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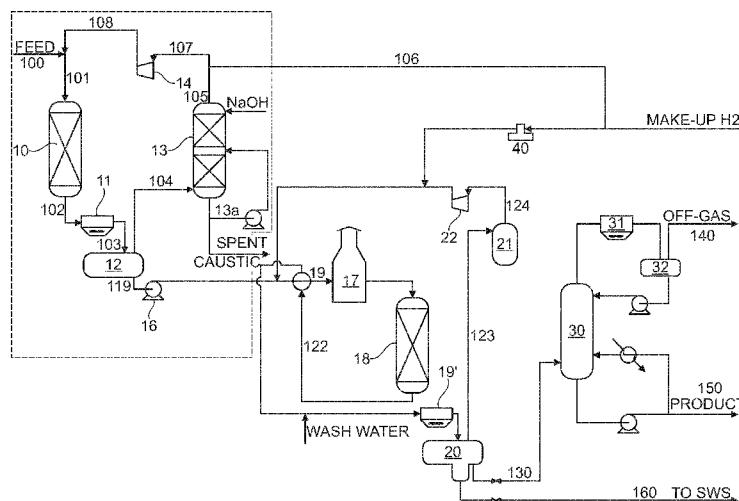
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(54) Title: REMOVAL OF HALIDES FROM HYDROCARBON LIQUID



Title: Removal of halides from hydrocarbon liquid**FIELD OF THE INVENTION**

5 This invention relates to a process and a system for removing halides from a hydrocarbon stream comprising one or more halides.

BACKGROUND OF THE INVENTION

10 Refinery processes comprise a plurality of treatments of hydrocarbon rich streams in order to provide products in the form of gasoline, diesel, etc. Such treatments comprise hydro-treatment, hydro-cracking, fractionation and stripping, as well as intermediate heat exchange and removal of impurities.

15 Typically, the hydrocarbon rich stream to be processed in the refinery comprises halides, e.g. comprising chlorine. Halides are unwanted in the product(s) and are also disadvantageous within the refinery plant due to corrosion issues within the units of the plant.

20 US5,595,648 relates to a process for removing acidic halides from dry liquid hydrocarbon streams by contact with large particles of low surface area solid caustic, such as a bed of NaOH pellets. Salt formed by neutralization reaction deposit as solids on the surface of the solid caustic.

25 In US5,595,648 the solid caustic treater 30 has to be periodically removed from service or bypassed for bed rejuvenation. This either provides downtime or halides in the system. The invention aims at providing a process and system for removing halides from a hydrocarbon stream arranged for continuous operation.

BRIEF SUMMARY OF THE INVENTION

30 The process of the invention for removing halides from a hydrocarbon stream comprising one or more organic halides, comprises the steps of:

- (a) providing said hydrocarbon stream to a hydro-treatment reactor comprising a material catalytically active in converting organic halides into inorganic halides,
- (b) providing a hydrogen rich stream to said hydro-treatment reactor,

- (c) letting the hydrocarbon stream and the hydrogen rich stream react at the presence of the catalytically active material, rendering a first hydrogenated product stream,
(d) separating said first hydrogenated product stream into a first vapor phase and a first liquid phase in a separator unit, and
5 (e) removing halides from said first vapor phase to create a second vapor phase.

Steps (a) and (b) may be combined, so that the hydrocarbon stream and the hydrogen rich stream are combined to a hydrogen enriched hydrocarbon stream, prior to being directed into the hydro-treatment reactor. Alternatively, the hydrocarbon stream and the
10 hydrogen rich stream may be inlet into the reactor at separate inlets and mixed prior to reaching the catalyst material of the hydro-treatment reactor.

Since the removing of halides is taking place from the first vapor phase, it is possible to carry out the removal by means of a liquid. Thus, in a case where the first vapor phase
15 of the first hydrogenated product stream comprises hydrogen (H_2) and hydrogen chloride (HCl), it is possible to remove hydrogen chloride continuously from the first vapor phase.

The liquid for removing halides may be added continuously to a unit for this removal.
20 Therefore, such a unit for removing halides from the first vapor phase will be able to operate continuously without the need for stopping the process in order to regenerate or replace or change material within the unit.

