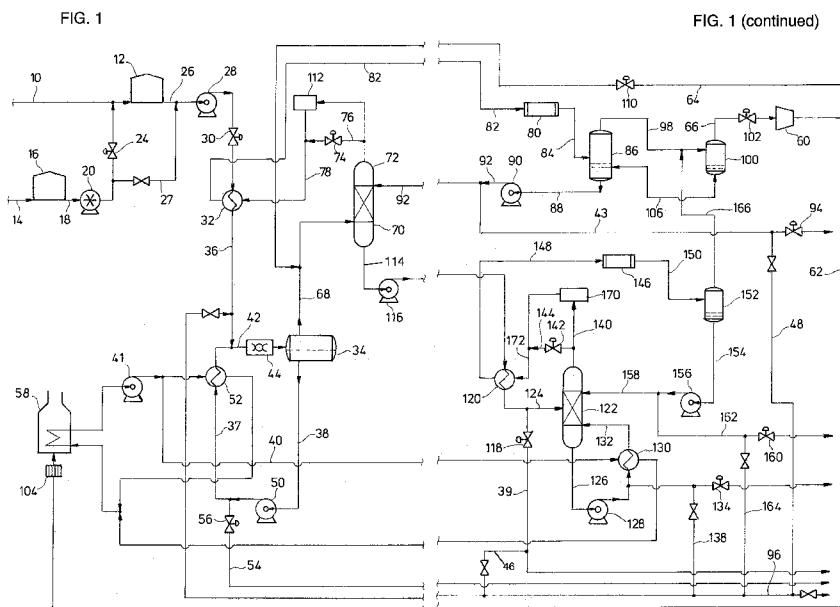




- (51) **International Patent Classification:**
B01D 53/14 (2006.01) *B01D 53/96* (2006.01)
- (21) **International Application Number:**
PCT/US2017/026374
- (22) **International Filing Date:**
6 April 2017 (06.04.2017)
- (25) **Filing Language:** English
- (26) **Publication Language:** English
- (30) **Priority Data:**
62/319,024 6 April 2016 (06.04.2016) US
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- (81) **Designated States** (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) **Designated States** (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) **Title:** PROCESS FOR TREATING CONTAMINATED FLUIDS

(57) **Abstract:** A process for recovering a processing liquid from a feed mixture having components which are more volatile and less volatile than the processing liquid in which the feed mixture is heated under conditions to prevent degradation of the processing liquid to produce a volatile component and a residuum stream, the volatile component and residuum stream being separated, at least a portion of the volatile component being compressed to produce a compressed vapor stream, the heat of compression of the compressed stream being used to heat the feed mixture.

PROCESS FOR TREATING CONTAMINATED FLUIDS

CROSS REFERENCE TO RELATED APPLICATION

This application claims priority to U.S. Application No. 62/319,024 filed on April 6, 2016 the disclosure of which is incorporated herein by reference for all purposes.

5 FIELD OF THE INVENTION

The process relates to the generation of compressible fluid streams and in its separation from or reaction with other compressible or non-compressible fluids. In a specific embodiment the present invention relates to an improved process for recovering processing fluids from contaminated fluid streams and, more particularly,
10 to a process for recovering a processing fluid from a mixture comprising water, a processing fluid having a higher boiling point than water, optionally at least one additional component that is more volatile than the processing fluid and water, and one component that is less volatile than, and can be dissolved or suspended in, the processing fluid under selectively chosen conditions e.g., pressure, temperature and
15 or fluid density.

BACKGROUND OF THE INVENTION

There are numerous industrial processes wherein a liquid, hereinafter referred to as a processing fluid, which can comprise one or more components, is
20 used in such a fashion that it becomes contaminated with, or contains, various components, some of which are more volatile than the processing fluid and some of which are less volatile and can be dissolved in the processing fluid. Usually, the components in the processing fluid are contaminants, although they may be desirable recovered components, depending on the process in which the processing
25 fluid is used. In such cases, it is almost universally desirable to separate the processing fluid from the less volatile and more volatile components so that the processing fluid can be reused in the process or simply recovered in a substantially pure state for reuse or other uses.

Numerous examples of the above described general scheme of using a
30 processing fluid abound. For example, it is well known that natural gas produced

from oil and gas wells, in addition to containing gaseous hydrocarbons, such as methane, ethane, etc., almost invariably contains water and acidic gases, such as CO₂ and H₂S. In cases where the natural gas contains water, it is very common for so-called gas hydrates or clathrate hydrates to form. These clathrate hydrates are crystalline compounds that occur when water forms a cage-like structure around guest molecules, particularly gaseous molecules.

While the phenomena can occur in any system wherein there is water and gaseous compounds, e.g., hydrocarbons, the problem, at times, becomes especially acute in the petroleum industry, not only with respect to the production of gaseous hydrocarbons such as natural gas, but also in the transporting and processing of natural gas. As noted, typical gas hydrates formed in petroleum (hydrocarbon) environments are composed of water and one or more guest molecules, such as methane, ethane, propane, isobutane, nitrogen, carbon dioxide, and hydrogen sulfide. However, it is also known that other guest molecules such as nitrous oxide, acetylene, vinyl chloride, ethyl bromide, oxygen, etc., can form clathrate hydrates.

With particular reference to natural gas systems and by example only, when gas hydrate crystals form, they can become a nuisance at least and pose a serious problem at worst. Gas hydrates can block transmission lines and plug blowout preventers, jeopardize the foundations of deep water platforms and pipelines, collapse tubing and casing, and foul process equipment, such as heat exchangers, compressors, separators, and expanders. To overcome these problems, several thermodynamic measures are possible in principle: removal of free water, maintaining an elevated temperature and/or reduced pressure, or the addition of freezing point depressants. As a practical matter, the last mentioned measure, i.e., adding freezing point depressants, has been most frequently applied. Thus, lower alcohols, such as methanol, ethanol, etc., and glycols have been added to act as antifreezes.

