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(54) Title: METHOD FOR REMOVING RESIDUES BY MEANS OF A STRIPPING DEVICE

(57) Abstract: The present invention provides method for removing residues from a first process stream comprising an organic solvent, water, a derivative of the organic solvent and the residues, comprising the steps of: i) feeding the first process stream to a solvent stripper comprising a still pot and a stripper column; ii) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and iii) removing a residue stream from the still pot; characterised in that: the still pot and the stripper column form an integrated unit and the stripper column comprises a plurality of sieve trays. The present invention further provides an apparatus for carrying out the method, and processes for the production of a purified aromatic dicarboxylic acid incorporating the method.



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METHOD FOR REMOVING RESIDUES BY MEANS OF A STRIPPING DEVICE

TECHNICAL FIELD

The present invention relates to a process and apparatus for the production of an aromatic dicarboxylic acid.

BACKGROUND ART

Aromatic dicarboxylic acids are commonly manufactured by the catalytic oxidation of a hydrocarbon precursor in an organic solvent. An example is terephthalic acid (TA), which is widely used in the manufacture of polyesters, such as poly(ethylene terephthalate) (PET). The TA required as a reactant for PET production is known as "purified terephthalic acid" (PTA) and generally contains over 99.97 wt%, preferably over 99.99 wt%, of terephthalic acid, and less than 25 ppm 4-carboxybenzaldehyde (4-CBA). On the commercial scale, PTA suitable for use in PET production is generally prepared in a two-stage process. First, p-xylene is oxidized (e.g. using air) in the presence of a metal catalyst (e.g. a cobalt and/or manganese salt or compound) to provide "crude terephthalic acid" (CTA), as described in, for example, US 2,833,816. Second, the CTA produced by this oxidation reaction is then purified, as it is typically contaminated by impurities such as 4-CBA, p-toluic acid, and various coloured impurities that impart a yellowish colour to the TA. Purification of the CTA typically requires at least one chemical transformation (e.g. hydrogenation) in addition to at least one physical procedure (e.g. crystallization, washing, etc.) to yield PTA.

PTA is generally considered to be a commodity item, with several million tonnes being produced annually, and it is therefore desirable for manufacturers to reduce their costs to maximise the economy and efficiency of PTA production. This can be achieved both by reducing capital costs (e.g. equipment costs) and variable costs (e.g. costs associated with waste disposal, use of starting materials, organic solvent, heating fuel and demineralised water).

It is therefore desirable to minimise the consumption of organic solvent (e.g. acetic acid) by designing the manufacturing process to recover and recycle the organic solvent. Also, as derivatives of the organic solvent such as methyl acetate accumulate to a steady state level within the oxidation reaction, the consumption of organic solvent by its conversion to these derivatives can be minimised by designing the process to also recover these derivatives and recycle them to the oxidation reaction. Accordingly, the process may be designed to recover organic solvent, use it on scrubbing duties to recover its derivatives from effluent gas streams, and subsequently recycle it to the oxidation reaction to achieve both of the above aims. However, organic solvent from some streams in the manufacturing process (e.g. the mother liquor from which CTA is separated prior to its purification) contains residues that it is desirable to remove before the organic solvent can be used on scrubbing duties. It may be desirable to remove these residues because they are corrosive (e.g. hydrogen

bromide) and/or because they comprise valuable components (e.g. benzoic acid) that can be recovered and used elsewhere.

It is an object of the present invention to provide a more economic and efficient process and apparatus for the manufacture of aromatic dicarboxylic acids. Further objects will be apparent from the description below.

DISCLOSURE OF THE INVENTION

A first aspect of the present invention provides a method for removing residues from a first process stream comprising an organic solvent, water, a derivative of the organic solvent and the residues, comprising the steps of:

- i) feeding the first process stream to a solvent stripper comprising a still pot and a stripper column;
- ii) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
- iii) removing a residue stream from the still pot;

characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column comprises a plurality of sieve trays.

The first aspect of the invention further provides a solvent stripper comprising:

a still pot comprising:

a first residue stream outlet; and
a vent for removing an overhead vapour,

a stripper column comprising:

a first inlet for receiving the overhead vapour; and
a vent for removing a vapour stream,

wherein the solvent stripper further comprises a first process stream inlet and is characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column further comprises a plurality of sieve trays.

