contaminants from the feed stream before the feed stream enters the distillation tower **104, 204**. Depending on what contaminants are to be removed, the filtering unit may be before or after the dehydration unit **261** and/or before or after the heat exchanger **100**.

[0059] The systems may include a line **12** (Figures 1-8). The line may also be referred to as an inlet channel **12**. The feed stream **10** may be fed into the distillation tower **104, 204** via the line **12, 503** (Figure 9). The feed stream **10** (i.e., stream) may comprise contaminants (e.g., carbon dioxide). The feed stream **10** may also comprise hydrocarbons (e.g., methane). The line **12** may extend to the lower section **106** or the middle controlled freeze zone section **108** of the distillation tower **104, 204**. For example, the line **12** may extend to the lower section **106** such that the feed stream **10** may enter the lower section **106** of the distillation tower **104, 204** (Figures 1-8). The line **12** may directly or indirectly extend to the lower section **106** or the middle controlled freeze zone section **108**. The line **12** may extend to an outer surface of the distillation tower **104, 204** before entering the distillation tower **104, 204**.

[0060] If the system includes the feed separator **103** (Figures 3-4), the line **12** may comprise a plurality of lines **12**. Each line **12** may be the same line as one of the lines that extends from the feed separator **103** to a specific portion of the distillation tower **104, 204**.

[0061] The lower section **106** is constructed and arranged to separate the feed stream **10** into a liquid stream and a vapor stream. During normal operation, the lower section **106** separates the feed stream at a temperature and pressure at which no solids form. The liquid stream may comprise a greater quantity of contaminants than of methane. The vapor stream may comprise a greater quantity of methane than of contaminants. In any case, the vapor stream is lighter than the liquid stream. As a result, the vapor stream rises from the lower section **106** and the liquid stream falls to the bottom of the lower section **106**.

[0062] The lower section **106** may include and/or connect to equipment that separates the stream. The equipment may comprise any suitable equipment for separating methane from contaminants. Generally speaking, the suitable equipment comprises some type of mass transfer device. For example, the suitable equipment may comprise one or more packed sections **181**, or one or more distillation trays with perforations, downcomers, and weirs (Figures 1-8).

[0063] The equipment may include components that apply heat to the stream to form the vapor stream and the liquid stream. For example, the equipment may comprise a first

reboiler **112** that applies heat to the stream. The first reboiler **112** may be located outside of the distillation tower **104**, **204**. The equipment may also comprise a second reboiler **172** that applies heat to the stream. The second reboiler **172** may be located outside of the distillation tower **104**, **204**. Line **17** may lead from the distillation tower to the second reboiler **172**.

5 Line **117** may lead from the second reboiler **172** to the distillation tower. Additional reboilers, set up similarly to the second reboiler described above, may also be used.

[0064] The first reboiler **112** may apply heat to the liquid stream that exits the lower section **106** through a liquid outlet **160** of the lower section **106**. The liquid stream may travel from the liquid outlet **160** through line **28** to reach the first reboiler **112** (Figures 1-8).

10 The amount of heat applied to the liquid stream by the first reboiler **112** can be increased to separate more methane from contaminants. The more heat applied by the reboiler **112** to the stream, the more methane separated from the liquid contaminants, though more contaminants will also be vaporized.

[0065] The first reboiler **112** may also apply heat to the stream within the distillation tower **104**, **204**. Specifically, the heat applied by the first reboiler **112** warms up the lower section **106**. This heat travels up the lower section **106** and supplies heat to warm solids entering a melt tray assembly **139** of the middle controlled freeze zone section **108** so that the solids form a liquid slurry.

[0066] The second reboiler **172** applies heat to the stream within the lower section **106**. The heat applied by the second reboiler **172** is applied closer to the middle controlled freeze zone section **108** than the heat applied by the first reboiler **112**. As a result, the heat applied by the second reboiler **172** reaches the middle controlled freeze zone section **108** faster than the heat applied by the first reboiler **112**. The second reboiler **172** can help with energy integration if the second reboiler **172** comprises a heat exchanger that pre-cools the stream ahead of the heat exchanger **100**.

[0067] The equipment may include stripper section equipment. The stripper section equipment may include one or more chimney assemblies **135** (Figures 1-8). While falling to the bottom of the lower section **106**, the liquid stream may encounter one or more of the chimney assemblies **135**.

30 [0068] Each chimney assembly **135** includes a chimney tray **131** that collects the liquid stream within the lower section **106**. The liquid stream that collects on the chimney tray **131** may be fed to the second reboiler **172**. After the liquid stream is heated in the second reboiler **172**, the stream may return to the middle controlled freeze zone section **106** to supply heat to the middle controlled freeze zone section **106** and/or the melt tray assembly **139**.

Unvaporized stream exiting the second reboiler **172** may be fed back to the distillation tower **104, 204** below the chimney tray **131**. Vapor stream exiting the second reboiler **172** may be routed under or above the chimney tray **131** when the vapor stream enters the distillation tower **104, 204**.

5 **[0069]** The chimney tray **131** may include one or more chimneys **137**. The chimney **137** serves as a channel that the vapor stream in the lower section **106** traverses. The vapor stream travels through an opening in the chimney tray **131** at the bottom of the chimney **137** to the top of the chimney **137**. The opening is closer to the bottom of the lower section **106** than it is to the bottom of the middle controlled freeze zone section **108**. The top is closer to
10 the bottom of the middle controlled freeze zone section **108** than it is to the bottom of the lower section **106**.

[0070] Each chimney **137** has attached to it a chimney cap **133**. The chimney cap **133** covers a chimney top opening **138** of the chimney **137**. The chimney cap **133** prevents the liquid stream from entering the chimney **137**. The vapor stream exits the chimney assembly
15 **135** via the chimney top opening **138**.

[0071] After falling to the bottom of the lower section **106**, the liquid stream exits the distillation tower **104, 204** through the liquid outlet **160**. The liquid outlet **160** is within the lower section **106**. The liquid outlet **160** may be located at the bottom of the lower section **106**.

