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- (71) Applicant: **INVISTA NORTH AMERICA S.A.R.L.**
[LU/US]; 3 Little Falls Centre, 2801 Centerville Road,
Wilmington, Delaware 19808 (US).
- (72) Inventors: **DE FEO, Demain Mauro**; INVISTA Performance
Technologies, The Wilton Centre, Redcar, Wilton
Cleveland TS10 4RF (GB). **HUGHES, Robert**; INVISTA
Performance Technologies, The Wilton Centre, Redcar,
Wilton Cleveland TS10 4RF (GB). **HOUSLEY, Samuel
Duncan**; INVISTA Performance Technologies, The Wilton

Centre, Redcar, Wilton Cleveland TS10 4RF (GB). **MC-DONNELL, Finbar Gerald**; INVISTA Performance
Technologies, The Wilton Centre, Redcar, Wilton Cleve-
land TS10 4RF (GB).

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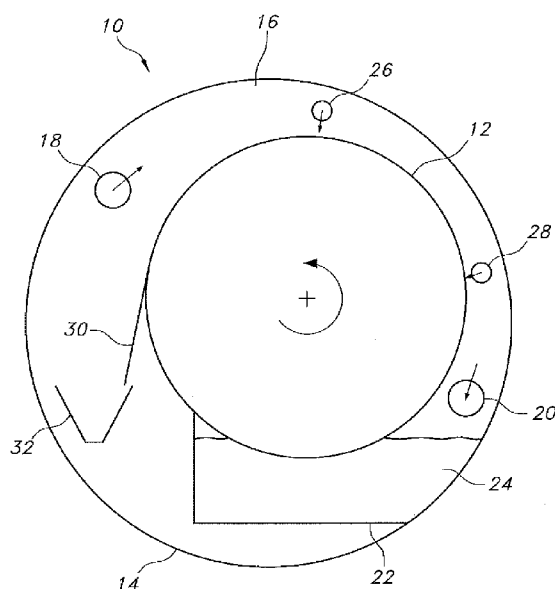


FIG. 1

(57) Abstract: The present invention relates to a gas-driven rotary filter comprising: a) a housing; b) a filter drum positioned within the housing and rotatable about its axis in a first direction within the housing; and c) means to provide a flow of gas within a gas space between the circumference of the filter drum and the housing in a second direction, characterised in that the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through a gas space between an end of the filter drum and the housing. The invention also relates to a method for separating a solid from a solvent in the gas-driven rotary filter, and a process for manufacturing an aromatic dicarboxylic acid incorporating this method.

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GAS-DRIVEN ROTARY FILTER**TECHNICAL FIELD**

The present invention relates to a gas-driven rotary filter, a method for separating a solid from a solvent in the gas-driven rotary filter, and a process for manufacturing an aromatic dicarboxylic acid incorporating this method.

BACKGROUND ART

Gas-driven filters, such as rotary filters, are used in numerous chemical processes that require the separation of solids from liquids, such as the manufacture of synthetic intermediates, for instance aromatic dicarboxylic acids such as terephthalic acid (TA), which is widely used in the manufacture of polyesters, such as poly(ethylene terephthalate) (PET). A typical gas-driven rotary filter comprises a plurality of filter cells arranged around the circumference of a rotatable filter drum, the interior of which contains a filter mechanism typically made up of a series of drainage pipes that connect filter cloths or membranes at the base of the filter cells to a mechanism for collecting and redirecting (e.g. to a different location in the filter) or discharging fluids (e.g. filtrate, wash fluids). The gas space between the circumference of the filter drum and the housing in which it resides is maintained at a higher pressure than the space below the filter cloths by the injection of an inert gas (e.g. nitrogen) into this gas space, which provides a motive force for filtration. The end walls of the housing are typically "dished" (i.e. convex) to withstand the force exerted by the pressurised gas within it. A slurry is contacted with the outer surface of the filter drum at a load point, the drum is rotated, and a resultant filter cake is discharged from the outer surface of the filter drum at a discharge point. In addition to separating a slurry into filter cake and filtrate, gas-driven rotary filters may subject the filter cake to subsequent treatment (e.g. washing) before discharging it. These gas-driven rotary filters are therefore useful in the solvent interchange step of processes for manufacturing aromatic dicarboxylic acids.

Aromatic dicarboxylic acids are commonly manufactured by the catalytic oxidation of a hydrocarbon precursor in an organic solvent. 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. in 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).

5 The CTA is transferred from the organic solvent-based system (typically comprising acetic acid) in which it is produced to a water-based system via a solvent interchange step prior to the purification process. It is desirable to minimise carry-forward of organic solvent from this step to minimise the contamination of the PTA product by this organic solvent, and to maximise the recovery of the organic solvent raw material
10 for recycle to the oxidation reaction. In addition, where the organic solvent is acetic acid, its removal at this stage avoids the need to use materials with increased corrosion resistance in the equipment downstream of this stage, thus reducing capital cost. In this solvent interchange step, CTA may be filtered out of the organic solvent-based slurry resulting from the oxidation reaction using a gas-driven rotary filter, and the resultant filter cake washed on the filter drum with water to remove as much organic
15 solvent as possible from the filter cake before it is discharged to the purification process.

The residual organic solvent content of the CTA discharged to the purification process has been found to be higher than might be expected from simple washing efficiency calculations. It is believed that this is due, in part, to contamination of the CTA filter cake via the gas space between the circumference of the
20 filter drum and the housing. This contamination is caused by entrainment of droplets of wash liquid contaminated with the organic solvent, and/or by entrainment of organic solvent vapour, into the filter cake. It has been suggested that the contamination of the CTA filter cake may be reduced by providing a flow of inert gas in the gas space between the circumference of the filter drum and the housing that is counter-current to the direction of travel of the CTA filter cake, i.e. from a location near the discharge
25 point to a location near the load point.

It is an object of the present invention to provide a gas-driven rotary filter in which the contamination of the filter cake with the solvent from which it has been separated is minimised. It is a further object of the present invention to provide a more economic and efficient process for the manufacture of aromatic
30 dicarboxylic acids. Further objects will be apparent from the description below.

DISCLOSURE OF THE INVENTION

It has been found that even when a counter-current flow of inert gas in the gas space between the circumference of the filter drum and the housing of a typical gas-driven rotary filter is provided, the
35 contamination of the filter cake with the solvent from which it has been separated remains higher than might be expected. Unexpectedly, it has also been found that this contamination may occur, at least in

part, due to migration of solvent vapour through the gas space between the ends of the filter drum and the housing.