The first aspect of the invention further provides a process for the production of a purified aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in an organic solvent, comprising the steps of:

- I) oxidising the hydrocarbon precursor in the organic solvent in the presence of a metal catalyst to provide a crude aromatic dicarboxylic acid; and

II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid, wherein the process further comprises the steps of:

III) feeding a first process stream comprising the organic solvent, water, a derivative of the organic solvent and residues from the process for the production of a purified aromatic dicarboxylic acid to a solvent stripper comprising a still pot and a stripper column;

IV) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and

V) removing a residue stream from the still pot;

characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column comprises a plurality of sieve trays.

In the first aspect of the invention, the still pot and the stripper column form an integrated unit, i.e. they are not discrete units separated by connecting pipework but instead are joined directly to one another. This configuration removes the need for any pipework connecting the still pot and the stripper column and thus reduces the capital cost of the manufacturing plant. The inventors have surprisingly found that the use of a stripper column that comprises a plurality of sieve trays increases the efficacy of hydrogen bromide removal relative to previous configurations, such as a stripper column comprising a plurality of baffle trays, such that the level of hydrogen bromide in the vapour stream removed from the stripper column is reduced. A first portion of the residue stream may be heated in a residue stream reboiler circuit with medium-pressure steam and then returned to the still pot via a first residue stream inlet in the still pot. As used herein, "medium-pressure steam" refers to saturated steam at a pressure of about 5-10 barA, or about 6-9 barA, or about 8 barA. The medium-pressure steam may be derived by pressure let-down of a higher-pressure steam (e.g. an "intermediate-pressure steam", which refers to a saturated steam at a pressure of about 10-50 barA, or about 12.5-40 barA, or about 15-35 barA, or about 17.5-30 barA, or about 20 barA, or a "high-pressure steam", which refers to a saturated steam at a pressure of about 50-150 barA, or about 75-125 barA, or about 100 barA), and/or by flash evaporation of a higher-pressure and higher-temperature condensate, and/or may be raised within a process for the production of an aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in the organic solvent, e.g. by transferring heat from the process to a water stream).

A second aspect of the invention provides a method for removing residues from a first process stream comprising an organic solvent, water, a derivative of the organic solvent and the residues, comprising the steps of:

i) feeding the first process stream to a solvent stripper comprising a still pot and a stripper column;

- ii) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
 - iii) removing a residue stream from the still pot;
- characterised in that the process further comprises the steps of:
- iv) heating a first portion of the residue stream with medium-pressure steam; and
 - v) returning the first portion of the residue stream to the still pot.

The second aspect of the invention further provides a solvent stripper comprising:

a still pot comprising:

- a first residue stream outlet; and
- a vent for removing an overhead vapour,

a stripper column comprising:

- a first inlet for receiving the overhead vapour; and
- a vent for removing a vapour stream,

wherein the solvent stripper further comprises a first process stream inlet and is characterised in that:

the solvent stripper further comprises a residue stream reboiler circuit that is configured to heat a first portion of a residue stream removed from the first residue stream outlet using a source of medium-pressure steam and to return the first portion of the residue stream to the still pot via a first residue stream inlet in the still pot.

The second aspect of the invention further provides a process for the production of a purified aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in an organic solvent, comprising the steps of:

- I) oxidising the hydrocarbon precursor in the organic solvent in the presence of a metal catalyst to provide a crude aromatic dicarboxylic acid; and
 - II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid,
- wherein the process further comprises the steps of:
- III) feeding a first process stream comprising the organic solvent, water, a derivative of the organic solvent and residues from the process for the production of a purified aromatic dicarboxylic acid to a solvent stripper comprising a still pot and a stripper column;
 - IV) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
 - V) removing a residue stream from the still pot;
- characterised in that the process further comprises the steps of:
- VI) heating a first portion of the residue stream with medium-pressure steam; and
 - VII) returning the first portion of the residue stream to the still pot.

The heated first portion of the residue stream is therefore able to provide the heat necessary to evaporate any organic solvent, water, and derivative of the organic solvent that are present in the still pot. The use of medium-pressure steam, rather than low-pressure steam (i.e. saturated steam at a pressure of less than about 5 barA) as used in previous configurations, on the reboil circuit allows the use of a smaller heat exchanger for transferring heat from the steam to the first portion of the residue stream, thus saving on capital costs, and a reduced rate of circulation in the reboil circuit, thus allowing a lower-capacity circulation pump to be used and reducing both the power consumption and the cost of the circulation pump. The stripper column may comprise a plurality of sieve trays.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic of a process and apparatus according to the first aspect of the invention.