While processing fluids such as alcohols and glycols used in natural gas production, transportation, and processing are effective at reducing gas hydrate formation, their use is not without problems. As is well known, the production of natural gas is frequently accompanied by the production of brine, containing sodium chloride and other water-soluble salts. While these halides, such as the alkali metal halides, are readily soluble in water, they also exhibit substantial solubility in the

alcohols and glycols used to prevent gas hydrate formation. Accordingly, the processing fluid--in this case the alcohol, glycol, or the like--becomes contaminated with dissolved salts present in the produced water, as well as with certain gases, which, depending on the particular gas, are soluble in the processing fluid. Thus, this
5 presents a specific example where a processing fluid has been used, in this case to prevent hydrate formation, and has now become contaminated with a more volatile component and a less volatile, and in this case dissolved, component.

Again, using the example of natural gas production, transportation, and processing, it is necessary that the natural gas be freed of acidic components, such
10 as CO₂, H₂S, sulfur oxides, etc., some of which are quite toxic, all of which can lead to severe corrosion problems and in certain cases the formation of unwanted by-products. It is common to scrub the natural gas stream with processing fluids such as liquid amines, particularly alkanolamines such as monoethanolamine (MEA); diethanolamine (DEA); methyldiethanolamine (MDEA), proprietary blends of
15 additives and alkanolamines, as well as glycols such as mono-, di-, or tri-ethylene glycol and non-aqueous heat transfer fluids. Since scrubbing of natural gas to remove acidic gases is normally conducted on natural gas streams that have been substantially freed of water, the dissolved salt content of the natural gas stream from the gas stream is generally quite small. However, even though the ingress of
20 dissolved salt is low from the natural gas stream, continuous use of the amine process liquid for acid gas removal tends to cause the amine to break down with contaminants and create heat-stable, unregenerable salts. If the residual buildup of heat-stable salts (HSS) is permitted to build to typical levels in excess of 1% by weight, the amine performance will decline, corrosion increases rapidly with a
25 decline in pH, and the amine solution begins to foam, creating excessive process liquid losses. Accordingly, the processing fluid, e.g., the alkanolamine, will generally contain dissolved, less volatile components at a much smaller concentration than in the case of an alcohol or glycol used to prevent gas hydrate formation. Nonetheless, even in this instance, the processing fluid now presents a case where, after use, it
30 contains more volatile components, e.g., CO₂ H₂S, etc., and perhaps a small amount of less volatile and dissolved component.

In the case where treatment of the natural gas to prevent gas hydrate formation and/or remove acidic gases is conducted on offshore platforms, several

problems are encountered. For one, the alcohols, glycols, and alkanolamines can be toxic to marine life and accordingly, once spent, e.g., saturated with contaminants that they are being used to remove, cannot be discharged overboard. Aside from ecological concerns, such a method is economically not feasible since it requires a
5 constant replenishment of the processing fluid. Indeed, such a process would not be economically feasible in land-based refineries, chemical plants, or the like.

U.S. Pat. Nos. 5,152,887; 5,158,649; 5,389,208; and 5,441,605 all of which are incorporated herein by reference and deal with processes and apparatus for reclaiming and/or concentrating waste aqueous solutions of gas treating chemicals.
10 Additionally, U.S. Pat. Nos. 4,315,815, and 4,770,747, both of which are incorporated herein by reference, likewise deal with processes for reclaiming or recovering gas-treating liquids. U.S. Pat. No. 5,389,208, incorporated herein by reference for all purposes, discloses and claims a method for reclaiming an impurity-containing waste aqueous solution of a gas-treating chemical that basically involves
15 vacuum distillation of the spent material under temperature conditions that prevent decomposition of the gas-treating chemical and in such a fashion that the process can be operated in apparatuses made of carbon steel, as opposed to more exotic materials of construction, without causing substantial corrosion of the apparatus.

In U.S. Pat. No. 5,993,608 and U.S. Pat. No. 6,508,916, incorporated herein
20 by reference for all purposes, disclose and claim processes for recovering processing fluids wherein components less volatile than the processing fluid such as dissolved and/or suspended solids are removed from the processing fluid under conditions that prevent any substantial degradation of the processing fluid and provide for recycle of water, refined processing fluid or a mixture thereof back to the
25 front end of the process.

SUMMARY OF THE INVENTION

It is therefore an object of the present invention to provide a process for separating contaminants from a processing fluid comprised of at least one or more volatile and one or more less volatile components contained in the processing fluid by manipulating the conversion of the processing fluid from a substantially non-compressible fluid to a compressible fluid and then further manipulating the thus formed compressible fluid generated to convert it to a state more readily convertible back to a substantially non-compressible fluid. Through selective process manipulation and use of appropriate processing equipment, intermediate process pressure(s) can be altered. The concept can be applied to sub-atmospheric, atmospheric or above atmospheric systems for components within the process being considered. Location of the processing equipment can be at a stage in the process where compressible fluids exist or are created. By example the equipment can be located in a single intermediate location in the system or can be staged at various locations within the system utilizing multiple equipment units in series or in parallel. More specifically, the equipment can be used to alter midstream pressure profiles to impart a lower pressure stream upstream and direct the higher pressure stream outlet flow to one or more locations that can benefit from the pressure increase. This can be used to assure optimum pressure at a specific dew point or at the point of vaporization.