A first aspect of the present invention provides a gas-driven rotary filter comprising:

- a) a housing;
- b) a filter drum positioned within the housing and rotatable about its axis in a first direction within the housing; and
- c) means to provide a flow of gas within a gas space between the circumference of the filter drum and the housing in a second direction,

characterised in that the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through a gas space between an end of the filter drum and the housing.

The inventors have found that by inhibiting (i.e. reducing or preventing altogether) migration of solvent vapour through the gas space between the end of the filter drum and the housing, contamination of the filter cake that is discharged from the gas-driven rotary filter with the solvent is reduced. Preferably, the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through gas spaces between both ends of the filter drum and the housing to maximise the reduction in the contamination of the filter cake. The migration of the solvent vapour may be inhibited by providing one or more baffle(s) extending between the housing and the filter drum, providing the filter drum and the housing with at least one flat end wall to minimise or eliminate the gas space between this end wall of the filter drum and the housing, and/or by providing a positive gas pressure within the gas space between the end of the filter drum and the housing to inhibit migration of the solvent vapour into this gas space.

The first direction and the second direction are suitably not the same. Preferably, the second direction is opposite to the first direction (e.g. if the first direction is clockwise, the second direction is anti-clockwise) to reduce the migration of solvent vapour around the circumference of the filter drum, thus reducing the contamination of the filter cake with the solvent from which it has been separated.

This aspect of the present invention also provides a method of separating a solid from a solvent in a gas-driven rotary filter comprising the steps of:

- (i) introducing a mixture of the solid and the solvent onto an outer surface of a filter drum within a housing of the gas-driven rotary filter;
- (ii) rotating the filter drum about its axis in a first direction within the housing;
- (iii) providing a flow of gas within a gas space between the circumference of the filter drum and the housing in a second direction,

characterised in that the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through a gas space between an end of the filter drum and the housing.

The solid may be an aromatic dicarboxylic acid, in particular a crude aromatic dicarboxylic acid, such as crude terephthalic acid. The solvent may be an organic solvent, such as acetic acid.

- 5 Accordingly, a further aspect of the invention 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
- 10 II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid, wherein the process further comprises the step of separating solid crude aromatic dicarboxylic acid from the organic solvent via the method of the first aspect of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

- 15 **Error! Reference source not found.** is a cross section of a conventional gas-driven rotary filter.
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- 20 **Error! Reference source not found.** is a radial section and a cross section of a gas-driven rotary filter of the present invention.
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- 25 **Error! Reference source not found.** is a radial section and a cross section of a gas-driven rotary filter of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

- 30 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.

A gas-driven rotary filter comprises a filter drum positioned within a housing.

35 *Housing*

The housing contains the filter drum, which is rotatable about its axis in a first direction within the housing. The housing is typically substantially cylindrical, but other shapes may be employed. If the housing is

cylindrical, the axis of the housing may be parallel to the axis of the filter drum, and these axes may be aligned but are typically offset relative to one another. Alternatively, the axis of the housing may be perpendicular to the axis of the filter drum. The housing typically contains the means to provide the flow of gas within the gas space between the circumference of the filter drum and the housing in the second direction. The housing typically also contains means for contacting a slurry (i.e. a mixture of solid and solvent) with the outer surface of the filter drum at a load point, means for discharging a filter cake from the outer surface of the filter drum at a discharge point, and one or more wash liquid inlets.

The gas space between the circumference of the filter drum and the housing is maintained at a higher pressure than the space within the filter drum by the injection of an inert gas (e.g. nitrogen) into this gas space. This pressure differential provides a motive force for filtration. The gas pressure in the gas space between the circumference of the filter drum and the housing is typically about 2 barg, although pressures of up to about 4 barg, up to about 6 barg, up to about 8 barg, or up to about 10 barg may be used. Conventionally, the end walls of the housing (commonly known as "heads") are dished (e.g. hemispherical or torispherical) to withstand the force exerted by the pressurised gas within it. However, in the present invention, the end wall(s) of the filter drum and the wall(s) of the housing adjacent the end(s) of the filter drum at at least one end, and preferably at both ends, of the filter drum may be flat to minimise or eliminate any gas space between the end(s) of the filter drum and the housing, thus inhibiting migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. In this embodiment, the end wall(s) of the filter drum and the wall(s) of the housing are suitably adjacent in that they are positioned as close together as possible without interfering with the rotation of the filter drum, thus minimising or eliminating any gas space between the end(s) of the filter drum and the housing.

The gas space(s) between the end, preferably both ends, of the filter drum and the housing may be provided with a positive gas pressure to inhibit migration of the solvent vapour into the gas space(s). Specifically, the gas-driven rotary filter may comprise means for injecting an inert gas (e.g. nitrogen) into the gas space(s) between the end, preferably both ends, of the filter drum and the housing, suitably at a pressure greater than that in the gas space between the circumference of the filter drum and the housing, e.g. the inert gas may be injected into the gas space between the end (or both ends) of the filter drum and the housing at greater than about 2 barg, or greater than about 4 barg, or greater than about 6 barg, or greater than about 8 barg, or greater than about 10 barg.

Baffles

The gas-driven rotary filter may comprise one or more baffle(s) extending between the housing and the filter drum, inhibiting migration of solvent vapour through the gas space between the end, preferably both ends, of the filter drum and the housing, particularly where the ends of the housing are dished. The baffle(s) may be attached to the housing or the filter drum, although, as the filter drum is rotatable, it is

preferred that the baffle(s) be attached to the housing. The baffle(s) suitably extend all the way from the housing to the filter drum, i.e. the baffle(s) suitably extend from the housing to make contact with the filter drum (or vice versa), typically forming an airtight seal and thereby forming a physical barrier to the migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. Alternatively, one or more baffle(s) may extend from the housing and one or more baffle(s) may extend from the filter drum to communicate with (e.g. contact) the one or more baffle(s) extending from the housing, thus typically forming an airtight seal and thereby forming a physical barrier to the migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. In either configuration, if there is a small gap between the baffle and the filter drum/housing, or between the respective baffles, migration of solvent vapour through the gas space between the end(s) of the filter drum and the housing can still be inhibited.