Figure 2 is a schematic of a process and apparatus according to the second aspect of the invention.

DETAILED DESCRIPTION OF THE INVENTION

Various embodiments of the invention are described herein. It will be recognised that features specified in each embodiment may be combined with other specified features to provide further embodiments.

It will be appreciated that the general operation of a process and apparatus for the production of an aromatic dicarboxylic acid by the catalytic oxidation of a hydrocarbon precursor in an organic solvent is well known. For example, as discussed above, terephthalic acid suitable for use in PET production (i.e. purified terephthalic acid) is generally prepared in a two-stage process. First, p-xylene is oxidized (e.g. using air) in the presence of a metal catalyst (e.g. a cobalt and/or manganese salt or compound) to provide crude terephthalic acid. Second, the crude terephthalic acid produced by this oxidation reaction is then purified to remove impurities, such as 4-CBA and p-toluic acid, to yield purified terephthalic acid. Purification of crude terephthalic acid typically requires at least one chemical transformation (e.g. hydrogenation) in addition to at least one physical procedure (e.g. crystallization, washing, etc.).

Production of an aromatic dicarboxylic acid

The aromatic dicarboxylic acid produced in the present invention is preferably selected from terephthalic acid, phthalic acid and isophthalic acid. The aromatic dicarboxylic acid is preferably terephthalic acid. Accordingly, the residues typically comprise isophthalic acid, orthophthalic acid, p-toluic acid, benzoic acid, 4-carboxybenzaldehyde, bromides (e.g. hydrogen bromide), catalyst components or a mixture of two or more of these components. The residues may further comprise the aromatic dicarboxylic acid itself. The hydrocarbon precursor is a compound that may be oxidised

to form the aromatic dicarboxylic acid. Thus, the hydrocarbon precursor is typically benzene or naphthalene substituted with groups such as C₁₋₆alkyl, formyl, or acetyl in the positions of the carboxylic acid substituents in the desired end product. Preferred hydrocarbon precursors are C₁₋₆alkyl-substituted benzene, in particular p-xylene. The organic solvent is typically an aliphatic carboxylic acid, such as acetic acid, or a mixture of such aliphatic carboxylic acid(s) and water. The derivative of the organic solvent is a compound that is formed from the organic solvent as a by-product of the oxidation reaction. For instance, when the organic solvent is acetic acid, the derivative of the organic solvent may be methyl acetate. The oxidation reaction may be carried out under any conditions wherein oxygen is available, e.g. the reaction can be carried out in air. The reaction catalyst typically comprises soluble forms of cobalt and/or manganese (e.g. their acetates), with a source of bromine, such as hydrogen bromide, used as a promoter. The temperature of the oxidation reaction is typically in the range of about 100-250 °C, preferably about 150-220 °C. Any conventional pressure may be used for the reaction, suitably to maintain the reaction mixture in a liquid state.

An oxidation stage performs the function of catalytically oxidizing the hydrocarbon precursor in the organic solvent, thus forming a product stream and a vent gas. The product stream is typically transferred to a crystallisation stage to form a first slurry of crude aromatic dicarboxylic acid crystals and an overhead vapour. The first slurry of crude aromatic dicarboxylic acid crystals is typically passed to a separation stage in which a mother liquor is separated from the crude aromatic dicarboxylic acid crystals, which may then be mixed with an aqueous liquid to form a second slurry of crude aromatic dicarboxylic acid crystals. This second slurry of crude aromatic dicarboxylic acid crystals is typically transferred to a purification plant, heated and subjected to hydrogenation, before being cooled to form a slurry of purified aromatic dicarboxylic acid crystals.

The vent gas from the oxidation stage is typically separated in a distillation stage into an organic solvent-rich liquid stream and a water-rich vapour stream. The organic solvent-rich liquid stream from the distillation stage typically comprises 80-95 % w/w organic solvent and is typically returned to the oxidation stage. The water-rich vapour stream from the distillation stage typically comprises 0.1-5.0 % w/w organic solvent and is typically condensed to form a condensate stream and an overhead gas in a condensing stage. A portion of the condensate stream is typically used as a source of the aqueous liquid used to form the second slurry of crude aromatic dicarboxylic acid crystals mentioned above. A portion of the condensate stream also typically forms a source of wash fluid for the purified aromatic dicarboxylic acid crystals from the purification plant.