In the process for removing halides of the invention, organic halides are converted into inorganic halides by steps (a) to (c) of the process of the invention. Up to 80-90% of the
25 organic halides, or even more, may be converted to inorganic halides by the process of the invention. The first hydrogenated product is separated into a first vapor phase and a first liquid phase, and typically, most of the inorganic halides are in the gas phase (in the first vapor phase), whilst the first liquid phase from the separator unit comprises
30 most of the organic halides from the hydrocarbon stream.

By the process for removing halides of the invention, the majority of the inorganic halides from the hydrocarbon stream are removed from the first vapor phase. These inorganic halides removed from the hydrocarbon stream are advantageously taken away from the system, e.g. by flushing them out. When the inorganic halides are not recycled, but are removed from the system, it is possible to remove a substantial fraction of the halides present in the hydrocarbon stream from the system. Thereby, e.g. corrosion issues from chlorides are alleviated within the system for carrying out the process of the invention.

The process of the invention may advantageously be a part of a process for refining a hydrocarbon stream.

In an embodiment the process comprises a further step of (f) recycling at least part of said second vapor phase to the hydro-treatment reactor. Hereby, any hydrocarbons and/or hydrogen present in the second vapor phase is/are reused instead of being removed from the process.

In an embodiment, step (e) of removing halides from said first vapor phase comprises the step of directing the first vapor phase to contact an alkaline solution in a scrubber unit, thereby rendering a spent alkaline solution and a said second vapor phase, where the second vapor phase is a scrubbed vapor phase. Whilst at least part of the second vapor phase is recycled back into the hydro-treatment reactor, the spent alkaline solution and thereby the halides contained therein may be removed from the process.

In an embodiment, a make-up hydrogen stream is added to the second vapor phase prior to the recycling into the hydro-treatment reactor. This is in order to ensure the required hydrogen to be present within the hydro-treatment reactor for the conversion of organic halides into inorganic halides, and possibly also further reactions, such as olefin saturation.

In an embodiment, the one or more halides comprise chloride. Since the large majority of the chloride will be in the first vapor phase, it is possible to remove it from the system by the method of the invention. This is advantageous in order to reduce the risk of corrosion within the system.

In an embodiment, the material catalytically active in converting organic halides to inorganic halides is also catalytically active in olefin saturation. In this case the hydro-treatment reactor may be a di-olefin reactor.

5 In an embodiment, the scrubber unit used in the process of the invention comprises at least two beds of packing material and the process further comprises a step of:
(g) recycling at least a part of the spent alkaline solution from the scrubber unit back into the scrubber unit. The recycling is e.g. downstream at least one bed of packing material. The term "packing material" is meant to denote material within the scrub-
10 ber unit arranged to facilitate the contacting between the first vapor phase from the hydrogenated product stream and the alkaline solution within the scrubber unit. The packing material may for example be a high grade steel packing.

The beds of packing material may comprise solid material, for example in random
15 packing or in structured packing. Typically, relatively large interstitial volume exists within the packing material.

In the case where two beds of packing material exist within the scrubber unit, the most upstream bed is typically arranged for the bulk removal of the halides, whilst the sec-
20 ond (and further, if relevant) bed(s) is(are) arranged for removal of any remaining halides. In the case of two beds of packing material, a recycling of spent alkaline solution from the scrubber unit may take place into the scrubber unit, for example at a location between the two beds. Hereby, the amount of alkaline solution to be inlet into the scrubber unit may be reduced compared to a case with no recycling of the spent alka-
25 line solution.

In an embodiment, the alkaline solution is a NaOH solution.

30 In an embodiment, the material catalytically active in converting organic halides into inorganic halides comprises: (i) a group VIII metal, (ii) a group VIB metal, and (iii) a support, said support comprising one or more of the following: aluminium oxide, silicium oxide, and titanium oxide. The catalytic material could e.g. be a Nickel-Molybden catalyst on a support or a cobalt-molybdenum catalyst on a support.