Non-limiting examples of equipment suitable for use in the process of the present invention include dynamic compressors such as centrifugal compressors, axial flow compressors, acoustic compressors, reciprocating compressors, scroll compressors etc. Another mode of application could use a steam ejector or steam inductor. The process application dictates the selection of the type of equipment installed and the specific location thereof.

Any temperature increase downstream of the selected equipment can be utilized to pre-heat or heat feed streams, intermediate streams or recycle streams or any combination thereof. By way of example, the increased temperature can be utilized to improve chemical reaction kinetic rates downstream of the equipment. Upstream of the equipment, there is the opportunity to operate the system at a lower temperature.

Density increases beneficially increase the mass flow rate of the fluids for a given upstream energy input. The increase in density from such compression equipment will improve the rate of condensation at a specific dew point. Alternately, the system can be adjusted to retain the same mass flow rate with less upstream
5 energy input.

The above objects have the cumulative advantage of a smaller carbon footprint, lower energy consumption and energy efficiency per mass of fluid treated. Further to energy related advantages the resulting equipment required for the process can be made smaller which translates into a capital saving as well as a
10 savings in overall equipment size and weight. This is especially beneficial to offshore or mobile applications.

This invention can be applied to batch mode operations or a continuous mode operation for recovery of a processing fluid wherein decomposition of the processing fluid is prevented at reduced vacuum conditions and 95% or more of the processing
15 fluid is recovered essentially free of the at least one of the more and less volatile components.

A further object of the present invention is to provide a process for treating a processing fluid to remove from the processing fluid more volatile and less volatile components using, at least in part, processing equipment made of carbon steel.

20 Yet another object of the present invention is to provide a process for separating dissolved and/or suspended solids from a processing fluid under conditions that prevent any substantial degradation of the processing fluid and enhance recovery of the processing fluid.

Another object of the present invention is to provide a multiple stage process
25 for separating dissolved and/or suspended solids from a processing fluid in a continuous or batch mode operation and under conditions that prevent any substantial degradation of the processing fluid.

Still a further aspect of the present invention is a feed of processing fluid that is subjected to flashing to produce a vapour stream at least a portion of the vapour
30 stream being compressed to increase demand for the feed to the flashing stage to maintain equilibrium.

The above and other objects of the present invention will become apparent from the drawing, the description given herein, and the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic, flow diagram of one embodiment of the process of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The term "processing fluid" as used herein refers to any aqueous or non-aqueous liquid that can contain one or more components and includes, without limitation, gas treating chemicals such as alkanolamines, e.g., monoethanolamine (MEA), diethanolamine (DEA), methyldiethanolamine (MDEA), proprietary blends of additives and alkanolamines; or glycols such as monoethylene glycol (MEG), diethylene glycol (DEG), triethylene glycol (TEG), tetraethylene glycol (TTEG), and propylene glycol (PEG), as well as halogenated solvents, liquid hydrocarbons including aromatic compounds, olefinic compounds, aliphatic compounds, water, and mixtures of water and other water-miscible materials, etc. Further, a processing fluid as used herein refers to a liquid that is used in a particular process such that it becomes contaminated with, or at least after use contains, components not normally present in the processing fluid. Thus, the processing fluid can be a gas scrubbing medium used to remove undesirable contaminants from gas streams, a selective solvent to recover desirable components from gaseous or liquid streams, a medium used to treat solids to selectively remove certain components of the solids, etc. Accordingly, while in the examples given herein the invention will be described with reference to the scrubbing of gas streams, it will be understood that the invention is not so limited.

In cases where the processing fluid is used in greenhouse gas operations, non-limiting examples of contaminants or components that may be present in the processing fluid and that need to be removed include acid gases such as carbon dioxide, sulfur oxides, nitrogen oxides, combustion byproduct particulates and more volatile liquid components such as water, etc. Non-limiting examples of less volatile components or contaminants present in the processing fluid that need to be removed there from include particulates, inorganic salts such as alkali metal halides; iron salts; salts of organic acids; carbonates; and numerous other organic and inorganic components that are less volatile than the processing fluid and that are dissolved in the process liquid or that are present in generally non-filterable form, e.g., colloidal suspensions. While generally speaking the less volatile component will be a dissolved and/or suspended solid, the latter being generally non-filterable, it is to be understood that the less volatile component can comprise a liquid that is higher boiling than the processing fluid and that, because it is a liquid, would not normally

cause fouling or solid buildup in the lines of the process but that, under certain conditions, can form solids or emulsions and therefore must be removed from the processing fluid. Further, such high boiling liquids may affect the operating efficiency and corrosivity of the processing fluid and therefore must be removed or at least
5 have their concentration reduced in the processing fluid to maintain overall processing fluid performance efficiency.

As used herein, the term "feed mixture" includes water if present in the processing fluid, a processing fluid having a higher boiling point than water, and optionally at least one additional component which is (a) more volatile than the
10 processing fluid or (b) at least one less volatile component than the processing fluid, the concentration of such more or less volatile components being dependent upon the nature of the processing fluid, the type of processing in which the processing fluid is used, or (c) both (a) and (b). Thus, the feed mixture, as used in the description that follows, refers to the material that is to be treated in accordance with
15 the process of the present invention to reduce or substantially eliminate the more volatile components from the feed mixture and substantially reduce, if not eliminate, the less volatile components from the feed mixture.