20 **[0072]** After exiting through the liquid outlet **160**, the bottoms stream may travel via line **28** to the first reboiler **112**. The bottoms stream may be heated by the first reboiler **112** and vapor may then re-enter the lower section **106** through line **30**. Unvaporized liquid may continue out of the distillation process via line **24**.

[0073] The system may include an expander device **114** (Figures 1-8). After entering line
25 **24**, the heated liquid stream may be expanded in the expander device **114**. The expander device **114** may be any suitable device, such as a valve. The valve **114** may be any suitable valve, such as a J-T valve.

[0074] The system may include a heat exchanger **116** (Figures 1-8). The liquid stream heated by the first reboiler **112** may be cooled or heated by the heat exchanger **116**. The heat
30 exchanger **116** may be a direct heat exchanger or an indirect heat exchanger. The heat exchanger **116** may comprise any suitable heat exchanger. The stream may exit the heat exchanger **116** through line **26**.

[0075] The vapor stream in the lower section **106** may rise from the lower section **106** to the middle controlled freeze zone section **108**. The vapor stream may include contaminants

and hydrocarbons. The middle controlled freeze zone section **108** may be maintained to form solids from the carbon dioxide within the distillation tower **104**, **204**, **502** (Figure 9). The carbon dioxide in the solids may be the contaminants that rise from the lower section **106**.

[0076] The middle controlled freeze zone section **108** is constructed and arranged to
5 separate the feed stream **10** introduced into the middle controlled freeze zone section into the solids and the vapor stream. The solids and the vapor stream are formed in the middle controlled freeze zone section **108** when a liquid stream is sprayed into the middle controlled freeze zone section **108** at a temperature and pressure at which the solids and the vapor stream form. The solids may be comprised more of contaminants than of methane. The
10 vapor stream may comprise more methane than contaminants.

[0077] The middle controlled freeze zone section **108** includes a lower section **40** and an upper section **39** (Figure 2, 4 and 7-8). The lower section **40** is below the upper section **39**. The lower section **40** directly abuts the upper section **39**. The lower section **40** is primarily but may not exclusively be a heating section of the middle controlled freeze zone section **108**.
15 The upper section **39** is primarily but may not exclusively be a cooling section of the middle controlled freeze zone section **108**. During normal operation, the temperature and pressure of the upper section **39** are chosen so that the solids can form in the middle controlled freeze zone section **108**.

[0078] The middle controlled freeze zone section **108** may comprise a melt tray assembly
20 **139** that is maintained in the middle controlled freeze zone section **108** (Figures 1-8). The melt tray assembly **139** is within the lower section **40** of the middle controlled freeze zone section **108**. The melt tray assembly **139** is not within the upper section **39** of the middle controlled freeze zone section **108**.

[0079] The melt tray assembly **139** is constructed and arranged to melt the solids in the
25 middle controlled freeze zone section **108**. When the warm vapor stream rises from the lower section **106** to the middle controlled freeze zone section **108**, the vapor stream immediately encounters the melt tray assembly **139** and supplies heat to melt the solids. The melt tray assembly **139** may comprise at least one of a melt tray **118**, a bubble cap **132**, a liquid **130** and heat mechanism(s) **134**.

[0080] The melt tray **118** may collect a liquid slurry. The melt tray **118** divides at least a
30 portion of the middle controlled freeze zone section **108** from the lower section **106**. The melt tray **118** is at the bottom **45** of the middle controlled freeze zone section **108**.

[0081] One or more bubble caps **132** may act as a channel for the vapor stream rising from the lower section **106** to the middle controlled freeze zone section **108**. The bubble cap

132 may provide a path for the vapor stream that forces the vapor stream up the riser **140** and then down and around the riser **140** to the melt tray **118**. The riser **140** is covered by a cap **141**. The cap **141** prevents the liquid **130** from travelling into the riser **140**. The cap **141** helps prevent solids from travelling into the riser **140**. The vapor stream's traversal through
5 the bubble cap **132** allows the vapor stream to transfer heat to the liquid **130** within the melt tray assembly **139**.

[0082] One or more heat mechanisms **134** may further heat up the liquid **130** to facilitate melting of the solids into the liquid slurry. The heat mechanism(s) **134** may be located anywhere within the melt tray assembly **139**. For example, a heat mechanism **134** may be
10 located around the bubble caps **132** (Figures 1-8). The heat mechanism **134** may be any suitable mechanism, such as a heat coil. The heat source of the heat mechanism **134** may be any suitable heat source.

[0083] The liquid **130** in the melt tray assembly is heated by the vapor stream. The liquid **130** may also be heated by the one or more heat mechanisms **134**. The liquid **130** helps melt
15 the solids formed in the middle controlled freeze zone section **108** into the liquid slurry. Specifically, the heat transferred by the vapor stream heats up the liquid, thereby enabling the heat to melt the solids. During normal operation, the liquid **130** is at a level sufficient to melt the solids.

[0084] The middle controlled freeze zone section **108** may also comprise a spray
20 assembly **129**. The spray assembly **129** cools the vapor stream that rises from the lower section **40**. The spray assembly **129** sprays liquid, which is cooler than the vapor stream, on the vapor stream to cool the vapor stream. The spray assembly **129** is within the upper section **39**. The spray assembly **129** is not within the lower section **40**. The spray assembly **129** is above the melt tray assembly **139**. In other words, the melt tray assembly **139** is below
25 the spray assembly **129**.

[0085] The temperature in the middle controlled freeze zone section **108** cools down as the vapor stream travels from the bottom of the middle controlled freeze zone section **108** to the top of the middle controlled freeze zone section **108**. The methane in the vapor stream rises from the middle controlled freeze zone section **108** to the upper section **110**. Some
30 contaminants may remain in the methane and also rise. The contaminants in the vapor stream tend to condense or solidify with the colder temperatures and fall to the bottom of the middle controlled freeze zone section **108**.