According to another aspect, the invention also relates to a process for hydro-treating a hydrocarbon stream comprising the process of for removing halides from a hydrocarbon stream, followed by the step of (h) further treating the first liquid phase from said separator unit in order to provide a hydrocarbon product. Such further treatment may e.g. be hydro-treating, for example including distilling, fractionation, and/or stripping.

In an embodiment, step (h) comprises further removal of organic halides downstream of the steps (a) to (e). This further removal could e.g. be carried out by washing via an acidic solution or water.

According to another aspect, the invention relates to a system for removing halides from a hydrocarbon stream comprising one or more halides. The system comprises:

- (a) a hydro-treatment reactor comprising a material catalytically active in converting organic halides into inorganic halides, said hydro-treatment reactor comprising an inlet for inletting a hydrogen enriched hydrocarbon stream and an outlet for outletting a first product stream,
- (b) a separator unit arranged for receiving said first product stream and for separating said first product stream into a first vapor phase and a first liquid phase, and
- (c) a halide removal unit arranged for receiving said first vapor phase and for rendering a second vapor phase.

The system for removing halides from a hydrocarbon stream provides similar advantages as the process for removing halides from a hydrocarbon stream.

Throughout this text, the term “a material catalytically active in converting organic halides into inorganic halides” is meant to denote catalyst material arranged for and/or suitable for catalyzing the conversion. “Organic halides” are chemical compounds in which one or more carbon atoms are linked by covalent bonds with one or more halogen atoms (fluorine, chlorine, bromine or iodine – group 17 in current IUPAC terminology). “Inorganic halides” are chemical compounds between a halogen atom and an element or radical that is less electronegative (or more electropositive) than the halogen, to make a fluoride, chloride, bromide, iodide, or astatine compound, with the further limitation that carbon is not part of the compound.

The term "removing halides" is meant to include situations where either some of the halides present or all of the halides present is removed. The term is thus not limited to situation where a certain percentage of the halides present are removed.

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The term "letting the stream react at the presence of the catalytically active material" is meant to cover bringing the stream into contact with the catalytically active material under circumstances relevant for catalysis to take place. Such circumstances typically relates to temperature, pressure and stream composition.

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BRIEF DESCRIPTION OF THE FIGURE

Figure 1 discloses a system for treating a hydrocarbon stream.

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DETAILED DESCRIPTION OF THE FIGURE

Figure 1 discloses a system for treating hydrocarbons. Even though some heat exchange units, pumps and compressors are shown in figure 1, further pumps, heaters, valves and other process equipment may be part of the system of figure 1.

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The system of figure 1 comprises a sub-system for removing halides from a hydrocarbon stream before the hydrocarbon stream enters a stripper and/or fractionation section. This sub-system is shown in figure 1 as the sub-system delimited by the dotted line. However, unit 16 is not necessarily part of the sub-system. In the example shown in figure 1, the hydrocarbon stream comprises one or more halides, e.g. chloride, and also di-olefins.

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Figure 1 shows a hydrocarbon stream 100 containing chlorine which is routed to a hydro-treatment reactor 10; in figure 1 the hydro-treatment reactor is a di-olefin reactor 10 for saturating di-olefins and for hydro-dechlorination. Prior to inputting the hydrocarbon stream 100 into the di-olefin reactor 10, the hydrocarbon stream 100 is combined with a hydrogen rich stream 108 to a hydrogen enriched hydrocarbon stream 101 in order to ensure the provision of the required hydrogen for the reaction in the di-olefin reactor 10. As an example only, the di-olefin reactor 10 operating conditions are a pressure of

about 30 Barg and a temperature of about 170°C. The di-olefin reactor 10 comprises a material catalytically active in olefin saturation and hydro-dechlorination. Within the di-olefin reactor 10, the hydrogen enriched hydrocarbon stream 101 reacts at the presence of the catalytically active material, rendering a first hydrogenated product stream 102, also denoted a reactor effluent stream 102.