As indicated above, depending upon which processing fluid is being used and the conditions under which it is being used, it will contain more or less of the less
20 volatile component, i.e., the dissolved and/or suspended component. In cases where the less volatile component is present in relatively small amounts, e.g., from about 10 ppm to about 100,000 ppm by weight of the feed mixture, and depending on the particular processing fluid employed, reduction of the concentration of the less volatile component can generally be accomplished by purge, e.g. a blowdown
25 stream in the process. Alternately, when the less volatile components in the feed mixture are present in higher amounts, e.g., from about 1 to about 40% by weight, and again depending on the particular processing fluid employed, other steps may be necessary to reduce the concentration of the less volatile component in the processing fluid to maximize recovery of the processing fluid. For example, a solids-
30 liquid separation step can be employed with the separated liquid being recycled to the first separation zone.

Additionally, depending on the application, the processing fluid may become contaminated not only with dissolved and/or suspended contaminants, but may have

more volatile or less volatile contaminants that require separation from the processing fluid to re-establish the purity of the original contaminant free processing fluid through the application of multiple fractionation steps. All of which can be enhanced by incorporating the intermediate stage compression devices and
5 controls.

According to the process of the present invention, a stream of a feed mixture comprising a processing fluid having a higher boiling point than water, optionally at least one additional component that is more volatile than the processing fluid and at least one component that is less volatile than the processing fluid, is introduced into
10 a flash zone e.g., by combining it with a first residuum recycle stream that passes through a first heating zone and is initially heated to a temperature sufficient to volatilize at least some of the water, if present in the feed mixture, and at least a portion of the processing fluid prior to being introduced into the flash zone. For the ease of description, it shall be assumed that the feed mixture contains water and this
15 is reflected in the following discussion.

The temperature in the first heating zone is maintained below the decomposition temperature of the processing fluid, and there is produced a first vapour stream comprising volatilized water and a volatilized portion of the processing fluid, and a first residuum comprising the unvolatilized processing fluid, a
20 reduced concentration (perhaps none) of the more volatile component, and at least some of the less volatile component. The first vapour stream is separated from the first residuum in a first separation zone and directed through a feed cross-exchanger wherein some of the heat of vapourization is transferred to the feed mixture prior to its introduction into the first residuum recycle stream.

Alternately, the first vapour stream can be directed completely or partially through a first compression zone prior to the feed cross-exchanger. The presence of the first compression zone at this location will reduce the pressure in the first separation zone making the feed mixture volatilize more rapidly at a given heat input. This also increases mass flow rate as it results in more demand for feed mixture to
25 the flash zone or still. This lowering of pressure can be utilized to reduce the required heat input for the feed mixture mass flow rate, thereby reducing energy requirements and a commensurate reduction of greenhouse gases associated with the heat source.
30

Further energy efficiency and greenhouse gas reduction results from the utilization of the heat generated at the outlet of the first compression zone in the feed cross-exchanger to further heat the feed mixture prior to its introduction into the flash zone e.g., via the first heating zone.

- 5 The partially condensed first vapour stream exiting the feed cross-exchange zone enters a fractionation zone where the less volatile components can be condensed and delivered to an off unit location or recycled back to the front of the process to facilitate start-up or as operating conditions demand.

10 With reference then to Figure 1, a feed mixture, such as, for example, an alkyl ethanolamine such as MDEA, that has been used to remove acidic gases from a greenhouse gas stream and that contains acidic gases, water, MDEA (the processing fluid), and less volatile components, e.g., dissolved or suspended solids, high boiling liquids, low boiling liquids, etc., is introduced via line 10 from a gas processing facility to a feed surge tank, 12. Chemical additives used to condition the
15 feed mixture may include but not be limited to antifoaming agents, pH adjustment chemicals, corrosion inhibitors, flocculating agents, etc. and can be delivered to a chemical additive tank or tanks via line 14. For sake of simplicity only one such tank, 16, has been illustrated. The required chemical additive is delivered through line 18 to the chemical additive injection pump 20, which in turn delivers the required
20 amount of chemical additive via line 22 at a rate controlled by a flow control valve 24 to line 10. Alternately, chemical additives can bypass the conditioning tank and be directly added to the feed stream exiting the feed surge tank via line 26 via line 27 to inject the correct amount of chemical additive. The conditioned feed mixture is transferred from line 26 via feed pump 28, through a flow control valve 30, to the
25 feed cross-exchanger 32. The pre-heated feed mixture can be directed to the first separation zone formed by a still or flash vessel 34 via line 36 where the feed mixture will be mixed with the first heated residuum recycle stream introduced to the still or flash vessel 34 through line 38 and line 37 via line 42, as well as through flow mixer 44. Those skilled in the art can appreciate that line 36 may be directed into
30 the still 34 directly to allow the feed mixture to be vapourized upon contact with the hot vapours and first residuum stream contained therein.

Line or flow mixers are employed since it is preferable to operate the process in a continuous manner, albeit both batch mode and continuous modes of operation

apply equally. Typical of such mixers are jet mixers, injectors, orifices and mixing nozzles, centrifugal pumps, cyclonic devices and agitated line mixers. It will be appreciated that while line or flow mixers are preferred, in certain cases, if holding time is desired, agitated vessels may be employed. The mixture of the first heated
5 residuum recycle stream and the pre-heated feed mixture from line 63, after being thoroughly mixed in mixer 44, is introduced via line 42 into a first separation zone formed by a still or flash vessel 34. As explained hereafter, heat necessary to effect separation in the first separation zone is imparted to the feed mixture by heat transfer from the heated first residuum recycle stream from line 42 to the feed
10 mixture entering mixer 44 from line 36 into the still or flash vessel 34. It will be appreciated that the first separation zone in still or flash vessel 34 includes a lower, substantially liquid phase zone and an upper, substantially vapour phase zone, an interface being formed between the two zones. It can be further appreciated that depending on the operating conditions desired, the feed mixture can be
15 simultaneously directed through line 42 to still 34 along with recycled products streams via line 48 and line 36 to line 42 through the mixer 44 to still 34. Such simultaneous flow conditions can be utilized to adjust temperature profiles in still 34, modify fluid levels in still 34 and increase feed mixture rates to still 34.