Solvent stripper

The solvent stripper comprises a still pot and a stripper column. The solvent stripper further comprises a first process stream inlet for receiving the first process stream. This inlet is typically

located in the still pot so that the first process stream is fed to the still pot, although it may be located in the stripper column. The still pot comprises a first residue stream outlet for removing a residue stream. This outlet is typically located in the base or sump of the still pot and is below the level of the liquid in the still pot when in use. The still pot also comprises a vent for removing an overhead vapour to a first inlet in the stripper column. In the first aspect of the invention, the still pot and the stripper column form an integrated unit, in which case the vent of the still pot is joined directly to the first inlet in the stripper column without any intermediate pipework. It is also preferred in the second aspect of the invention that the still pot and the stripper column form an integrated unit. The stripper column typically has a smaller diameter than the still pot. The stripper column also comprises a vent for removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent. The stripper column is typically a distillation column comprising at least one theoretical separation stage, which can be provided by trays, such as sieve, valve or bubble cap trays, structured packing or other suitable structures that provide surfaces for mass transfer between gaseous and liquid phases within the column. In the first aspect of the invention, the stripper column comprises a plurality of sieve trays, which provide an unexpected improvement in the reduction in the level of hydrogen bromide in the vapour stream removed from the stripper column. It is also preferred in the second aspect of the invention that the stripper column comprises a plurality of sieve trays.

In the second aspect of the invention, the solvent stripper further comprises a residue stream reboiler circuit that is configured to heat a first portion of a residue stream removed from the first residue stream outlet and to return the first portion of the residue stream to the still pot via a first residue stream inlet in the still pot. It is also preferred in the first aspect of the invention that the solvent stripper further comprises a residue stream reboiler circuit that is configured to heat a first portion of a residue stream removed from the first residue stream outlet and to return the first portion of the residue stream to the still pot via a first residue stream inlet in the still pot. The first residue stream inlet is preferably located in a region of the still pot above the level of the liquid in the still pot when in use. Accordingly, the first portion of the residue stream may undergo flash evaporation in the still pot on its return, i.e. at least part of the stream may evaporate and become part of the overhead vapour that passes from the still pot to the stripper column. Accordingly, the reboiler circuit increases the concentration of residues in the liquid in the still pot and thus in the residue stream, i.e. the concentration of the organic solvent, water, and the derivative of the organic solvent in this liquid is reduced. Heating is achieved in the second aspect of the invention (and preferably also in the first aspect of the invention) using a source of medium-pressure steam, from which heat is typically transferred to the first portion of the residue stream using a heat exchanger (e.g. a shell and tube heat exchanger). As mentioned above, the medium-pressure steam may be derived by pressure let-down of a higher-pressure steam, and/or flash evaporation of a higher-pressure and higher-

temperature condensate. Alternatively, the medium-pressure steam may be raised within a process for the production of an aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in the organic solvent, e.g. by transferring heat generated by the process to a water stream. The medium-pressure condensate generated from the medium-pressure steam in heating the residue stream is typically fed to a lower-pressure steam recovery system for further use.

The still pot may further comprise a residue stream recirculation circuit that is configured to return a second portion of the residue stream under pressure (e.g. by using a pump) to the still pot via a second residue stream inlet. The second residue stream inlet is typically located in the base or sump of the still pot and is preferably below the level of the liquid in the still pot when in use. The recirculation of the residue stream is thus able to agitate the liquid in the still pot such that a mechanical agitator is not required.

The solvent stripper may further comprise one or more additional process stream inlets to receive one or more further process streams. For instance, the solvent stripper may comprise a second process stream inlet located in the still pot. This inlet may receive a second process stream from an atmospheric scrubber for removing the derivative of the organic solvent from a vapour stream generated elsewhere in the process. Accordingly, the second process stream may be a scrubbed liquid stream comprising water and the organic solvent from which the derivative of the organic solvent has been removed in the atmospheric scrubber. Alternatively or additionally, the solvent stripper may comprise a third process stream inlet located in the stripper column, for instance at the top of the stripper column. This inlet may receive a third process stream, which may comprise the organic solvent, water, and the derivative of the organic solvent. This third process stream may serve as a scrubbing fluid for the overhead vapour removed from the still pot.