The first hydrogenated product stream or reactor effluent stream 102 is cooled at a cooling device 11, rendering cooled first hydrogenated product stream 103. The cooling device 11 is for example as fin-fan air cooler. Subsequently, the cooled first hydrogenated product stream 103 is routed to a separator unit 12 arranged to separate the first hydrogenated product stream into a first vapor phase 104 and a first liquid phase 119. The first vapor phase 104 mainly comprises HCl and H₂ and is routed to a caustic scrubber unit 13 in order to remove HCl. The gas 105, viz. the second vapor phase, exiting the caustic scrubber unit 13 is combined with makeup hydrogen gas 106 to a gas stream 107. The gas stream 107 is compressed in a recycle gas compressor 14 to the compressed hydrogen rich stream 108. The compressed hydrogen rich stream 108 is recycled back into the di-olefin reactor 10 subsequent to addition of hydrocarbon feed 100.

A NaOH solution is inlet into the scrubber unit 13 as indicated by the arrow denoted "NaOH". In the figure, the scrubber unit 13 is shown as comprising two beds of packing material (shown by the boxes with X). The invention is not limited to a scrubber unit with two beds of packing material, in that any appropriate number of beds of packing material is conceivable. However, in the example shown, the NaOH solution is inlet downstream of the alkaline bed(s) as seen from the first vapor phase 104. A compressor or pump 13a recycles at least a part of the spent alkaline solution from the scrubber unit 13 back into the scrubber unit downstream at least one bed of packing material. The remainder of the spent alkaline solution from the scrubber unit 13 may be discharged.

The units 10, 11, 12, 13, 13a and 14 described above are the units of the system for removing halides from a hydrocarbon stream according to the invention.

The first liquid phase 119 of the hydrocarbon stream from the separator unit 12 is led to a reactor charge heater 17 by means of a pump 16 before entering a hydro-treater reactor 18 for conversion of remaining chlorine and for sulfur removal from the first liquid phase of the hydrocarbon stream.

5

As an example only, the operating conditions of the hydro-treater reactor 18 are for example a pressure of about 90 Barg and a temperature of about 370°C. To maintain operating temperature in the hydro-treater reactor 18, the liquid phase 119 of the hydrocarbon stream is routed to hydro-treater reactor through a hydro-treater reactor feed effluent heat exchanger 19 and the reactor charge heater 17.

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A reactor effluent stream 122 outlet from the hydro-treater reactor 18 is routed to the hydro-treater reactor feed effluent heat exchanger 19 in order to cool the reactor effluent 122. The reactor effluent stream 122 is then combined with wash water and subsequently provided to a cooling unit 19', such as an air cooler, and led to high pressure separator 20 to separate the liquid and gaseous portions of the hydrocarbon stream. The wash water injected upstream the cooling unit 19' is injected to avoid salt formation in the cooling unit 19', such as an air cooler.

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The gaseous portion 123 of the hydrocarbon stream exits the separator and is provided to a recycle gas knock out drum 21 for removal of impurities. The resulting recycle gas 124 is then routed to recycle gas compressor 22. The recycle gas compressor discharge is combined with makeup gas stream from makeup gas compressor 40 for recycling to a point upstream the hydro-treater reactor feed effluent heat exchanger 19.

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The liquid portion 130 of the hydrocarbon stream exits the high pressure separator 20 and enters a stripper 30 to further separate liquid and gaseous components. One part of the output fluid from the stripper is cooled in cooler 31 and directed to a further separator 32 arranged to separate gas from liquid. The liquid part from the further separator 32 is recycled to the stripper, whilst the gaseous part is removed as off gas 140. Liquid hydrocarbon 150 from the stripper is routed to fractionator (not shown in fig. 1). Even though fig. 1 does not show a steam input to the stripper, it is implicit that the stripper would operate by addition of steam.

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A sour water stream (SWS) 160 exits the high pressure separator 20 and is routed to a SWS unit (not shown in figure 1) for further processing.