With continued reference then to Figure 1, the first residuum exiting pump 50
20 can be directed via line 37 to heat exchanger 52 and exit as the first heated residuum stream via line 42 through mixer 44 to the still 34, or a portion of the first residuum can be directed via line 54 through flow control valve 56 for discharge to an off unit location. The discharge of the first residuum through line 54 is done to maintain stable operating conditions through the removal of the non-volatile
25 contaminants in the feed mixture that are concentrated in the first residuum stream. This can be done on either a continuous purge or batch purge basis, depending on the type and quantity of the non-volatile contaminant or contaminants. Those skilled in the art will appreciate that actual contaminant levels in the first residuum stream in line 37 will dictate the actual proportion of first residuum diverted to line 54, relative
30 to the flow directed to heat exchanger 52. It can also be appreciated by those skilled in the art that the discharge from the first residuum pump 50 can be directed to a direct fired heater 58 to provide heat energy into the recycled first residuum stream.

In the embodiment shown in Figure 1, the first separation zone in still 34 is maintained under a vacuum by means of a vacuum pump such as compressor 60, forming part of a downstream, separation zone. In the process described in U.S. Pat. No. 5,993,608 and U.S. Pat. No. 6,508,916, vacuum conditions ranging from 5 inches of mercury to 16 inches of mercury were employed to effect separation of purified processing fluid from the contaminants without degrading the processing fluid. Ultimate discharge conditions desired in line 62 and line 64 will dictate which vacuum condition is set in line 66.

Compressible fluid vapours or gases flashed from the heated feed mixture in still 34 as the first vapour stream pass overhead via line 68 to a first fractionation column 70.

The more volatile component or components of the first vapour stream will exit the top of the fractionation column 70 as the second vapour stream into line 72 to a pressure control valve 74, line 76 and line 78 and enter the feed cross exchanger 32, wherein it will be partially condensed to its substantially non-compressible liquid state. The mixed second vapour stream and partially condensed second vapour stream are directed by gravity flow to an air cooled exchanger 80 via line 82 and the mostly condensed second vapour stream via line 84 into a gas/liquid separator 86, e.g., a gravity separator allowing sufficient stilling time to effect gas/liquid separation.

This mostly condensed second vapour stream will exit the gas/liquid separator 86 through line 88, to pump 90 where a portion of this condensed second vapour stream is recycled back to the top of the first fractionation column 70 via line 92 as reflux so as to control the composition of the second vapour stream exiting the top of the first fractionation column 70, with the balance passing through a flow control valve 94 on line 43 for delivery to an off unit location, recombining with the less volatile condensed first, second and third vapour streams via line 48 into line 96 or recycle to the front of the process into line 36 to facilitate start-up or as operating conditions demand.

Any non-condensed components that were in the second vapour stream are removed from separator 86 via line 98 to a second gas/liquid separator 100 with the same separation capabilities as separator 86, and via line 66 and a flow control valve 102 to compressor 60 and are sent via line 62 through a flame arrestor 104

into the firebox of heater 58 for combustion. Any vapours that are condensed in the second gas/liquid separator 100 are directed back to the first gas/liquid separator 86 via line 106 via gravity flow. Those skilled in the art can appreciate that depending on equipment elevation, a transfer pump may be required on line 106 to affect any liquid transfer back to the first gas/liquid separator 86.

A portion of the compressor 60 discharge stream can also be directed to line 68 via line 64 through a pressure control valve 110 to adjust overall system pressure.

Alternately, the more volatile component or components of the first vapour stream exiting the fractionation column 70 as the second vapour stream can be directed completely or partially through a first compression device 112 prior to the feed cross exchanger 32 as dictated by manipulation of valve 74. Selective control of the discharge pressure from the second compression device 112 will allow the more volatile component or components to be more readily condensed in the feed cross exchanger 32 and the downstream air cooled exchanger 80. Design of the first fractionation column 70 will take into consideration the lower pressure induced upstream of the first compression device 112 to maintain its ability to properly fractionate the less volatile components from the more volatile components.

The presence of the first compression device 112 at this location will selectively and further reduce the pressure in the first separation zone 34, making the feed mixture volatilize more rapidly at a given heat input. This lowering of pressure can therefore be utilized to reduce the required heat input required provided by heat exchanger 52 via heater 58 for the same feed mixture mass flow rate, thereby reducing energy requirements and a commensurate reduction of greenhouse gases associated with the heat source. Further, this compression stage increases mass flow as noted above.

Further energy efficiency and greenhouse gas reduction results from the utilization of the heat generated at the outlet of the first compression device 112 in the feed cross-exchanger to further heat the feed mixture prior to its introduction into the first residuum recycle stream.

The more volatile components in the first vapour stream can be fractionated from the less volatile components and depending on the operating parameters set for the first fractionation column 70 the latter components can be thoroughly

condensed, collected at the base of the first fractionation column 70 and via line 114, pump 116, and through a flow control valve 118 on line 39 and be delivered to an off unit location or recycled back to the front of the process to facilitate start-up or as operating conditions demand or recombined with the other condensed vapour streams via line 46 as described more thoroughly below.

With continued reference to Figure 1, for applications in which further fractionation of the condensed first vapour stream components is required, the condensed first vapour stream exiting the first fractionation column via line 114 to pump 116 can be directed through second column feed cross exchanger 120, line 124 into a second fractionation column 122.