In one configuration, the gas-driven rotary filter comprises one or more annular baffle(s) extending between the end(s), preferably both ends, of the filter drum and the housing, inhibiting solvent vapour from entering the gas space between the end(s) of the filter drum and the housing. The annular baffle(s) suitably extend longitudinally between the end(s) of the filter drum and the housing, and suitably have the same diameter as the filter drum, thus can be viewed as extending the outer surface of the filter drum to join the wall(s) of the housing. The annular baffle(s) may extend around the whole circumference of the filter drum, or around part of the circumference whilst remaining effective to inhibit solvent vapour from entering the gas space(s) between the end(s) of the filter drum and the housing. For instance, the annular baffle(s) may extend around the circumference of the filter drum from the load point to the discharge point in the direction of rotation of the filter drum (i.e. the first direction).

In a further configuration, the gas-driven rotary filter comprises one or more circumferential baffle(s) extending between the filter drum and the housing (i.e. suitably extending radially between the circumference of the filter drum and the housing), thus inhibiting solvent vapour from migrating axially across the filter drum to enter the gas space(s) between the end(s) of the filter drum and the housing. As the concentration of solvent vapour in the gas space between the circumference of the filter drum and the housing is typically greatest around the load point, the baffle(s) suitably extend circumferentially from the load point towards the discharge point, thus inhibiting solvent vapour from migrating axially across the filter drum to enter the gas space(s) between the end(s) of the filter drum and the housing adjacent the load point. The baffle(s) may extend circumferentially across the whole distance from the load point towards the discharge point in the direction of rotation of the filter drum (i.e. the first direction), or across part of this distance, e.g. across up to half of this distance, or across up to a quarter of this distance. Preferably, the gas-driven rotary filter comprises a circumferential baffle extending between the filter drum and the housing at both ends of the filter drum, thus inhibiting solvent vapour from entering the gas spaces between the ends of the filter drum and the housing. The gas-driven rotary filter may further

comprise additional circumferential baffle(s) between these two circumferential baffles to inhibit solvent vapour from migrating axially across the filter drum.

If the housing is cylindrical and its axis is aligned with that of the filter drum, one or more circular circumferential baffle(s) may be attached to the filter drum and extend between the housing and the filter drum. Alternatively, particularly if the housing is not cylindrical and/or its axis is not aligned with that of the filter drum, one or more circular circumferential baffle(s) may be attached to the filter drum and extend between the filter drum and one or more corresponding baffle(s) attached to the housing. The circular circumferential baffle(s) attached to the filter drum may thus communicate (e.g. they may abut, or overlap) with the baffle(s) attached to the housing, typically forming an airtight seal and thereby inhibiting migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. A sealing bead or a ring of a sealant material may be included where the baffles communicate to ensure the formation of an airtight seal. The baffle(s) attached to the housing may therefore include a circular opening that is the same size as or smaller than the circular circumferential baffle(s) attached to the filter drum. If the baffles overlap, the baffle(s) attached to the housing may be displaced outward (i.e. towards the wall(s) of the housing) of the circular circumferential baffle(s) attached to the filter drum, or may be displaced inward of the circular circumferential baffle(s) attached to the filter drum. The circular circumferential baffle(s) are suitably positioned at the end, preferably both ends, of the filter drum, and the baffle(s) attached to the housing are positioned in a corresponding location on the housing.

In a further configuration, the gas-driven rotary filter comprises one or more longitudinal baffle(s) extending between the end(s), preferably both ends, of the filter drum and the housing, thus inhibiting migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. In particular, the baffle(s) may extend radially from the axis of the filter drum to the circumference of the filter drum, preferably from the axis of the filter drum to the housing, and extend longitudinally between the end(s) of the filter drum and the housing, inhibiting solvent vapour that has entered the gas space(s) between the end(s) of the filter drum and the housing from exiting the gas space(s) in the location of the discharge point. The gas-driven rotary filter may comprise a plurality of such longitudinal/radial baffles, spaced at intervals around the whole circumference of the filter drum, or around part of the circumference, e.g. from the load point to the discharge point in the direction of rotation of the filter drum (i.e. the first direction). Thus, if the gas-driven rotary filter comprises a first longitudinal/radial baffle and a second longitudinal/radial baffle arranged between the load point and the discharge point in the direction of rotation of the filter drum and the angle between the first longitudinal/radial baffle and the second longitudinal/radial baffle is X° , solvent vapour entering the gas space between the ends of the filter drum and the housing and between the first longitudinal/radial baffle and the second longitudinal/radial baffle cannot exit this gas space more than X° further around the circumference of the filter drum in the direction of rotation of the filter drum from the point at which it

entered this gas space. The gas-driven rotary filter may suitably comprise at least two, at least three, at least four, at least five, or at least six longitudinal/radial baffles at one, or preferably at both, ends of the filter drum. These longitudinal/radial baffles may be evenly spaced around the filter drum, and/or may be separated by at least 10°, at least 20°, at least 30°, at least 40°, at least 50°, or at least 60°, and/or by less than 120°, less than 110°, less than 100°, less than 90°, less than 80°, or less than 70°. The gas-driven rotary filter may comprise one or more longitudinal/radial baffle(s) spaced at least 10°, at least 20°, at least 30°, at least 40°, at least 50°, at least 60°, at least 70°, at least 80°, at least 90°, and/or by less than 180°, less than 170°, less than 160°, less than 150°, less than 140°, less than 130°, less than 120°, less than 110°, or less than 100° from the load point in the first direction (i.e. the direction of rotation of the filter drum).

In one embodiment, the gas-driven rotary filter comprises the annular baffle(s) and circumferential baffle(s) described above. In another embodiment, the gas-driven rotary filter comprises the annular baffle(s) and longitudinal/radial baffle(s) described above. In this embodiment, the longitudinal/radial baffle(s) extend longitudinally between the filter drum and the housing and radially between the annular baffle(s) and the housing. In another embodiment, the gas-driven rotary filter comprises the circumferential baffle(s) and longitudinal/radial baffle(s) described above. In another embodiment, the gas-driven rotary filter comprises the annular baffle(s), circumferential baffle(s) and longitudinal/radial baffle(s) described above.

The gas-driven rotary filter may comprise any combination of the above features for inhibiting migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing. In particular, the gas-driven rotary filter may comprise one or more baffle(s) extending between the housing and the filter drum and may comprise means for injecting an inert gas into the gas space(s) between the end(s) of the filter drum and the housing.