First process stream

The first process stream comprises an organic solvent, water, a derivative of the organic solvent and residues. The first process stream is preferably a stream from a process for the production of an aromatic dicarboxylic acid (e.g. TA) comprising the catalytic oxidation of a hydrocarbon precursor in the organic solvent. In particular, the first process stream is preferably a mother liquor from which aromatic dicarboxylic acid crystals have been separated. More specifically, the first process stream is preferably a mother liquor from which crude aromatic dicarboxylic acid crystals have been separated (i.e. the mother liquor is derived from the “first slurry” described above).

Further treatment

A third portion of the residue stream may be fed to a residue evaporator to vaporise at least a portion of any water, organic solvent, and/or derivative of the organic solvent that is remaining in the residue

stream. The resultant molten residue stream may then be quenched and processed to isolate and recover its various components as described in co-pending application GB 1414292.1, which is incorporated herein by reference in its entirety. The vapour stream from the residue evaporator typically comprises the organic solvent and water and may be returned from the residue evaporator to the solvent stripper, typically to the still pot.

The vapour stream comprising the organic solvent, water and the derivative of the organic solvent that is removed from the stripper column may be transferred to a stripping device for recovering the derivative of the organic solvent from the vapour stream, thus providing a purified organic solvent stream in which the mass concentration of the derivative of the organic solvent is decreased relative to the mass concentration of the derivative of the organic solvent in the vapour stream.

The invention will be further described with reference to the figures.

Figure 1 is a schematic of a process and apparatus according to a preferred embodiment of the first aspect of the present invention. First process stream 10a, which comprises an organic solvent (preferably acetic acid), water, a derivative of the organic solvent (preferably methyl acetate) and residues (preferably a mixture of isophthalic acid, orthophthalic acid, p-toluic acid, benzoic acid, 4-carboxybenzaldehyde, bromides, and catalyst components) is fed to solvent stripper 10. Solvent stripper 10 is made up of still pot 20 and stripper column 30, which form an integrated unit. The overhead vapour removed from still pot 20 is scrubbed in stripper column 30 with stream 10b, which comprises the organic solvent, water and the derivative of the organic solvent. Vapour stream 10c, which comprises the organic solvent, water, and the derivative of the organic solvent, is removed from stripper column 30.

Residue stream 10d is removed from still pot 20. A first portion of residue stream 10d is passed to first pump 40 and residue stream 40a is fed to heat exchanger 60, which is supplied with medium-pressure steam feed 60a at a pressure of about 8 barA. Condensate stream 60b is removed from heat exchanger 60 and fed to a lower-pressure steam recovery system for further use. Heated residue stream 60c is fed to still pot 20 via an inlet above the level of the liquid in still pot 20. Residue stream 40b is fed to residue evaporator 70. Stream 70a, which comprises the organic solvent and water, is fed to still pot 20 via an inlet above the level of the liquid in still pot 20. Molten residue stream 70b is removed from residue evaporator 70 for further processing. A second portion of residue stream 10d is passed to second pump 50. Residue stream 50a is returned to still pot 20 via an inlet below the level of the liquid in still pot 20.

Figure 2 is a schematic of a process and apparatus according to a preferred embodiment of the second aspect of the present invention. First process stream 110a, which comprises an organic solvent (preferably acetic acid), water, a derivative of the organic solvent (preferably methyl acetate) and residues (preferably a mixture of isophthalic acid, orthophthalic acid, p-toluic acid, benzoic acid, 4-carboxybenzaldehyde, bromides, and catalyst components) is fed to solvent stripper 110. Solvent stripper 110 is made up of still pot 120 and stripper column 130. Overhead vapour stream 120a is removed from still pot 120 via a vent and fed to an inlet in stripper column 130, where it is scrubbed with stream 110b, which comprises the organic solvent, water and the derivative of the organic solvent. Liquid stream 130b is removed from stripper column 130 and fed to an inlet in still pot 120 (although drawn separately, streams 120a and 120b may be carried by a single line or pipe). Vapour stream 110c, which comprises the organic solvent, water, and the derivative of the organic solvent, is removed from stripper column 130.