A second residuum stream consisting on the less volatile components in the condensed first vapour stream recycle through line 126, to circulation pump 128, through heat exchanger 130 and line 132 to the second fractionation column 122. Heat energy imparted to the pre-heated first vapour stream by this recycled second residuum and hot vapours travelling up the second fractionation column 122 will vapourize the more volatile components in the condensed first vapour stream entering the column 122.

The recycled second residuum content in the second fractionation column 122 can be controlled by continuous or batch discharge through flow control valve 134 on line 136 for delivery to an off unit location, recombining with other condensed vapour streams via line 138, or for recycle to the front of the process to facilitate start-up or as operating conditions demand or for delivery off unit via line 96.

The more volatile component or components of the condensed first vapour stream will exit the top of the fractionation column 122 as the third vapour stream into line 140 to a pressure control valve 142, line 140 and line 144 and enter the second feed cross exchanger 120, wherein it will be partially condensed to its substantially non-compressible liquid state. The mixed third vapour stream and partially condensed third vapour stream are directed by gravity flow to an air cooled exchanger 146 via line 148 and the mostly condensed third vapour stream via line 150 into a gas/liquid separator 152, e.g., a gravity separator allowing sufficient stilling time to effect gas/liquid separation.

This mostly condensed third vapour stream will exit the gas/liquid separator 152 through line 154, to pump 156 where a portion of this condensed third vapour

stream is recycled back to the top of the second fractionation column 122 via line 158 as reflux so as to control the composition of the third vapour stream exiting the top of the second fractionation column 122, with the balance passing through a flow control valve 160 on line 162 for delivery to an off unit location, recombining with
5 other condensed streams via line 164 into line 48 or recycle to the front of the process into line 36 to facilitate start-up or as operating conditions demand or for delivery off unit via line 96.

Any non-condensed components that were in the third vapour stream are removed from separator 152 via line 166 to a second gas/liquid separator 100 with
10 the same separation capabilities as separator 86, and via line 60 and a flow control valve 102 to compressor 60 and are sent via line 62 through a flame arrestor 104 into the firebox of heater 58 for combustion. Any vapours that are condensed in the second gas/liquid separator 100 are directed back to the first gas/liquid separator 86 via line 106 via gravity flow. Those skilled in the art can appreciate that depending
15 on equipment elevation, a transfer pump may be required on line 106 to affect any liquid transfer back to the first gas/liquid separator 86.

Alternately, the more volatile component or components of the third vapour stream exiting the second fractionation column 122 as the third vapour stream can be directed completely or partially through a second compression device 170 prior to
20 the second feed cross exchanger 120 as dictated by the setting of control valve 142. Selective control of the discharge pressure from the second compression device 170 will allow the more volatile component or components to be more readily condensed in the second feed cross exchanger 120 and the downstream air cooled exchanger 146. Design of the second fractionation column 122 will take into consideration the
25 lower pressure induced upstream of the second compression device 170 to maintain its ability to properly fractionate the less volatile components from the more volatile components.

Further energy efficiency and greenhouse gas reduction results from the utilization of the heat generated at the outlet of the second compression device 170
30 in the second feed cross-exchanger to further heat the condensed first vapour stream mixture prior to its introduction into the second residuum recycle stream.

With continued reference to Figure 1, the more volatile component or components of the second vapour stream will exit the top of the second fractionation

column 122 as the third vapour stream into line 140 to a pressure control valve 142, line 144, line 172 and enter the second feed cross exchanger 120, wherein it will be partially condensed to its substantially non-compressible liquid state. The mixed third vapour stream and partially condensed third vapour stream are directed by gravity flow to an aerial cooler 146, wherein it will be condensed to its substantially non-compressible liquid via line 148 and the mostly condensed third vapour stream via line 150 into a gas/liquid separator 152, e.g., a gravity separator allowing sufficient stilling time to effect gas/liquid separation. Again those skilled in the art can appreciate that depending on equipment elevation, a transfer pump may be required on line 150 to affect any liquid transfer to the first gas/liquid separator 152.

This mostly condensed third vapour stream will exit the gas/liquid separator 152 through line 154, to pump 156 where a portion of this condensed third vapour stream is recycled back to the top of the second fractionation column 122 via line 158 as reflux so as to control the composition of the third vapour stream exiting the top of the second fractionation column 122, with the balance passing through a flow control valve 160 on line 162 for delivery to an off unit location, recombining with other condensed streams via line 164 into line 48 or recycle to the front of the process into line 36 to facilitate start-up or as operating conditions demand or for delivery off unit via line 96.

Any non-condensed components that were in the third vapour stream are removed from separator 152 via line 166 to a second gas/liquid separator 100 with the same separation capabilities as separator 86, and via line 66 and a flow control valve 102 to compressor 60 and are sent via line 62 through a flame arrestor 104 into the firebox of heater 58 for combustion. Any vapours that are condensed in the second gas/liquid separator 100 are directed back to the first gas/liquid separator 86 via line 106 via gravity flow. Those skilled in the art can appreciate that depending on equipment elevation, a transfer pump may be required on line 106 to affect any liquid transfer back to the first gas/liquid separator 86.

In reference to Figure 1, heating is demonstrated through the use of a direct fired heater with the recycle residuum circulating through the heated zone at conditions to maintain the residuum bulk fluid below its degradation temperature and any heat gained by the recycled residuum passing through the heater is transferred to the feed mixture entering the still 34 as previously described. Heating is also

demonstrated through the use of a secondary heating loop that can be either a hot oil system, steam, high pressure hot water or other that can impart sufficient energy through the heat exchangers 52 and 120. Again the heat transferred in all cases is maintained such that the bulk fluid temperature of the residuum streams is below
5 their respective degradation temperatures.