Filter drum

The filter drum is suitably cylindrical and comprises a plurality of filter cells (typically between 16 and 24) arranged around its circumference (i.e. its curved surface, as opposed to its end surface, which is typically flat). Thus, the outer surface of the filter drum may comprise a plurality of circumferentially arranged filter cells and/or a plurality of longitudinally arranged filter cells that may form an array of filter cells across at least part (and preferably all) of the surface of the filter drum. The filter cells are defined, and separated from each other, by filter cell walls which extend radially out from the surface of the filter drum, and are typically arranged circumferentially and longitudinally across its surface. In a typical filter drum, the filter cells accommodate filter cell inserts, which incorporate the filter medium (typically referred to as a filter cloth or membrane) and are fastened (typically bolted) to the drum. The filter cloth allows fluids (e.g. filtrate (solvent), wash fluids) to pass through but collects filter cake (i.e. solids) upon its

surface when a slurry is applied to the filter drum. The filter cloth is typically made of plastic or metal fabric, as required by the application, as is conventional in the art. Thus, each filter cell is suitably defined by four walls, with the filter cloth at the base of the cell, thus forming a pocket in the outer surface of the filter drum into which fluids (e.g. slurry, wash fluids) can flow and in which solid material can collect. Cake thickness is typically within a range of 5 to 200mm, or 50 to 175mm, or 120 to 150mm, and can be varied by inserting spacers with the inserts.

The filter cell therefore provides a fluid inlet in the outer surface of the filter drum. Fluid that passes through the filter cloth is collected by a filter mechanism that is typically made up of a series of drainage pipes that connect the filter cloths to a mechanism outside the filter drum for collecting and redirecting or discharging the fluid. A fluid pathway from the outer surface of the filter drum to the mechanism for collecting and redirecting or discharging the fluid is therefore provided by the filter cells, the filter cloths, and the drainage pipes. The filter mechanism is typically enclosed by the filter drum, thus maintaining the pressure differential across the filter cloths.

Gas flow within the housing

The housing typically contains the means to provide the flow of gas within the gas space between the circumference of the filter drum and the housing in the second direction. Typically, this means comprises one or more gas inlet(s) and one or more gas outlet(s) displaced from the one or more gas inlet(s) in the second direction. As noted above, the first direction and the second direction are suitably not the same, i.e. the gas does not flow in the direction of rotation of the filter drum. For instance, the second direction may be perpendicular to the first direction. Preferably, the second direction is opposite to the first direction, i.e. the flow of gas is in the opposite direction (counter-current) to the rotation of the filter drum. Accordingly, the one or more gas outlet(s) may be displaced from the one or more gas inlet(s) in the direction of the load point. The one or more gas inlet(s) and, optionally, the one or more gas outlet(s) may extend longitudinally across the length of the filter drum to provide a consistent gas flow in the second direction across the length of the filter drum.

Load point

The gas-driven rotary filter suitably comprises means for contacting a slurry with the outer surface of the filter drum at a load point within the housing. Typically, this means comprises a trough that, during operation of the gas-driven rotary filter, contains a slurry in which the filter drum is partially submerged. Accordingly, the slurry in the trough prevents migration of solvent vapour from the load point to the discharge point between the circumference of the filter drum and the housing in the opposite direction to the direction of rotation of the filter drum. One or more baffle(s) may be provided between the trough and the housing to inhibit migration of solvent vapour towards the discharge point via this route. If one or more circular circumferential baffle(s) is attached to the end(s) of the filter drum, a seal may be provided

between the end(s) of the trough and the circular circumferential baffle(s). The trough is suitably continuously supplied with a slurry, such as a slurry of a solid crude aromatic dicarboxylic acid in an organic solvent, at a pressure similar to that of the gas space between the circumference of the filter drum and the housing, thus maintaining the level of the slurry in the trough.

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Discharge point

The gas-driven rotary filter suitably comprises means for discharging a filter cake from the outer surface of the filter drum at a discharge point within the housing. Typically, this means comprises a plough or knife, which is arranged to scrape the filter cake off the outer surface of the filter drum as the filter drum rotates (e.g. the plough or knife may be arranged tangentially to the outer surface of the filter drum) and which is also suitably arranged above a chute or a conveyor for removing the filter cake from the gas-driven rotary filter. The gas-driven rotary filter may also comprise means for reslurrying the filter cake at or adjacent to the discharge point, e.g. the gas-driven rotary filter may comprise one or more water inlet(s) for applying water to filter cake that has been scraped off the outer surface of the filter drum.

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Wash liquid inlets

The gas-driven rotary filter suitably comprises one or more wash liquid inlet(s), which are suitably positioned within the housing to wash filter cake on the outer surface of the filter drum, i.e. between the circumference of the filter drum and the housing and between the load point and the discharge point. The one or more wash liquid inlet(s) irrigate the filter cake, typically with an aqueous wash liquid (e.g. water), to wash residual solvent and, optionally, one or more other contaminants out of the filter cake. The one or more wash liquid inlet(s) may extend longitudinally across the length of the filter drum to provide consistent washing of the filter cake. The gas-driven rotary filter may comprise one or more first wash liquid inlet(s) and one or more second wash liquid inlet(s) that are displaced from the one or more first wash liquid inlet(s) in the opposite direction to the direction of rotation of the filter drum. Fresh wash liquid (e.g. clean water, or water from elsewhere in an aromatic dicarboxylic acid manufacturing process) may be supplied to the one or more first wash liquid inlet(s). Wash liquid from the one or more first wash liquid inlet(s) that passes through the filter cloth may be collected by the filter mechanism that connects the filter cloth to the mechanism outside the filter drum for collecting and redirecting or discharging the fluid, which may thus redirect the collected wash fluid to the one or more second wash liquid inlet(s). In other words, the gas-driven rotary filter may be configured to provide a counter-current flow of wash liquid relative to the direction of rotation of the filter drum via one or more first wash liquid inlet(s) and one or more second wash liquid inlet(s). The gas-driven rotary filter may further comprise one or more further wash liquid inlet(s) that are displaced from the one or more second wash liquid inlet(s) in the opposite direction to the direction of rotation of the filter drum. Accordingly, the counter-current flow of wash liquid may continue around the circumference of the filter drum via the one or more further wash liquid inlet(s).

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The use of counter-current flow of wash fluid in which fresh wash liquid (e.g. fresh water) is supplied to the supplied to the first wash liquid inlet(s) only, rather than to each wash liquid inlet(s), reduces the fresh water requirement of the gas-driven rotary filter, and also reduces the amount of water discharged from the gas-driven rotary filter, which may require further processing to remove the solvent and other contaminants before it can be released to the environment. However, solvent from which the solid is separated in the gas-driven rotary filter (e.g. acetic acid) may become entrained in the wash fluid through the washing of the filter cake, and may thus be introduced into the gas space between the circumference of the filter drum and the housing via subsequent wash liquid inlet(s), providing a potential source of solvent vapour that may contaminate the filter cake. Nevertheless, the use of the present invention mitigates the risk of this contamination, allowing the benefits of reducing the water usage to be obtained without increasing contamination of the filter cake to a degree that would outweigh these benefits.