Residue stream 110d is removed from still pot 120. A first portion of residue stream 110d is passed to first pump 140 and residue stream 140a is fed to heat exchanger 160, which is supplied with medium-pressure steam feed 160a at a pressure of about 8 barA. Condensate stream 160b is removed from heat exchanger 160 and fed to a lower-pressure steam recovery system for further use. Heated residue stream 160c is fed to still pot 120 via an inlet above the level of the liquid in still pot 120. Residue stream 140b is fed to residue evaporator 170. Stream 170a, which comprises the organic solvent and water, is fed to still pot 120 via an inlet above the level of the liquid in still pot 120. Molten residue stream 170b is removed from residue evaporator 170 for further processing. A second portion of residue stream 110d is passed to second pump 150. Residue stream 150a is returned to still pot 120 via an inlet below the level of the liquid in still pot 120.

CLAIMS

1. A method for removing residues from a first process stream comprising an organic solvent, water, a derivative of the organic solvent and the residues, comprising the steps of:
 - i) feeding the first process stream to a solvent stripper comprising a still pot and a stripper column;
 - ii) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
 - iii) removing a residue stream from the still pot;characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column comprises a plurality of sieve trays.
2. The method of claim 1, further comprising the steps of:
 - iv) heating a first portion of the residue stream with medium-pressure steam; and
 - v) returning the first portion of the residue stream to the still pot.
3. A method for removing residues from a first process stream comprising an organic solvent, water, a derivative of the organic solvent and the residues, comprising the steps of:
 - i) feeding the first process stream to a solvent stripper comprising a still pot and a stripper column;
 - ii) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
 - iii) removing a residue stream from the still pot;characterised in that the process further comprises the steps of:
 - iv) heating a first portion of the residue stream with medium-pressure steam; and
 - v) returning the first portion of the residue stream to the still pot.
4. The method of claim 3, wherein the stripper column comprises a plurality of sieve trays.
5. The method of any one of claims 2-4, wherein the first portion of the residue stream undergoes flash evaporation in the still pot in step v).
6. The method of any one of claims 2-5, wherein the medium-pressure steam is derived from let-down of a higher-pressure steam, and/or flash of a higher-pressure and higher-temperature condensate and/or is raised within a process for the production of an aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in the organic solvent.

7. The method of any preceding claim, wherein the first process stream is a stream from a process for the production of an aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in the organic solvent.
8. The method of claim 7, wherein the first process stream is a mother liquor from which crude aromatic dicarboxylic acid crystals have been separated.
9. The method of claim 7 or claim 8, wherein the aromatic dicarboxylic acid is terephthalic acid.
10. The method of claim 9, wherein the residues comprise isophthalic acid, orthophthalic acid, p-toluic acid, benzoic acid, 4-carboxybenzaldehyde, bromides, catalyst components or a mixture thereof.
11. The method of any preceding claim, wherein the organic solvent is acetic acid.
12. The method of claim 11, wherein the derivative of the organic solvent is methyl acetate.
13. The method of any preceding claim, wherein the first process stream is fed to the still pot in step i).
14. The method of any preceding claim, further comprising the step of:
 - vi) returning a second portion of the residue stream under pressure to the still pot via one or more inlets located below the level of the liquid in the still pot.
15. The method of any preceding claim, further comprising the steps of:
 - vii) feeding a third portion of the residue stream to a residue evaporator; and
 - viii) returning a stream comprising the organic solvent and water from the residue evaporator to the still pot.
16. The method of any preceding claim, further comprising the step of:
 - ix) feeding the vapour stream to a stripping device for stripping the derivative of the organic solvent from the organic solvent.
17. A solvent stripper comprising:
 - a still pot comprising:
 - a first residue stream outlet; and
 - a vent for removing an overhead vapour,

a stripper column comprising:

- a first inlet for receiving the overhead vapour; and
- a vent for removing a vapour stream,

wherein the solvent stripper further comprises a first process stream inlet and is characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column further comprises a plurality of sieve trays.

18. The solvent stripper of claim 17, wherein the still pot further comprises a residue stream reboiler circuit that is configured to heat a first portion of a residue stream removed from the first residue stream outlet using a source of medium-pressure steam and to return the first portion of the residue stream to the still pot via a first residue stream inlet.

19. A solvent stripper comprising:

a still pot comprising:

- a first residue stream outlet; and
- a vent for removing an overhead vapour,

a stripper column comprising:

- a first inlet for receiving the overhead vapour; and
- a vent for removing a vapour stream,

wherein the solvent stripper further comprises a first process stream inlet and is characterised in that:

the solvent stripper further comprises a residue stream reboiler circuit that is configured to heat a first portion of a residue stream removed from the first residue stream outlet using a source of medium-pressure steam and to return the first portion of the residue stream to the still pot via a first residue stream inlet in the still pot.