Air or aerial cooling can also be supplemented with ancillary cooling such as water misting, etc. Thus water cooling can be applied to these embodiments. Further or supplemental cooling may include refrigeration, evaporative cooling, etc. As such, all known cooling technologies can be applied as appropriate to the final
10 design, layout and availability of utilities.

For those skilled in the art, it can be seen that the heating and cooling configurations described are considered interchangeable such that depending on the availability of utilities, functional space, etc. the specific configuration will be determined by such constraints and shall not limit the descriptions of these
15 embodiments.

The foregoing description illustrate selected embodiments of the present invention. In light thereof, variations and modifications will be suggested to one skilled in the art, all of which are in the spirit and purview of this invention.

The process of the present invention offers great advantages. Pressure can
20 be altered and the process can be used at sub-atmospheric, atmospheric or above atmospheric conditions. Location of the compression equipment can be at the point in the process where compressible fluids exist or are created. By example the compression equipment can be located in middle of system or can be staged within the system utilizing compression equipment units in series or in parallel. More
25 specifically, the compression equipment can be used to alter midstream pressure profiles to impart lower pressure upstream and direct the higher pressure outlet flow to one or locations that can benefit from the pressure increase. This can be used to assure optimum pressure at a specific dew point or at the point of vaporization. Perhaps more importantly, the overall effect of compression downstream from the
30 still intensifies mass flow rate through the system. The boost offered by compression raises the vapor demand from the flash vessel or still resulting in a concomitant enhanced feed into the still in order to maintain the vapor withdrawal

rate from the still. Additionally, production/flow rate can be accomplished by adjusting the speed of the compressors in the compression zone(s).

Temperature increase downstream of the selected compression equipment resulting from heat of compression can be utilized to pre-heat or heat feed streams, intermediate streams or recycle streams or any combination thereof. By way of example, the increased temperature can be utilized to improve chemical reaction kinetic rates downstream of the compression equipment. Upstream of the compression equipment, there is opportunity operate the system at a lower temperature, depending on system operating conditions.

10 Density beneficially increases the mass flow rate of the fluids for a given upstream energy input. Alternately, the process can be adjusted to retain the same mass flow rate for less upstream energy input. The increase in density from such compression equipment will improve the rate of condensation at a specific dew point.

As noted, among other benefits the process of the present invention utilizes heat of compression to heat or preheat streams thereby reducing overall energy required by the process. Furthermore, the use of one or more compression stages results in reduction in upstream pressures resulting in higher recovery rates and again overall energy required by the process.

20 While the process has been described with specific reference to the use of compressors or compression zones, other equipment can be utilized non-limiting examples of which include steam injectors or steam inductors.

The above benefits have the cumulative advantage of a smaller carbon footprint, lower energy consumption and energy efficiency. Further to energy related advantages the resulting equipment required for the process can be made smaller which translates into a capital saving as well as a saving overall equipment size and weight. This is especially beneficial to offshore or mobile applications.

WHAT IS CLAIMED IS:

1. A process for recovering a processing liquid from a feed mixture comprising a processing liquid, at least one component that is more volatile than said processing liquid and at least one component that is less volatile than said
5 processing liquid comprising:
 - providing a stream of said feed mixtures;
 - introducing said feed mixture into a first heating zone and initially heating said feed mixture to a temperature sufficient to volatilize at least some of said more volatile component and at least a portion of said processing liquid to produce a first
10 vapour stream comprising at least some of said more volatile component and said volatilized portion of said processing liquid and a first residuum stream comprising said processing liquid and at least some of said at least one less volatile component;
 - separating said first vapour stream from said first residuum stream;
 - compressing at least a portion of said first vapour stream to produce a first
15 compressed stream; and
 - utilizing at least a portion of the heat of compression of said first compressed stream to heat said feed mixture.
2. The process of claim 1, wherein said first heating zone is operated at sub-atmospheric pressure.
- 20 3. The process of claim 1, wherein said first vapour stream is fractionated to produce an overhead stream and a bottoms stream.
4. The process of claim 3, wherein said overhead stream is introduced into a compression zone to produce said first compressed stream.
5. The process of claim 4, wherein compression of said first vapour
25 stream increases demand for feed mixture in said first heating zone.
6. A process for recovering a processing liquid from a feed mixture comprising a processing liquid, at least one component that is more volatile than said processing liquid, and at least one component that is less volatile than said processing liquid comprising:
30
 - providing a stream of said feed mixtures;
 - introducing said feed mixture into a first heating zone and initially heating said feed mixture to a temperature sufficient to volatilize at least some of said more volatile component and at least a portion of said processing liquid to produce a first

vapour stream comprising at least some of said more volatile component and said volatilized portion of said processing liquid, and a first residuum stream comprising said processing liquid and at least some of said one less volatile component;

5 separating a first vapour stream from said first residuum stream in a first separation zone;

separating said first vapour stream into a second vapour stream and a heavier fraction;

compressing at least a portion of said second vapour stream to produce a first compressed stream;

10 utilizing at least a portion of the heat of compression of at least a portion of said first compressed stream to heat said feed mixture;

separating said heavier fraction into a third vapour stream and a second residuum stream;

15 compressing at least a portion of said third vapour stream to produce a second compressed stream; and

utilizing at least a portion of the heat of compression of at least a portion of said second compressed stream to heat said heavier fraction prior to separation of said heavier fraction into said third vapour stream and said second vapour stream.

20 7. The process of claim 6, wherein said first heating zone is operated at sub-atmospheric pressure.