CLAIMS

1. A process for removing halides from a hydrocarbon stream comprising one or more halides, said process comprising the steps of:
- 5 (a) providing said hydrocarbon stream to a hydrogen enriched stream to a hydro-treatment reactor comprising a material catalytically active in converting organic halides into inorganic halides,
- (b) providing a hydrogen rich stream to said hydro-treatment reactor,
- (c) letting the hydrocarbon stream and the hydrogen rich stream react at the pres-
- 10 ence of the catalytically active material, rendering a first hydrogenated product stream,
- (d) separating said first hydrogenated product stream into a first vapor phase and a first liquid phase in a separator unit, and
- (e) removing halides from said first vapor phase to create a second vapor phase.
- 15 2. A process according to claim 1, further comprising the step of:
- (f) recycling at least part of said second vapor phase to the hydro-treatment reactor.
3. A process according to claim 1 or 2, wherein step (e) of removing halides from said
- 20 first vapor phase comprises directing said first vapor phase to contact an alkaline solution in a scrubber unit, thereby rendering a spent alkaline solution and a said second vapor phase, said second vapor phase being a scrubbed vapor phase.
4. A process according to any of the claims 1 to 3, wherein a make-up hydrogen
- 25 stream is added to the second vapor phase prior to the recycling into the hydro-treatment reactor.
5. A process according to any of the claims 1 to 4, wherein the one or more halides comprise chloride.
- 30 6. A process according to any of the claims 1 to 5, wherein the material catalytically active in converting organic halides into inorganic halides is also catalytically active in olefin saturation.

7. A process according to any of the claims 3 to 6, wherein said scrubber unit comprises at least two beds of packing material and wherein the process further comprises a step of:

(g) recycling at least a part of the spent alkaline solution from the scrubber unit back into the scrubber unit.

8. A process according to any of the claims 3 to 7, wherein said alkaline solution is a NaOH solution.

9. A process according to any of the claims 1 to 8, wherein the material catalytically active in converting organic halides into inorganic halides comprises: (i) a group VIII metal, (ii) a group VIB metal, and (iii) a support, said support comprising one or more of the following: aluminium oxide, silicium oxide, and titanium oxide.

10. A process for hydro-treating a hydrocarbon stream comprising the process of any of the claims 1 to 9, followed by the step of:

(h) further treating the first liquid phase from said separator unit in order to provide a hydrocarbon product.

11. A process according to claim 10, wherein step (h) comprises further removal of organic halides downstream of the steps (a) to (e).

12. A system for removing halides from a hydrocarbon stream comprising one or more halides, said system comprising:

(a) a hydro-treatment reactor comprising a material catalytically active in converting organic halides into inorganic halides, said hydro-treatment reactor comprising an inlet for inletting a hydrogen enriched hydrocarbon stream and an outlet for outletting a first product stream,

(b) a separator unit arranged for receiving said first product stream and for separating said first product stream into a first vapor phase and a first liquid phase, and

(c) a halide removal unit arranged for receiving said first vapor phase and for rendering a second vapor phase.

13. A system according to claim 12, further comprising:

(d) means for recycling at least part of said second vapor phase to said hydro-treatment reactor, where said means for recycling comprise piping and a compressor.

5 14. A system according to any of the claims 12 or 13, wherein said halide removal unit is a scrubber unit with one or more beds of packing material.

15 15. A system according to claim 14, further comprising a pump for recycling at least a part of the spent alkaline solution from the scrubber unit back into the scrubber unit downstream at least one bed of packing material.

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16. A system according to any of the claims 12 to 15, said system further means for inletting a water or a NaOH solution into the scrubber unit, downstream of one or more beds.

15 17. A system according to any of the claims 12 to 16, wherein said hydro-treatment reactor is a di-olefin reactor, and wherein the catalytically active material is also catalytically active in di-olefin saturation.