20. The solvent stripper of claim 19, wherein the stripper column comprises a plurality of sieve trays

21. The solvent stripper of any one of claims 17-20, wherein the solvent stripper further comprises a residue stream recirculation circuit that is configured to return a second portion of the residue stream to the still pot via a second residue stream inlet in the still pot.

22. A process for the production of a purified aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in an organic solvent, comprising the steps of:

- I) oxidising the hydrocarbon precursor in the organic solvent in the presence of a metal catalyst to provide a crude aromatic dicarboxylic acid; and
- II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid, wherein the process further comprises the steps of:
- III) feeding a first process stream comprising the organic solvent, water, a derivative of the organic solvent and residues from the process for the production of a purified aromatic dicarboxylic acid to a solvent stripper comprising a still pot and a stripper column;
- IV) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
- V) removing a residue stream from the still pot;

characterised in that:

the still pot and the stripper column form an integrated unit and the stripper column comprises a plurality of sieve trays.

23. A process for the production of a purified aromatic dicarboxylic acid comprising the catalytic oxidation of a hydrocarbon precursor in an organic solvent, comprising the steps of:

- I) oxidising the hydrocarbon precursor in the organic solvent in the presence of a metal catalyst to provide a crude aromatic dicarboxylic acid; and
- II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid, wherein the process further comprises the steps of:
- III) feeding a first process stream comprising the organic solvent, water, a derivative of the organic solvent and residues from the process for the production of a purified aromatic dicarboxylic acid to a solvent stripper comprising a still pot and a stripper column;
- IV) removing a vapour stream comprising the organic solvent, water and the derivative of the organic solvent from the stripper column; and
- V) removing a residue stream from the still pot;

characterised in that the process further comprises the steps of:

- VI) heating a first portion of the residue stream with medium-pressure steam; and
- VII) returning the first portion of the residue stream to the still pot.