[0086] The solids form the liquid slurry when in the liquid **130**. The liquid slurry flows from the middle controlled freeze zone section **108** to the lower distillation section **106**. The

liquid slurry flows from the bottom of the middle controlled freeze zone section **108** to the top of the lower section **106** via a line **22** (Figures 1-8). The line **22** may be an exterior line. The line **22** may extend from the distillation tower **104, 204**. The line **22** may extend from the middle controlled freeze zone section **108**. The line **22** may extend to the lower section **106**.

[0087] The vapor stream that rises in the middle controlled freeze zone section **108** and does not form solids or otherwise fall to the bottom of the middle controlled freeze zone section **108**, rises to the upper section **110**. The upper section **110** may be maintained in the distillation tower **104, 204, 501** (Figure 9). During normal operation, the upper section **110** operates at a temperature and pressure and contaminant concentration at which no solids form. The upper section **110** is constructed and arranged to cool the vapor stream to separate the methane from the contaminants. Reflux in the upper section **110** may cool the vapor stream. The reflux in the upper section **110** may absorb contaminants. The reflux may comprise liquid reflux. The reflux is introduced into the upper section **110** via line **18**. Line **18** may extend to the upper section **110**. Line **18** may extend from an outer surface of the distillation tower **104, 204**.

[0088] After contacting the reflux in the upper section **110**, the feed stream forms a vapor stream and a liquid stream. The vapor stream mainly comprises methane. The liquid stream comprises relatively more contaminants. The vapor stream rises in the upper section **110** and the liquid falls to a bottom of the upper section **110**.

[0089] To facilitate separation of the methane from the contaminants when the stream contacts the reflux, the upper section **110** may include one or more mass transfer devices **176**. Each mass transfer device **176** helps contact the liquid and vapor phases, which helps separate the methane from the contaminants. Each mass transfer device **176** may comprise any suitable contacting device, such as a tray with perforations, or a section of random or structured packing **176** to facilitate contact of the vapor and liquid phases.

[0090] After rising, the vapor stream may exit the distillation tower **104, 204** through outlet **401** to line **14**. The line **14** may emanate from an upper part of the upper section **110**. The line **14** may extend from an outer surface of the upper section **110**.

[0091] From line **14**, the vapor stream may enter a condenser **122**. The condenser **122** cools the vapor stream to form a cooled stream. The condenser **122** at least partially condenses the stream.

[0092] After exiting the condenser **122**, the cooled stream may enter a reflux accumulator **124**. The cooled stream may be a two-phase stream. The two-phase stream may comprise a

liquid stream and a vapor stream. The reflux accumulator **124** separates the two-phase stream into a liquid stream and a vapor stream. The reflux accumulator may be any suitable vessel that can separate a stream into a liquid stream and a vapor stream, such as a reflux drum. The reflux accumulator may serve as a holding unit for the cooled stream entering the
5 reflux accumulator **124**.

[0093] Once separated, the vapor stream may exit the reflux accumulator **124** as sales product. The sales product may travel through line **16** for subsequent sale to a pipeline and/or condensation to be liquefied natural gas.

[0094] Once separated, the liquid stream may return to the upper section **110** through line
10 **18** as the reflux. The reflux may travel to the upper section **110** via any suitable mechanism, such as a reflux pump **150** (Figures 1-2, 5-8) or gravity (Figures 3-4).

[0095] The liquid stream that falls to the bottom of the upper section **110** collects at the bottom of the upper section **110** as the liquid **330**. The liquid stream may collect on tray **183** (Figures 2, 4 and 7-8) or at the sump **331** of the upper section **110** (Figures 1, 3 and 5-6). The
15 sump **331** is at the bottom portion of the upper section **110**. The liquid stream collected may exit the distillation tower **104**, **204** through line **20** (Figures 2, 4 and 7-8) or outlet **260** (Figures 1, 3 and 5-6). The line **20** (Figures 2, 4 and 7-8) may emanate from the upper section **110**. The line **20** may emanate from a bottom end of the upper section **110**. The line **20** may extend from an outer surface of the upper section **110**.

[0096] The line **20** and/or outlet **260** connect to a line **41**. The line **41** leads to the spray
20 assembly **129** in the middle controlled freeze zone section **108**. The line **41** may emanate from the holding vessel **126**. While the holding vessel **126** is shown in Figures 1-8, the holding vessel **126** may or may not be present when the upper section **110** includes the sump **331**. The line **41** may extend to an outer surface of the middle controlled freeze zone section
25 **110**.

[0097] The line **20** and/or outlet **260** may directly or indirectly (Figures 1-8) connect to the line **41**. When the line **20** and/or outlet **260** directly connect to the line **41**, the liquid spray may be pumped to the spray nozzle(s) **120** via any suitable mechanism, such as the spray pump **128** or gravity. When the line **20** and/or outlet **260** indirectly connect to the line
30 **41**, the line **20** and/or outlet **260** and line **41** may directly connect to the holding vessel **126**. The holding vessel **126** may house at least some of the liquid spray before it is sprayed by the nozzle(s). The liquid spray may be pumped from the holding vessel **126** to the spray nozzle(s) **120** via any suitable mechanism, such as the spray pump **128** (Figures 1-8) or gravity. The holding vessel **126** may be needed when the sump **331** does not have sufficient

volume to hold the liquid stream at the bottom of the upper section **110**.

[0098] During an abnormal operation of the distillation tower **104, 204**, the distillation tower **104, 204** does not operate at steady-state conditions. To make the distillation tower **104, 204** go from not operating at steady-state conditions to operating at steady-state conditions, the system may reduce a carbon dioxide concentration of the stream received by the upper section **110, 504** (Figure 9). Reducing the carbon dioxide concentration may include feeding a first cryogenic fluid to the middle controlled freeze zone section **108, 505** (Figure 9) and accumulating reflux in at least one of the holding vessel **126** and the sump **331, 506** (Figure 9). The system may terminate reducing the carbon dioxide concentration when the carbon dioxide concentration of the stream travelling from the middle controlled freeze zone section **108** to the upper section **110** is less than or equal to a maximum carbon dioxide concentration, **507** (Figure 9).