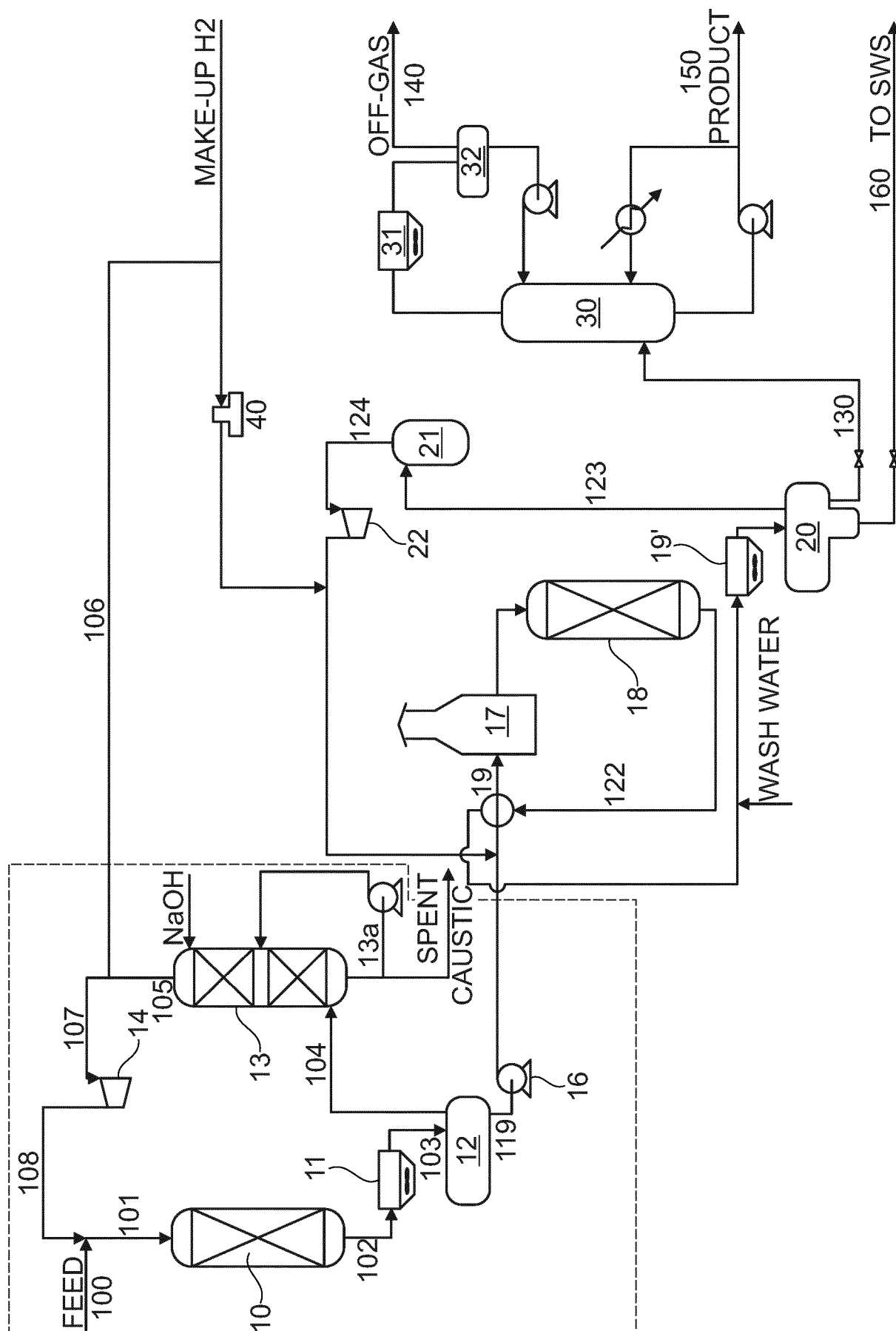


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. C10G45/02 C07C1/26 C07C1/30 C10G19/02
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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)
C10G C07C

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

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 806 233 A (JAMES JR ROBERT B [US] ET AL) 21 February 1989 (1989-02-21) column 3, lines 2-12,45-47,60-65; figure 1 column 4, line 17 - line 62 column 5, lines 32-36,55-61 column 6, lines 5-8,17-42 -----	1-17
X	US 2014/037512 A1 (TIMKEN HYE KYUNG CHO [US] ET AL) 6 February 2014 (2014-02-06) paragraphs [0025], [0029], [0030], [0033], [0048]; figure 2 -----	12-17
A	US 2010/200458 A1 (KALNES TOM N [US]) 12 August 2010 (2010-08-12) paragraphs [0006], [0010], [0014], [0021], [0027], [0041] - [0043]; figure 1 ----- -/--	1-17



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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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 892 818 A (SCHARFE GERHARD ET AL) 1 July 1975 (1975-07-01) column 1, line 5 - line 6 column 3, line 63 - line 66 -----	1-17

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2016/052224

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