8. The process of claim 6, wherein said first vapour stream is fractionated to produce a first overhead stream and a bottoms stream.

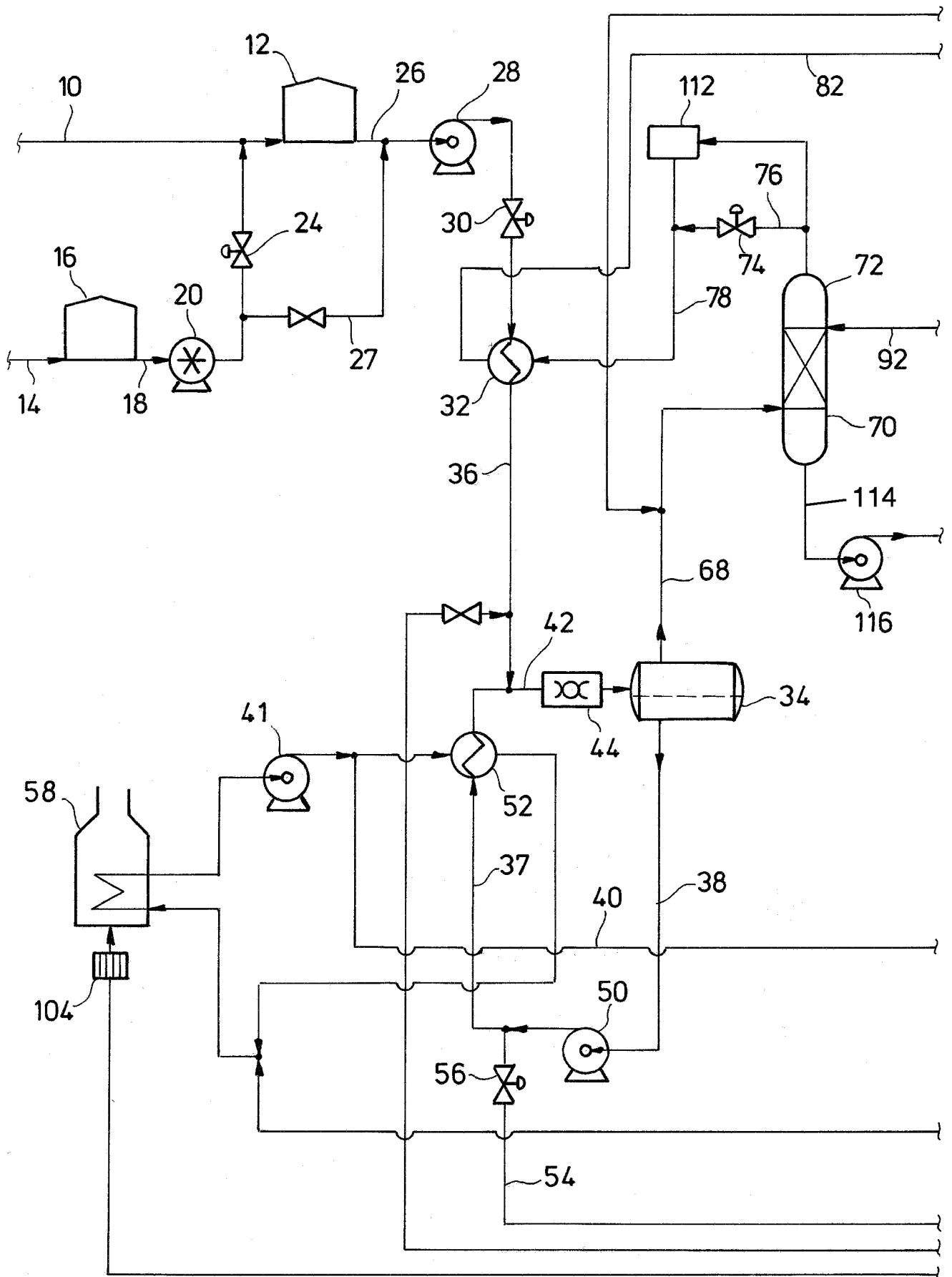
9. The process of claim 8, wherein said first overhead stream is introduced into a first compression zone to produce said first compressed stream.

25 10. The process of claim 9, wherein compression of said first vapour stream increases demand for feed mixture in said first heating zone.

11. The process of claim 8, wherein said bottoms stream is fractionated to produce a second overhead stream, said second overhead stream being introduced into a second compression zone to form said second compressed stream.

30

FIG. 1



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US17/26374

A. CLASSIFICATION OF SUBJECT MATTER

IPC - B01D 53/14, 53/96 (2017.01)

CPC - B01D 53/14, 53/1425, 53/96

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History document

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

See Search History document

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

See Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6,508,916 B1 (RAZZAGHI M et al.) January 21, 2003; figures 1, 2; column 6, lines 1-50; claim 1	1-3
Y	US 2,619,453 A (ANDERSEN R) November 25, 1952; column 4, lines 20-50; column 8, lines 5-15	1-3
A	US 5,441,605 A (BEASLEY T et al.) August 15, 1995; entire document	1-11
A	US 2012/0241309 A1 (MILLARD MG) September 27, 2012; entire document	1-11
A	US 2013/0192465 A1 (TROFIMUK T et al.) August 1, 2013; entire document	1-11
A	US 5,158,649 A (BEASLEY T et al.) October 27, 1992; entire document	1-11
A	US 2010/0062926 A1 (WOODHOUSE S et al.) March 11, 2010; entire document	1-11

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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

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

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

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

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

"&" document member of the same patent family

Date of the actual completion of the international search

15 June 2017 (15.06.2017)

Date of mailing of the international search report

17 JUL 2017

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