[0099] The carbon dioxide concentration is the concentration of carbon dioxide within the stream travelling from the middle controlled freeze zone section **108** to the upper section **110**. The carbon dioxide concentration may be determined by any suitable detecting mechanism. Examples of a suitable detecting mechanism may include, but is not limited to, a gas chromatograph or an infrared detector. The detecting mechanism may sample the stream travelling from the middle controlled freeze zone section **108** to the upper section **110** from an upper portion of the middle controlled freeze zone section **108**. The maximum carbon dioxide concentration may be equal to a carbon dioxide concentration in the stream travelling from the middle controlled freeze zone section **108** to the upper section **110** that is less than or equal to 6 percent (%) of the components within the stream. The maximum carbon dioxide concentration may be any number bounded by or included within the aforementioned range, such as but not limited to less than or equal to 5%, or less than or equal to 4%. The components within the stream may comprise any component within the stream (e.g., carbon dioxide, methane).

[00100] The first cryogenic fluid may comprise a substantially carbon-dioxide-free fluid. In other words, the first cryogenic fluid may not comprise carbon dioxide. For example, the first cryogenic fluid may comprise liquid natural gas or liquid nitrogen. The liquid natural gas may comprise methane.

[0100] The first cryogenic fluid may be stored in any suitable cryogenic fluid storage container **228, 247** (Figures 5-8). For example, the cryogenic fluid storage container **228** may comprise a tank. The cryogenic fluid storage container **228, 247** may be outside of the distillation tower **104, 204**. In other words, the cryogenic fluid storage container **228, 247**

may not be inside of the distillation tower **104, 204**.

[0101] To feed the first cryogenic fluid to the middle controlled freeze zone section **108**, the cryogenic fluid storage container **228, 247** may fluidly connect to the middle controlled freeze zone section **108** of the distillation tower **104, 204** via line **234, 263**. The first cryogenic fluid may be fed from the cryogenic fluid storage container **228, 247** to a first cryogenic fluid pump **226, 246** via line **234, 263**. The first cryogenic fluid may be fed from the first cryogenic fluid pump **226, 246** to a first cryogenic heat exchanger **225, 245** via line **234, 263**. The first cryogenic fluid may be fed from the first cryogenic heat exchanger **225, 245** to the middle controlled freeze zone section **108**.

[0102] The first cryogenic fluid pump **226, 246** may be configured to increase a pressure of a first cryogenic fluid. A pressure of the first cryogenic fluid, which enters the first cryogenic fluid pump **226, 246**, is increased when the first cryogenic fluid is pumped through the first cryogenic fluid pump **226, 246**. The first cryogenic fluid pump **226, 246** may increase the pressure of the first cryogenic fluid so that it can enter the distillation tower **104, 204** at a pressure closer to but above a pressure within the distillation tower **104, 204**. The first cryogenic fluid pump **226, 246** may be outside of the distillation tower **104, 204**. In other words, the first cryogenic fluid pump **226, 246** may not be within the distillation tower **104, 204**.

[0103] The first cryogenic heat exchanger **225, 245** may be configured to receive the first cryogenic fluid. The first cryogenic heat exchanger **225, 245** may receive the first cryogenic fluid from the first cryogenic pump **226, 246**. The first cryogenic heat exchanger **225, 245** may receive the first cryogenic fluid after the first cryogenic fluid exits the first cryogenic pump **226, 246**. The first cryogenic heat exchanger **225, 245** may increase a temperature of the first cryogenic fluid such that the temperature of the first cryogenic fluid goes from a first temperature when it enters the first cryogenic heat exchanger **225, 245** to a second temperature when it exits the first cryogenic heat exchanger **225, 245**. The second temperature may be higher than the first temperature. The first cryogenic heat exchanger **225, 245** may increase the temperature of the first cryogenic fluid so that it can enter the distillation tower **104, 204** at a temperature closer to that within the distillation tower **104, 204**. The first cryogenic heat exchanger **225, 245** may be outside of the distillation tower **104, 204**. In other words, the first cryogenic heat exchanger **225, 245** may not be within the distillation tower **104, 204**.

[0104] The system may include a spray assembly **129, 270** that feeds the first cryogenic fluid to the middle controlled freeze zone section **108** from the first cryogenic heat exchanger

225, 245. The spray assembly **129, 270** may be configured to spray the first cryogenic fluid into the middle controlled freeze zone section **108**.

[0105] The spray assembly **129, 270** may include a spray pump **128, 248** and spray nozzles **120, 240**. The spray pump **128, 248** may pump the first cryogenic fluid, which exits the first cryogenic heat exchanger **225, 245**, through line **232** to the spray nozzles **120, 240**. In other words, the first cryogenic fluid may travel from the first cryogenic heat exchanger **225, 245** to the spray pump **128, 248** and the spray pump **128, 248** may pump the first cryogenic fluid to the spray nozzles **120, 240**. The spray nozzles **120, 240** may spray the first cryogenic fluid into the middle controlled freeze zone section **108**.