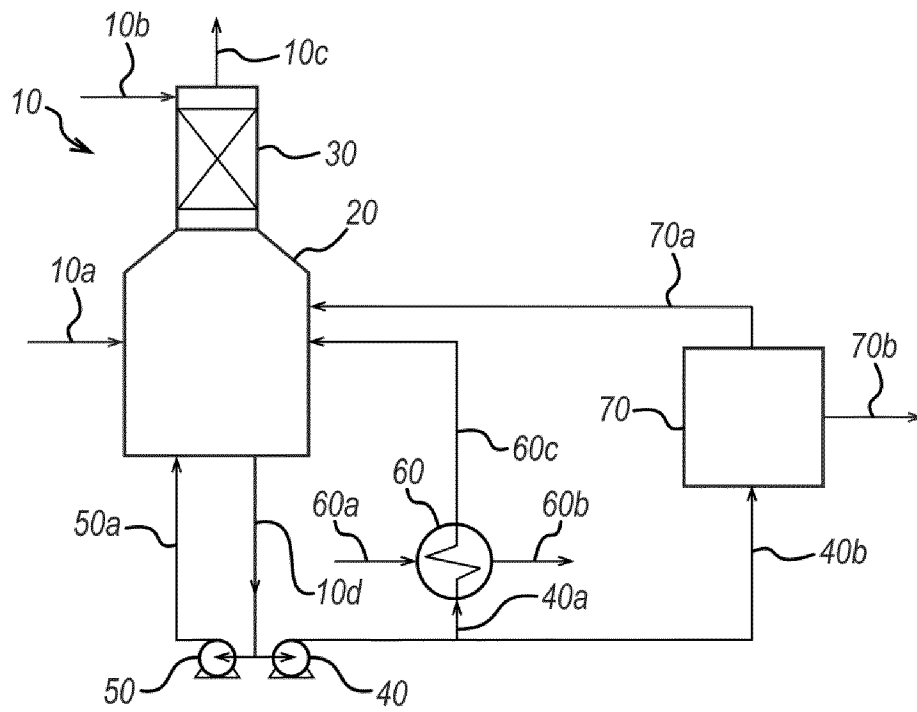
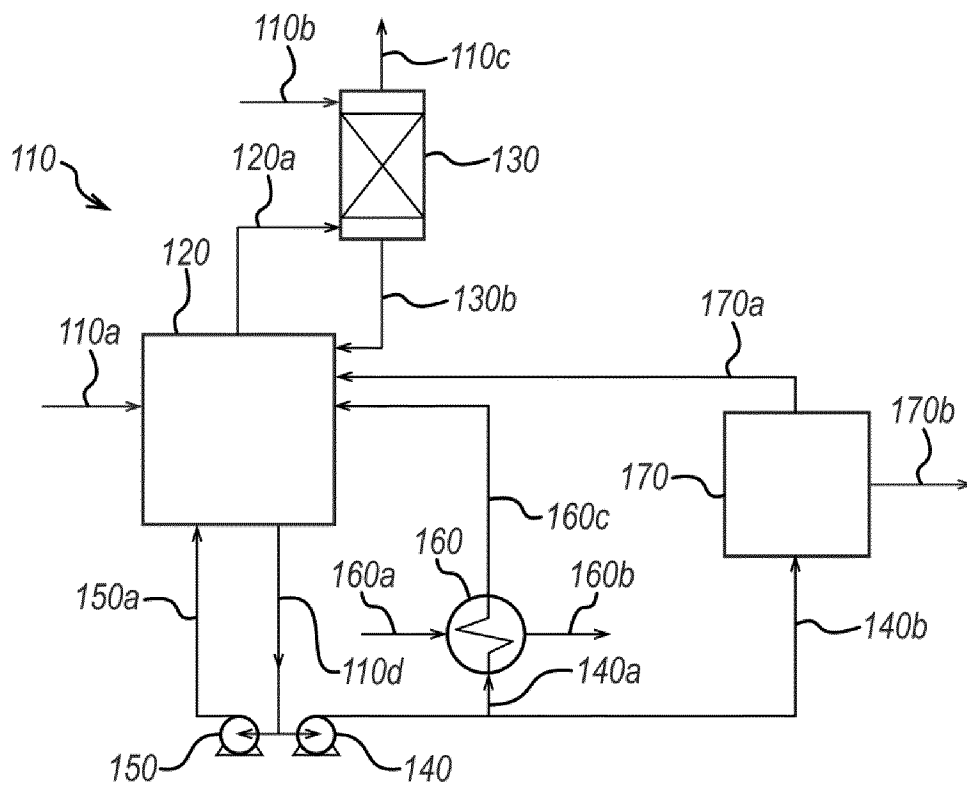
1/1
Figure 1

Figure 2



INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2015/073028

A. CLASSIFICATION OF SUBJECT MATTER INV. C07C51/16 C07C51/44 C07C51/46 C07C51/265 B01D3/14 B01D3/36 ADD. According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) C07C B01D 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 2003/150706 A1 (JANG JI-YOUNG [US] ET AL) 14 August 2003 (2003-08-14) the whole document, in particular [0008]; [0016]; [0017]; [0030]-[0032]; [0038]-[0039]; [0042]; [0047]; [0050]; [0058]; Figures; claims -----	1-23
X	CA 2 580 951 A1 (INVISTA TECH SARL [CH]) 14 August 1997 (1997-08-14) page 2, lines 5-13; page 3, lines 9-19; page 4, line 8 - page 5, line 14; page 6, lines 4-7; page 10, lines 12-23; Example; Figure 1; claims ----- -/--	1-23
☒ 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 "E" earlier application or patent but published on or after the international filing date "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 9 December 2015		Date of mailing of the international search report 11/01/2016
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Sen, Alina

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2015/073028

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Week 201377 Thomson Scientific, London, GB; AN 2013-N70622 XP002751973, -& CN 103 012 102 A (CHINA PETROCHEMICAL CO LTD) 3 April 2013 (2013-04-03) abstract; figure 1 -----	1-23
X	US 2007/027340 A1 (LEE MYRON M [KR] ET AL) 1 February 2007 (2007-02-01) [0018]-[0023]; Example 1 and Example 2; Figure 1; claims -----	1,2, 5-18,21, 22

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2015/073028

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2003150706	A1	14-08-2003	NONE
CA 2580951	A1	14-08-1997	NONE
CN 103012102	A	03-04-2013	NONE
US 2007027340	A1	01-02-2007	CN 1907940 A 07-02-2007
			KR 100733400 B1 29-06-2007
			US 2007027340 A1 01-02-2007