Production of an aromatic dicarboxylic acid

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.).

The terephthalic acid is typically produced by a process comprising the catalytic oxidation of a hydrocarbon precursor in an organic solvent. The hydrocarbon precursor is a compound that may be oxidised to form the terephthalic acid. Thus, the hydrocarbon precursor is typically benzene substituted with groups such as C₁₋₆alkyl, formyl, or acetyl in the positions of the carboxylic acid substituents in terephthalic acid. 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 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 the vent gas. The product stream is typically transferred to a crystallisation stage to form a first slurry of crude terephthalic acid crystals and an overhead vapour. The first slurry of crude terephthalic acid crystals is typically passed to a separation (or solvent interchange) stage, in which a mother liquor is separated from the crude terephthalic acid crystals, which may then be mixed with an aqueous liquid to form a second slurry of crude terephthalic acid crystals. This second slurry of crude terephthalic acid crystals is typically transferred to a purification plant, heated and subjected to hydrogenation, before being cooled to form a slurry of purified terephthalic acid crystals.

The vent gas from the oxidation stage is typically separated in a distillation stage into an organic solvent-rich stream and a water-rich vapour stream. The organic solvent-rich 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. The condensate stream typically forms a portion of the wash fluid for the purified terephthalic acid crystals from the purification plant. In the present invention, a portion of the condensate stream is suitably used as the fresh wash liquid, and/or as the aqueous liquid used to form the second slurry of crude terephthalic acid crystals mentioned above (alternatively, demineralised water may be used).

The operation of a conventional gas-driven rotary filter is described with reference to **Error! Reference source not found.** Gas-driven rotary filter 10 comprises filter drum 12 positioned within housing 14 and rotatable anti-clockwise about its axis within housing 14. Gas space 16 between the circumference of filter drum 12 and housing 14 is maintained at about 2 barg by the injection of nitrogen at gas inlet 18. Gas flows clockwise from gas inlet 18 to gas outlet 20. Trough 22 contains slurry 24 of solid and solvent. The solid is loaded onto filter drum 12 by the pressure differential across filter drum 12. First wash liquid inlet 26 irrigates the solid on the outer surface of filter drum 12 with fresh water, which passes through the solid and is collected by a filter mechanism (not shown) and redirected to second wash inlet 28, which also irrigates the solid on the outer surface of filter drum 12 with this water. Knife 30 scrapes the solid off the outer surface of filter drum 12 for collection in chute 32.

Error! Reference source not found. shows gas-driven rotary filter 110 in which annular baffles 116 extend between both ends of filter drum 112 and housing 114, which has dished ends. Annular baffles 116 have the same diameter as filter drum 112.

Error! Reference source not found. shows gas-driven rotary filter 120 in which annular baffles 126 extend between both ends of filter drum 122 and housing 124, similarly to gas-driven rotary filter 110. In

addition, longitudinal/radial baffles 128 extend both longitudinally and radially between annular baffles 126 and housing 124.

5 **Error! Reference source not found.** shows gas-driven rotary filter 130 in which circumferential baffles 136 extend between both ends of filter drum 132 and housing 134.

10 **Error! Reference source not found.** shows gas-driven rotary filter 140 in which the axis of filter drum 142 is offset relative to the axis of housing 144. Circular circumferential baffles 146 are attached to both ends of filter drum 142 and communicate with housing baffles 148, which are attached to housing 144. A seal may be included between circular circumferential baffles 146 and housing baffles 148.

15 **Error! Reference source not found.** shows gas-driven rotary filter 150 in which longitudinal/radial baffles 156 extend longitudinally between both ends of filter drum 152 and housing 154 and radially from the axis of filter drum 152 to housing 154.

Error! Reference source not found. shows gas-driven rotary filter 160 in which the end walls of filter drum 162 and housing 164 are flat.

CLAIMS

1. A gas-driven rotary filter comprising:
 - a) a housing;
 - b) a filter drum positioned within the housing and rotatable about its axis in a first direction within the housing; and
 - c) means to provide a flow of gas within a gas space between the circumference of the filter drum and the housing in a second direction,characterised in that the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through a gas space between an end of the filter drum and the housing.
2. A method of separating a solid from a solvent in a gas-driven rotary filter comprising the steps of:
 - (i) introducing a mixture of the solid and the solvent onto an outer surface of a filter drum within a housing of the gas-driven rotary filter;
 - (ii) rotating the filter drum about its axis in a first direction within the housing;
 - (iii) providing a flow of gas within a gas space between the circumference of the filter drum and the housing in a second direction,characterised in that the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through a gas space between an end of the filter drum and the housing.
3. The gas-driven rotary filter of claim 1 or the method of claim 2, wherein the gas-driven rotary filter is adapted to inhibit migration of solvent vapour through gas spaces between both ends of the filter drum and the housing.
4. The gas-driven rotary filter or the method of any preceding claim, wherein the second direction is opposite to the first direction.
5. The gas-driven rotary filter or the method of any preceding claim, wherein the gas-driven rotary filter comprises one or more baffle(s) extending between the housing and the filter drum to inhibit migration of solvent vapour through the gas space(s) between the end(s) of the filter drum and the housing.
6. The gas-driven rotary filter or the method of claim 5, wherein the gas-driven rotary filter comprises one or more annular baffle(s) extending between the end(s) of the filter drum and the housing.
7. The gas-driven rotary filter or the method of claim 6, wherein the one or more annular baffle(s) have the same diameter as the filter drum.