[0106] The spray assembly **129** may comprise a controlled freeze zone section spray assembly **129** or a cryogenic fluid pump assembly **270**. The spray assembly may comprise a controlled freeze zone section spray assembly **129** when the first cryogenic fluid comprises liquid natural gas. The controlled freeze zone section spray assembly **129** is the same spray assembly that sprays the liquid spray from the upper section **110** to the middle controlled freeze zone section **108** during normal operation. The spray assembly may comprise a cryogenic fluid pump assembly **270** when the first cryogenic fluid comprises liquid nitrogen. The spray assembly **270** may be specially designed to handle the liquid nitrogen. The liquid nitrogen may be sprayed in spray assembly **270** instead of spray assembly **129** because spray assembly **129** may not be designed to handle liquid nitrogen. Liquid nitrogen tends to be colder than liquid natural gas and, therefore, may require different equipment than liquid natural gas to be sprayed. It may be possible to design the spray assembly **129** so that it can handle a first cryogenic fluid that comprises liquid nitrogen. If the spray assembly **129** is so designed, there may not be a cryogenic fluid spray assembly **270**. If the spray assembly **129** is so designed it may comprise material that can handle liquid nitrogen. The material for handling liquid nitrogen may comprise any suitable material, such as but not limited to, special metal alloys like high nickel. If the spray assembly **129** is so designed it may comprise a mechanism that allows it to transition from handling liquid nitrogen to liquid natural gas and methane. The mechanism may comprise a way for initially spraying the liquid nitrogen in a dedicated spray bank and then start reducing the amount of liquid nitrogen sprayed when reflux builds up while increasing the reflux sprayed through the spray assembly **129**.

[0107] When the spray assembly **129, 270** sprays the first cryogenic fluid, the carbon dioxide concentration of the stream travelling from the middle controlled freeze zone section **108** to the upper section **110** may be reduced. The concentration of carbon dioxide may be

reduced because the first cryogenic fluid sprayed may lower the temperature of the carbon dioxide within the vapor stream, which is rising within the middle controlled freeze zone section **108**, to one in which the carbon dioxide forms solids. When the carbon dioxide forms solids, the solids fall toward the bottom of the middle controlled freeze zone section **108** and less carbon dioxide travels from the middle controlled freeze zone section **108** to the upper section **110** because so much of the carbon dioxide falls toward the bottom of the middle controlled freeze zone section **108** as solids.

[0108] When the spray assembly **270** comprises a cryogenic fluid pump assembly **270**, the system also comprises a spray assembly **129**. The spray assembly **129** is the same spray assembly that sprays the liquid spray (i.e., stream) from the upper section **110** during normal operation. The spray assembly **129** may comprise a controlled freeze zone section spray assembly **129**.

[0109] In addition to being the spray assembly that sprays the liquid spray, the controlled freeze zone section spray assembly **129** may receive a second cryogenic fluid that is fed to the middle controlled freeze zone section **110**. The second cryogenic fluid may comprise a substantially carbon-dioxide-free fluid. The second cryogenic fluid may comprise liquid natural gas. The second cryogenic fluid may be fed to the middle controlled freeze zone section **108** sometime after the first cryogenic fluid is fed to the middle controlled freeze zone section **108** via the spray assembly **270**. The second cryogenic fluid may be fed to the middle controlled freeze zone section **108** at the same time that the first cryogenic fluid is fed to the middle controlled freeze zone section **108** via the spray assembly **270**.

[0110] The controlled freeze zone section spray assembly **129** receives the second cryogenic fluid the same way that the previously described controlled freeze zone section spray assembly receives the first cryogenic fluid when the first cryogenic fluid comprises liquid natural gas. Specifically, the second cryogenic fluid may be fed from a second cryogenic fluid storage container **1228** to a second cryogenic fluid pump **1226**, from the second cryogenic fluid pump **1226** to a second cryogenic heat exchanger **1225**, and from the second cryogenic heat exchanger **1225** to the controlled freeze zone section spray assembly **129**.

[0111] The second cryogenic fluid storage container **1228** may fluidly connect to the middle controlled freeze zone section **108** of the distillation tower **104**, **204** via line **1234**, **1236**. The second cryogenic fluid may be fed from the second cryogenic fluid storage container **1228** to the second cryogenic fluid pump **1226** via line **1234**. The second cryogenic fluid may be fed from the second cryogenic fluid pump **1226** to the second cryogenic heat

exchanger via line **1234**.

[0112] The second cryogenic fluid pump **1226** may be configured to increase a pressure of the second cryogenic fluid. A pressure of the second cryogenic fluid, which enters the second cryogenic fluid pump **1226**, is increased when the second cryogenic fluid is pumped
5 through the second cryogenic fluid pump **1226**. The second cryogenic fluid pump **1226** may be outside of the distillation tower **104, 204**. In other words, the second cryogenic fluid pump **1226** may not be within the distillation tower **104, 204**.

[0113] The second cryogenic heat exchanger **1225** may be configured to receive the second cryogenic fluid. The second cryogenic heat exchanger **1225** may receive the second
10 cryogenic fluid from the second cryogenic pump **1226**. The second cryogenic heat exchanger **1225** may receive the second cryogenic fluid after the second cryogenic fluid exits the second cryogenic pump **1226**. The second cryogenic heat exchanger **1225** may increase a temperature of the second cryogenic fluid such that the temperature of the second cryogenic fluid goes from a first temperature when it enters the second cryogenic heat exchanger **1225**
15 to a second temperature when it exits the second cryogenic heat exchanger **1225**. The second temperature may be higher than the first temperature. The second cryogenic heat exchanger **1225** may be outside of the distillation tower **104, 204**. In other words, the second cryogenic heat exchanger **1225** may not be within the distillation tower **104, 204**.

[0114] The middle controlled freeze zone section **108** may receive a second cryogenic
20 fluid from the reflux accumulator **124**. The second cryogenic fluid may comprise a substantially carbon-dioxide-free fluid. The second cryogenic fluid may comprise liquid natural gas. The second cryogenic fluid may be fed from the reflux accumulator **124** to a second cryogenic fluid pump **150**. The second cryogenic fluid may be fed from the second cryogenic fluid pump **150** to the middle controlled freeze zone section **108**. The second
25 cryogenic fluid may be fed from the second cryogenic fluid pump **150** to the middle controlled freeze zone section **108** via line **233, 1233, 236, 1236** and the controlled freeze zone section spray assembly **129**. The second cryogenic fluid may be fed when the first cryogenic fluid is liquid natural gas or liquid nitrogen. The middle controlled freeze zone section **108** may receive the second cryogenic fluid from the reflux accumulator **124** after an
30 initial start-up of the distillation tower **104, 204** but sometime during abnormal operation. The distillation tower **104, 204** may not receive the second cryogenic fluid during the initial start-up because the reflux accumulator **124** needs to accumulate some of the second cryogenic fluid from the stream exiting the upper section **110** so that the reflux accumulator **124** has some second cryogenic fluid to feed to the middle controlled freeze zone section **108**.