8. The gas-driven rotary filter or the method of claim 6 or claim 7, wherein the gas-driven rotary filter comprises one or more annular baffle(s) extending between both ends of the filter drum and the housing.
- 5 9. The gas-driven rotary filter or the method of any one of claims 6-8, wherein one or more longitudinal/radial baffle(s) extend longitudinally between the filter drum and the housing and radially between the annular baffle(s) and the housing.
- 10 10. The gas-driven rotary filter or the method of claim 8, wherein one or more longitudinal/radial baffle(s) extend longitudinally between the filter drum and the housing and radially between the annular baffle(s) and the housing at both ends of the filter drum.
- 15 11. The gas-driven rotary filter or the method of any one of claims 5-10, wherein the gas-driven rotary filter comprises one or more circumferential baffle(s) extending between the filter drum and the housing.
- 20 12. The gas-driven rotary filter or the method of claim 11, wherein the gas-driven rotary filter comprises a circumferential baffle extending between the filter drum and the housing at both ends of the filter drum.
- 25 13. The gas-driven rotary filter or the method of any one of claims 5-10, wherein the gas-driven rotary filter comprises one or more circular circumferential baffle(s) attached to the filter drum that extend between the filter drum and one or more corresponding baffle(s) attached to the housing.
- 30 14. The gas-driven rotary filter or the method of claim 13, wherein the gas-driven rotary filter comprises a circular circumferential baffle attached to the filter drum that extends between the filter drum and a corresponding baffle attached to the housing at both ends of the filter drum.
- 35 15. The gas-driven rotary filter or the method of any one of claims 5-14, wherein the gas-driven rotary filter comprises one or more longitudinal baffle(s) extending between the end(s) of the filter drum and the housing.
16. The gas-driven rotary filter or the method of claim 15, wherein the gas-driven rotary filter comprises one or more longitudinal baffle(s) extending between both ends of the filter drum and the housing.
17. The gas-driven rotary filter or the method of claim 15 or claim 16, wherein the one or more longitudinal baffle(s) extend radially from the axis of the filter drum to the housing.

18. The gas-driven rotary filter or the method of any preceding claim, wherein the end wall(s) of the filter drum and the wall(s) of the housing adjacent the end(s) of the filter drum at at least one end are flat to minimise or eliminate any gas space between the ends of the filter drum and the housing.
- 5 19. The gas-driven rotary filter or the method of claim 18, wherein the end walls of the filter drum and the walls of the housing adjacent the ends of the filter drum at both ends are flat to minimise or eliminate any gas space between the ends of the filter drum and the housing.
- 10 20. The gas-driven rotary filter or the method of any preceding claim, wherein the gas-driven rotary filter comprises means for injecting an inert gas into the gas spaces between the end(s) of the filter drum and the housing.
- 15 21. The gas-driven rotary filter or the method of claim 20, wherein the gas-driven rotary filter comprises means for injecting an inert gas into the gas spaces between both ends of the filter drum and the housing.
- 20 22. The gas-driven rotary filter or the method of any preceding claim, wherein the gas-driven rotary filter comprises one or more gas inlet(s) and one or more gas outlet(s) displaced from the one or more gas inlet(s) in the second direction and positioned within the housing.
- 25 23. The gas-driven rotary filter or the method of any preceding claim, wherein the gas-driven rotary filter comprises one or more first wash liquid inlet(s) and one or more second wash liquid inlet(s) positioned within the housing to wash filter cake on the outer surface of the filter drum and wherein the gas-driven rotary filter is configured to provide a counter-current flow of wash liquid relative to the direction of rotation of the filter drum via the one or more first wash liquid inlet(s) and the one or more second wash liquid inlet(s).
- 30 24. The method of any one of claims 2-23, wherein the solid is an aromatic dicarboxylic acid, preferably a crude aromatic dicarboxylic acid.
25. The method of claim 24, wherein the crude aromatic dicarboxylic acid is crude terephthalic acid.
- 35 26. 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:
- l) oxidising the hydrocarbon precursor in the organic solvent in the presence of a metal catalyst to provide a crude aromatic dicarboxylic acid; and

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II) purifying the crude aromatic dicarboxylic acid to yield the purified aromatic dicarboxylic acid, wherein the process further comprises the step of separating solid crude aromatic dicarboxylic acid from the organic solvent via the method of any one of claims 2-25.

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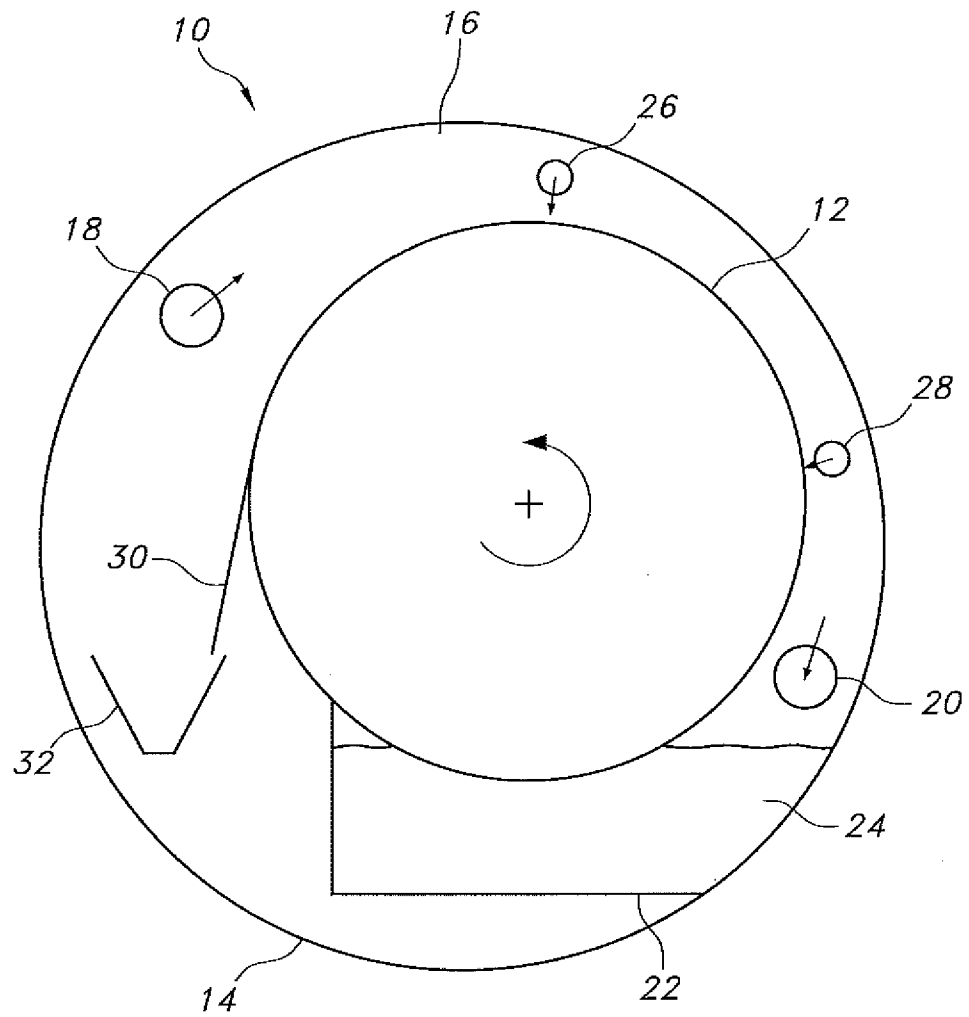


FIG. 1

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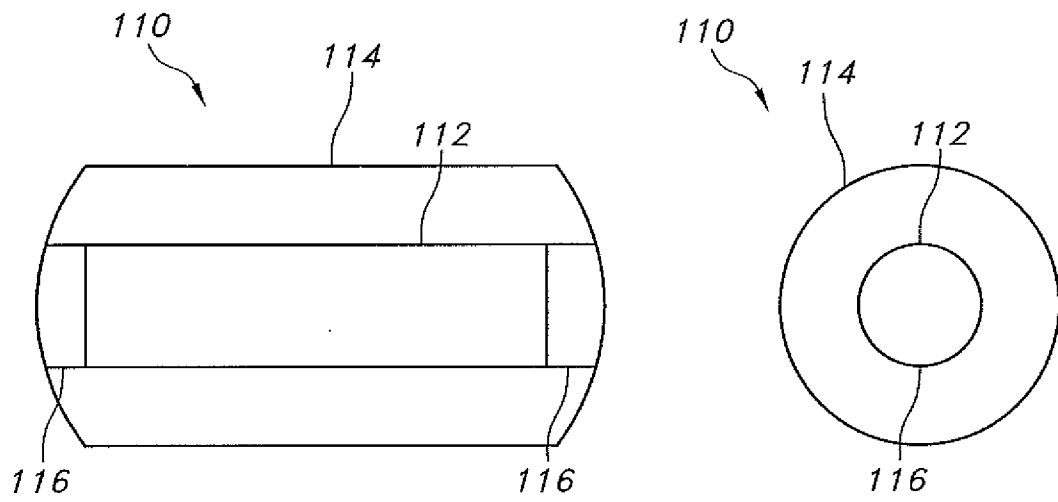


FIG. 2

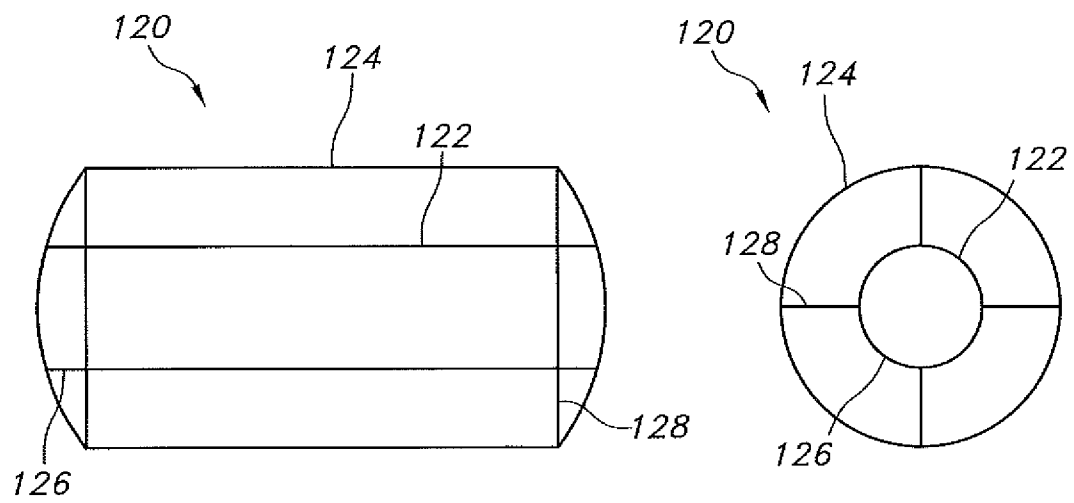


FIG. 3

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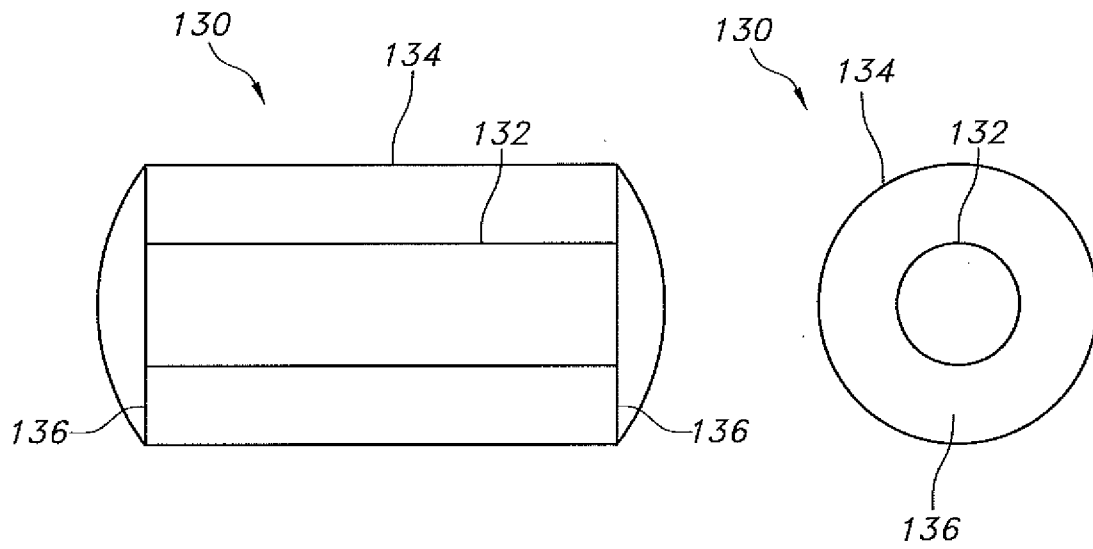