During the initial start-up it is unlikely that the reflux accumulator **124** will have accumulated any second cryogenic fluid to feed to the middle controlled freeze zone section **108**.

[0115] The system may comprise a way for feeding a rectifier section cryogenic fluid to the upper section **110**. The rectifier section cryogenic fluid may comprise a same cryogenic fluid as the first cryogenic fluid. If the rectifier section cryogenic fluid comprises a same cryogenic fluid as the first cryogenic fluid, the rectifier section cryogenic fluid and the first cryogenic fluid may be fed from the same cryogenic fluid storage container. The rectifier section cryogenic fluid may comprise a same cryogenic fluid as the second cryogenic fluid. If the rectifier section cryogenic fluid comprises a same cryogenic fluid as the second cryogenic fluid, the rectifier section cryogenic fluid and the second cryogenic fluid may be fed from the same cryogenic fluid storage container. The rectifier section cryogenic fluid may comprise a substantially carbon dioxide free fluid. The rectifier section cryogenic fluid may comprise no carbon dioxide.

[0116] Feeding the rectifier section cryogenic fluid to the upper section may comprise feeding the rectifier section cryogenic fluid from a cryogenic fluid storage container **228**, **1228**, **247** to a rectifier section cryogenic fluid pump **226**, **1226**, **246**, feeding the rectifier section cryogenic fluid from the rectifier section cryogenic fluid pump **226**, **1226**, **246** to a rectifier section cryogenic heat exchanger **225**, **1225**, **245**, and feeding the rectifier section cryogenic fluid from the rectifier section cryogenic heat exchanger **225**, **1225**, **245** to the upper section **110**. The rectifier section cryogenic fluid may be fed from the rectifier section cryogenic heat exchanger **225**, **1225**, **245** via a line **233**, **1233**. The rectifier section cryogenic fluid fed from the cryogenic fluid storage container **247** to the rectifier section **100** may be fed via lines **1233**, **1234**. The rectifier section cryogenic fluid fed from the cryogenic fluid storage container **247** to the rectifier section **110** may be fed via lines **231**, **1231**, **1233**, **1234**. Whether the rectifier section cryogenic fluid is fed from the cryogenic fluid storage container **247** or the cryogenic fluid storage container **1228** to the rectifier section **110**, line **1234** may include flow controlling device **3000**; line **231**, **1231** may include flow controlling device **1227**. When the rectifier section cryogenic fluid is fed from the cryogenic fluid storage container **247**, flow controlling device **3000** may be open and flow controlling device **1227** may be closed. When rectifier section cryogenic fluid is fed from the cryogenic fluid storage container **1228**, flow controlling device **1227** may be open and flow controlling device **3000** may be closed. The flow controlling device **3000** may be any suitable device. For example, the flow controlling device **3000** may be a valve. The flow controlling device **1227** may be any suitable device. For example, the flow controlling device **1227** may be a

valve.

[0117] The cryogenic fluid storage container **228, 1228, 247** may be the same storage container that the first cryogenic fluid or the second cryogenic fluid is stored in (Figures 5-8). If the cryogenic fluid storage container **228, 1228, 247** is the same storage container that the first cryogenic fluid is stored in, the rectifier section cryogenic fluid is the first cryogenic fluid. If the cryogenic fluid storage container **228, 1228, 247** is the same storage container that the second cryogenic fluid is stored in, the rectifier section cryogenic fluid is the second cryogenic fluid.

[0118] The rectifier section cryogenic fluid pump **226, 1226, 246** may be the same cryogenic fluid pump that the first cryogenic fluid is pumped through or that the second cryogenic fluid is pumped through. If the rectifier section cryogenic fluid pump **226, 1226, 246** is the same cryogenic fluid pump that the first cryogenic fluid is pumped through, the rectifier section cryogenic fluid is the first cryogenic fluid. If the rectifier section cryogenic fluid pump **226, 1226, 246** is the same cryogenic fluid pump that the second cryogenic fluid is pumped through, the rectifier section cryogenic fluid is the second cryogenic fluid.

[0119] The rectifier section cryogenic heat exchanger **225, 1225, 245** may be the same cryogenic heat exchanger that the first cryogenic fluid is pumped through or that the second cryogenic fluid is pumped through. If the rectifier section cryogenic heat exchanger **225, 1225, 245** is the same cryogenic heat exchanger that the first cryogenic fluid traverses through, the rectifier section cryogenic fluid is the first cryogenic fluid. If the rectifier section cryogenic heat exchanger **225, 1225, 245** is the same cryogenic heat exchanger that the second cryogenic fluid traverses through, the rectifier section cryogenic fluid is the second cryogenic fluid.

[0120] Accumulating the liquid stream in at least one of the holding vessel **126** and the sump **331** of the upper section **110** may comprise collecting the rectifier section cryogenic fluid in at least one of the holding vessel **126** and the sump **331** and/or collecting condensation of the vapor stream that travels from the middle controlled freeze zone section **108** to the upper section **110** in at least one of the holding vessel **126** and the sump **331**. The rectifier section cryogenic fluid may be collected after the rectifier section cryogenic fluid is fed to the upper section **110**. Initially, the rectifier section cryogenic fluid may vaporize as it cools down the rectifier section **110** as it falls through the rectifier section **110**. The rectifier section cryogenic fluid may be collected after it is fed because the rectifier section cryogenic fluid includes liquid and the liquid may fall toward the bottom of the upper section **110** to be fed to the holding vessel **126** and/or to fall into the sump **331**.

[0121] As liquid stream is accumulated, the amount of first cryogenic fluid and/or second cryogenic fluid fed to the middle controlled freeze zone section **108** may be reduced until no more first cryogenic fluid and/or second cryogenic fluid is fed to the middle controlled freeze zone section **108**. The liquid stream may interchangeably be referred to as rectifier section stream. As rectifier section stream is accumulated, the amount of rectifier section cryogenic fluid fed to the upper section **110** may be reduced until no more rectifier section cryogenic fluid is fed to the upper section **110** because the system is at normal operation.

[0122] At least a rectifier section stream portion of the rectifier section stream in the holding vessel **126** and/or the sump **331** may be fed to the cryogenic fluid storage container **228, 1228**. The rectifier section stream portion may be fed to the cryogenic fluid storage container **228, 1228** by travelling via line **231, 1231** to the cryogenic fluid storage container (Figures 5-8). The line **231, 1231** may include a flow controlling device **227, 1227** that restricts how much of the rectifier section stream portion may be fed to the cryogenic fluid storage container **228, 1228**. The flow controlling device **227, 1227** may be any suitable device. For example, the flow controlling device **227, 1227** may be a valve. The rectifier section stream portion may be fed to or from the cryogenic fluid storage container **228, 1228** during abnormal and/or normal operation. If the rectifier section stream portion is fed to the cryogenic fluid storage container **228, 1228** during normal operation it may be because the rectifier section stream portion contains excess rectifier section stream.

[0123] The rectifier section stream portion may comprise at least one of the first cryogenic fluid, the rectifier section cryogenic fluid and the second cryogenic fluid. After being fed to the cryogenic storage container **228, 1228**, the at least one of the first cryogenic fluid, the rectifier section cryogenic fluid and the second cryogenic fluid in the rectifier section stream portion may be separated from the solids in the rectifier section stream portion. The rectifier section stream portion may include solids after the rectifier section stream portion is passed through flow controlling device **227, 1227** because solids may be formed after the rectifier section stream portion passes through flow controlling device **227, 1227**. The rectifier section stream portion may not include solids before the rectifier section stream portion passes through flow controlling device **227, 1227**.

[0124] The rectifier section stream portion fed to the cryogenic fluid storage container **228, 1228** may comprise at least one of vapor, liquid and solids. The vapor within the cryogenic fluid storage container **228, 1228** may rise to the top of the cryogenic fluid storage container **228, 1228** and exit through the top of the cryogenic fluid storage container **228, 1228**. After exiting the cryogenic fluid storage container **228, 1228**, the vapor may be

compressed in a compressor **238, 1238** and transported to line **16** (i.e., sales pipeline) via line **235, 1235**. The liquid and/or solids in the cryogenic fluid storage container **228, 1228** may fall towards the bottom of the cryogenic fluid storage container **228, 1228**. The liquid that falls may be fed to a storage unit **230, 1230** via a flow control device **229, 1229** that allows
5 for the liquid to travel to the storage unit **230, 1230** but does not allow solids to travel to the storage unit **230, 1230**. In other words, the flow control device **229, 1229** may restrict the passage of solids. The solids may collect at a bottom **290, 1290** of the cryogenic fluid storage container **228, 1228**. If another abnormal operation arises, liquid from the cryogenic fluid storage container **228, 1228** may be fed to line **234, 1234** so that the cryogenic fluid can be
10 fed to the distillation tower **104, 204** to enable restoration to normal operation. Solids that fall to the bottom of the cryogenic fluid storage container may be pumped to a flare vessel (not shown) for disposal. Ambient heating of the flare vessel may melt/vaporize the solids.

[0125] It is important to note that the steps depicted in Figure 9 are provided for illustrative purposes only and a particular step may not be required to perform the inventive
15 methodology. Moreover, Figure 9 may not illustrate all the steps that may be performed. The claims, and only the claims, define the inventive system and methodology.

[0126] Disclosed aspects may be used in hydrocarbon management activities. As used herein, “hydrocarbon management” or “managing hydrocarbons” includes hydrocarbon extraction, hydrocarbon production, hydrocarbon exploration, identifying potential
20 hydrocarbon resources, identifying well locations, determining well injection and/or extraction rates, identifying reservoir connectivity, acquiring, disposing of and/ or abandoning hydrocarbon resources, reviewing prior hydrocarbon management decisions, and any other hydrocarbon-related acts or activities. The term “hydrocarbon management” is also used for the injection or storage of hydrocarbons or CO₂, for example the sequestration of
25 CO₂, such as reservoir evaluation, development planning, and reservoir management. The disclosed methodologies and techniques may be used in extracting hydrocarbons from a subsurface region and processing the hydrocarbons. Hydrocarbons and contaminants may be extracted from a reservoir and processed. The hydrocarbons and contaminants may be processed, for example, in the distillation tower previously described. After the
30 hydrocarbons and contaminants are processed, the hydrocarbons may be extracted from the processor, such as the distillation tower, and produced. The contaminants may be discharged into the Earth, etc. For example, the method for producing hydrocarbons may include producing the vapor stream extracted from the distillation tower. The method may also include removing the vapor stream from the distillation tower before producing the vapor

stream. The initial hydrocarbon extraction from the reservoir may be accomplished by drilling a well using hydrocarbon drilling equipment. The equipment and techniques used to drill a well and/or extract these hydrocarbons are well known by those skilled in the relevant art. Other hydrocarbon extraction activities and, more generally, other hydrocarbon management activities, may be performed according to known principles.

[0127] It should be understood that the numerous changes, modifications, and alternatives to the preceding disclosure can be made without departing from the scope of the disclosure. The preceding description, therefore, is not meant to limit the scope of the disclosure. Rather, the scope of the disclosure is to be determined only by the appended claims and their equivalents. It is also contemplated that structures and features in the present examples can be altered, rearranged, substituted, deleted, duplicated, combined, or added to each other.