FIG. 4

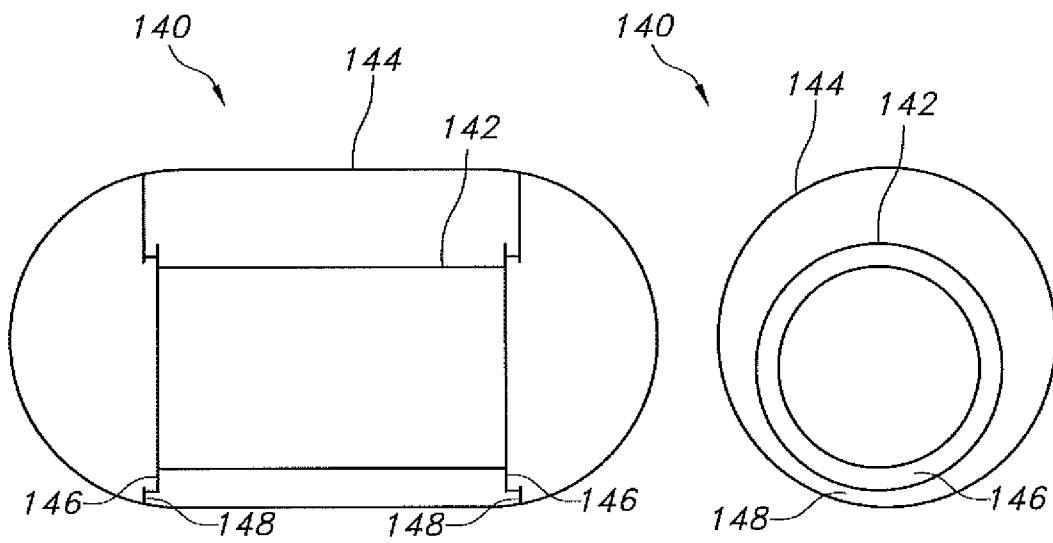


FIG. 5

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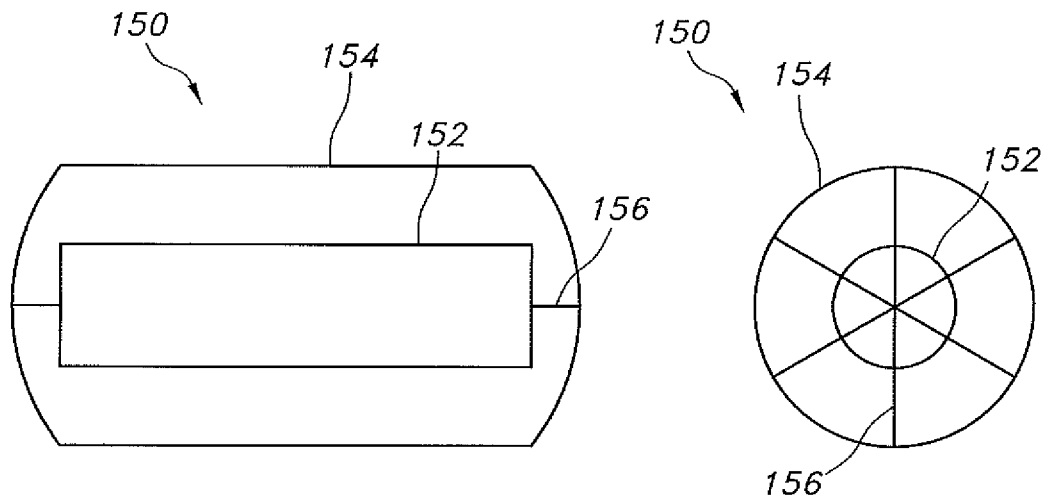


FIG. 6

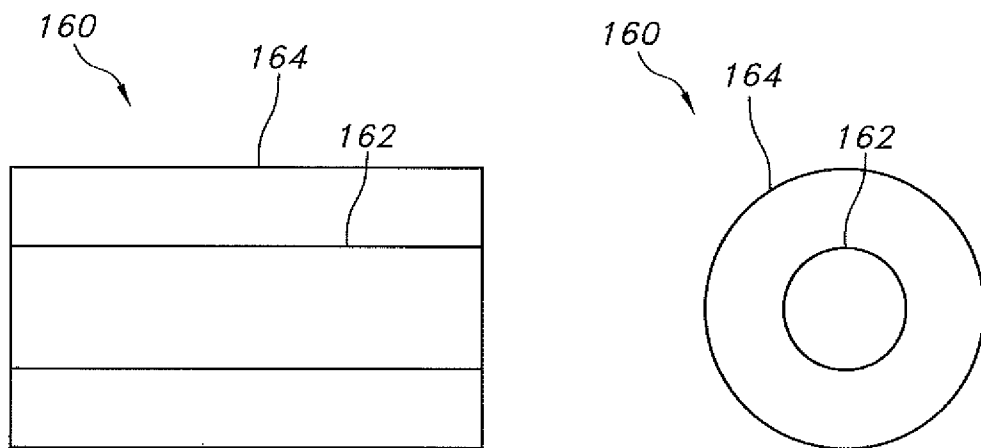


FIG. 7

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2016/012580**A. CLASSIFICATION OF SUBJECT MATTER****B01D 33/067(2006.01)i, B01D 33/06(2006.01)j**

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)

B01D 33/067; C07C 51/47; C07C 51/43; B01D 33/58; B01D 33/073; C07C 51/42; B01D 33/06; C07C 63/26

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

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

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

eKOMPASS(KIPO internal) & Keywords: rotary filter, gas-driven, migration, baffle, vapor

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2013-0190530 A1 (GRAY, JULIAN STUART et al.) 25 July 2013 See abstract; paragraphs [0005],[0006]; claims 1, 14; and figure 1.	1-3
A	US 2011-0269991 A1 (JACKSON, CHRISTOPHER HOWARD et al.) 03 November 2011 See abstract; paragraph [0018]; and figure 1.	1-3
A	EP 0630673 B2 (MITSUI CHEMICALS, INC.) 28 December 1994 See paragraph [0017]; and figure 1.	1-3
A	EP 1170280 B1 (MITSUBISHI GAS CHEMICAL COMPANY, INC. et al.) 09 January 2002 See paragraphs [0011]-[0013]; and figures 1, 2.	1-3
A	US 6336561 B1 (KOSSIK, JOHN MICHAEL et al.) 08 January 2002 See abstract; and claims 1, 20.	1-3

☐ 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

21 April 2016 (21.04.2016)

Date of mailing of the international search report

22 April 2016 (22.04.2016)

Name and mailing address of the ISA/KR

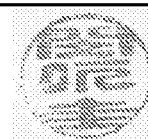
International Application Division
Korean Intellectual Property Office
189 Cheongsa-ro, Seo-gu, Daejeon, 35208, Republic of Korea

Facsimile No. +82-42-481-8578

Authorized officer

MIN, In Gyou

Telephone No. +82-42-481-3326



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2016/012580

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 6,7,10,12,14,16,19,21,25
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
* Claims 6,7,10,12,14,16,19,21,25 are unclear since they refer to claims which are not searchable due to not being drafted in accordance with the second and third sentence of Rule 6.4(a).

3. ☒ Claims Nos.: 4,5,8,9,11,13,15,17,18,20,22,23,24,26
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of any additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2016/012580

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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US 6336561 B1	08/01/2002	US 2002-0008061 A1	24/01/2002