CLAIMS

1. A method for separating fluids in a distillation tower, the method comprising:
providing a distillation tower comprising a controlled freeze zone section and a rectifier section, wherein the controlled freeze zone section and rectifier section are configured such that a vapor stream rises up from the controlled freeze zone section to the rectifier section;
maintaining the rectifier section in the distillation tower;
maintaining the controlled freeze zone section in the distillation tower, wherein the controlled freeze zone section forms solids from carbon dioxide within the distillation tower;
feeding a stream to the distillation tower, wherein the stream comprises the carbon dioxide;
reducing a carbon dioxide concentration of the vapor stream received by the rectifier section by:
feeding a first cryogenic fluid to the controlled freeze zone section, wherein the first cryogenic fluid comprises a substantially carbon-dioxide-free fluid; and
accumulating a rectifier section stream in at least one of a holding vessel and a sump of the rectifier section; and
terminating reducing the carbon dioxide concentration when the carbon dioxide concentration of the vapor stream travelling from the controlled freeze zone section to the rectifier section is less than or equal to a maximum carbon dioxide concentration.
2. The method of claim 1, wherein feeding the first cryogenic fluid comprises feeding the first cryogenic fluid from a cryogenic fluid storage container to a cryogenic fluid pump, then feeding the first cryogenic fluid from the cryogenic fluid pump to a cryogenic heat exchanger, and then feeding the first cryogenic fluid from the cryogenic heat exchanger to the controlled freeze zone section.
3. The method of claim 2, wherein feeding the first cryogenic fluid from the cryogenic heat exchanger to the controlled freeze zone section comprises spraying the first cryogenic fluid into the controlled freeze zone section.
4. The method of claim 3, wherein spraying the first cryogenic fluid comprises spraying the first cryogenic fluid via a controlled freeze zone section spray assembly of the controlled freeze zone section.
5. The method of claim 4, wherein the first cryogenic fluid comprises liquid natural gas.

6. The method of claim 3, wherein spraying the first cryogenic fluid comprises spraying the first cryogenic fluid via a cryogenic fluid spray assembly of the controlled freeze zone section and spraying a liquid stream from the rectifier section via a controlled freeze zone section spray assembly of the controlled freeze zone section.
7. The method of claim 6, wherein the first cryogenic fluid comprises liquid nitrogen.
8. The method of claim 1, wherein the maximum carbon dioxide concentration within the vapor stream comprises less than or equal to 6 percent of the stream.
9. The method of claim 1, further comprising directly feeding a rectifier section cryogenic fluid to the rectifier section.
10. The method of claim 9, wherein the first cryogenic fluid comprises a same cryogenic fluid as the rectifier section cryogenic fluid.
11. The method of claim 9, wherein the rectifier section cryogenic fluid comprises a substantially carbon dioxide free fluid.
12. The method of claim 9, wherein feeding the rectifier section cryogenic fluid comprises feeding the rectifier section cryogenic fluid from a cryogenic fluid storage container to a rectifier section cryogenic fluid pump, then feeding the rectifier section cryogenic fluid from the rectifier section cryogenic fluid pump to a rectifier section cryogenic heat exchanger and then feeding the rectifier section cryogenic fluid from the rectifier section cryogenic heat exchanger to the rectifier section.
13. The method of claim 1, further comprising feeding the rectifier section stream to a controlled freeze zone section spray assembly of the controlled freeze zone section when the carbon dioxide concentration travelling from the controlled freeze zone section to the rectifier section is less than or equal to the maximum carbon dioxide concentration.
14. The method of claim 6, further comprising reducing the carbon dioxide concentration of the stream received by the rectifier section by feeding a second cryogenic fluid to the controlled freeze zone section, wherein the second cryogenic fluid comprises a substantially carbon dioxide free fluid.

15. The method of claim 14, wherein feeding the second cryogenic fluid comprises feeding the second cryogenic fluid to a second cryogenic fluid pump, then feeding the second cryogenic fluid from the second cryogenic fluid pump to the controlled freeze zone section.
16. The method of claim 15, wherein feeding the second cryogenic fluid from the second cryogenic fluid pump to the controlled freeze zone section comprises spraying the second cryogenic fluid into the controlled freeze zone section.
17. The method of claim 14, wherein feeding the second cryogenic fluid comprises feeding the second cryogenic fluid to a second cryogenic fluid pump, then feeding the second cryogenic fluid from the second cryogenic fluid pump to a second cryogenic heat exchanger, and then feeding the second cryogenic fluid from the second cryogenic heat exchanger to the controlled freeze zone section.
18. The method of claim 17, wherein feeding the second cryogenic fluid from the second cryogenic heat exchanger to the controlled freeze zone section comprises spraying the second cryogenic fluid into the controlled freeze zone section.
19. The method of claim 16, wherein spraying the second cryogenic fluid comprises spraying the second cryogenic fluid via the controlled freeze zone section spray assembly.
20. The method of claim 14, wherein the second cryogenic fluid comprise liquid natural gas.
21. The method of claim 1, further comprising feeding a rectifier section stream portion of the rectifier section stream to a cryogenic fluid storage container.
22. The method of claim 21, wherein the rectifier section stream portion comprises at least one of the first cryogenic fluid, a second cryogenic fluid, a rectifier section cryogenic fluid and the solids.
23. The method of claim 22, further comprising separating the at least one of the first cryogenic fluid, the rectifier section cryogenic fluid and the second cryogenic fluid in the rectifier section stream portion from the solids in the rectifier section stream portion.
24. The method of claim 23, further comprising feeding the at least one of the first cryogenic fluid, the rectifier section cryogenic fluid and the second cryogenic fluid to at least one of a cryogenic fluid storage container and a sales pipeline.

25. The method of claim 23, further comprising collecting the solids at a cryogenic fluid storage container bottom of the cryogenic fluid storage container.