

PROCESS AND PLANT FOR PURIFICATION
OF A GAS STREAM CONTAINING ACROLEIN

The present invention relates to a process for the purification of a gas stream including acrolein to extract the acrolein therefrom.

Acrolein is a raw material, a major industrial use of which is the synthesis of beta-methylthiopropionaldehyde (MTPA) by reaction of acrolein with methyl mercaptan.

Acrolein is produced by gas-phase oxidation with air in the presence of water. The gas-phase oxidation conventionally produces a gaseous mixture comprising acrolein in an amount that is higher than 10 % by weight, gases such as nitrogen, oxygen and carbon monoxide and dioxide, propylene, water and reaction by-products such as acrylic acid, acetic acid and formic acid, formaldehyde, acetaldehyde, allyl alcohol and polymers resulting from the degradation of acrolein and of acrylic acid in particular. With a view to using the acrolein in subsequent reactions such as the direct synthesis of MTPA, the water, the acidic impurities, especially acrylic acid, and the heavy polymer reaction by-products must be at least partly separated from the complex gaseous mixture which is produced.

Direct synthesis of MTPA is described in patents USP 4,225,516 and USP 5,352,837 for example. The synthesis consists of reacting a purified gaseous mixture containing acrolein with a source of methyl

mercaptan in a solution of methylthiopropionaldehyde. The limits of water and of acids, particularly acrylic acid, in the purified gaseous mixture that can be employed are very important because they determine the performance of the reaction.

A number of methods for purifying acrolein are known and exploited.

A first method, according to USP 4,219,389, consists of extracting acids present in the gaseous mixture containing acrolein by absorption in an organic solvent (particularly 2-ethylhexanol or tributyl phosphate). However, this type of method has the disadvantage that the solvent which is used must be purified before being recycled into the process. This increases the cost of the process.

A second method of purifying acrolein, for example according to USP 3,433,840, consists of extracting acids present in the gaseous mixture containing acrolein by selective absorption with water and then recovering the acrolein present in the gaseous stream laden with water and non-condensables originating from the absorption stage by distilling the resulting liquid mixture to obtain an acrolein/water azeotrope. However, this type of process has the disadvantage of requiring large quantities of water for the absorption step. It also presents considerable risks of degradation of the acrolein and of polymerization during the distillation stage.

Another route has been developed with a view to the direct synthesis of MTPA. The gaseous mixture produced by the oxidation of propylene is first purified to remove as much of the acidic compounds as possible, as previously by absorption with water. After condensation of the water, acrolein is extracted from the residual gas by absorption in cold MTPA originating from an MTPA production reactor. The solution of acrolein in MTPA which is obtained is then introduced directly into the MTPA production reactor with methyl mercaptan. Such a process is described in USP 4,225,516. However, as preliminary purification of the starting mixture containing acrolein to remove the acids is still required, the disadvantages mentioned earlier are still encountered.

Another known process for purification of acrolein is described in EP-A-0,559,227. The process consists of cooling the reaction mixture in a cooling tower, where it is brought into contact with condensed liquid, an effluent gas containing predominantly non-condensables and acrolein being recovered at the top of the tower. Under the conditions described, where the condensate at the bottom of the tower is at a temperature of 35 to 50°C, the effluent gas at the top of the tower is at a temperature of 35 to 55°C and where the residence time of the condensate in the tower is short, purification of the reaction mixture results in absorption of a relatively large quantity of

acrolein in the condensate, corresponding to a content of approximately 3 % by weight.

The present invention seeks to overcome the disadvantages of the known processes and to provide a process for the purification of acrolein in a gaseous mixture of acrolein, water, acids and non-condensables, and thus to permit the direct use of acrolein, particularly in the synthesis of MTPA.

It is also an object of the present invention to produce acrolein such that the purification yield is as high as possible while reducing to a minimum the risk of degradation of the acrolein. Finally, the aim of the present invention is to avoid the fouling of equipment used in the process.

Accordingly, the present invention provides a process for the purification of acrolein present in a feed gas stream including acrolein and water, acids and inert gases, originating particularly from the gas-phase oxidation of propylene to acrolein, which process comprises, in a first stage, fractionating the feed gas stream into a gaseous effluent and a liquid stream in a cooling column operating such that the temperature of the liquid stream at the bottom of the column is lower than or equal to the condensation temperature of the feed gas stream, the difference in temperature not exceeding 20°C, preferably not exceeding 10°C, and then, in a second stage, condensing the gaseous effluent at a temperature that is lower than 20°C to

give a liquid fraction and a purified gaseous fraction.

The acids present in the feed gas stream are organic acids, usually acrylic acid, formic acid, acetic acid and maleic acid.

5 As used herein, the term "inert gases" is intended to mean all of the gaseous compounds which remain in the gaseous phase from the beginning to the end of the production process of the invention and which are found in the purified gaseous fraction after
10 the condensation stage. In this respect the inert gases in the mixture to be purified may, in what follows, be occasionally called "non-condensables", since they are not condensed under the temperature and pressure conditions used in the process of the invention. They
15 may be nitrogen, oxygen and other gases from air, carbon oxides and propylene.

By virtue of the process of the invention it is possible, at the end of the second stage, to obtain a purified gaseous fraction containing acrolein and
20 non-condensables, in which the weight content of water is lower than or equal to 2 % and the weight content of acids is lower than or equal to 100 ppm.

In the first stage according to the process of the invention the feed gas stream which typically
25 originates from the gas-phase oxidation of propylene is preferably cooled from its production temperature to a temperature of 100 to 200°C, and is introduced into the bottom part of the cooling column.

The feed stream of the cooling column usually contains between 10 and 15 % by weight of acrolein and approximately 20 to 30 % by weight of water and less than 5 % by weight of acrylic acid, preferably less than 2 % by weight, the remainder consisting of non-
5 condensable gases and of various organic constituents produced in the oxidation of propylene.

The circulation of the gaseous stream in the column countercurrentwise to a cold liquid results in
10 the condensation of the water and of the acrylic acid and of the other condensable components that may be present. The condensed liquid flows back down under gravity to the bottom of the column. The gases at the top of the column are depleted in impurities. They
15 consist essentially of acrolein and of non-condensable gases.

The temperature of the gases at the top is preferably 30 to 60°C and still more preferably between 50 and 60°C.

20 The temperature of the liquid stream at the bottom of the column is less than 20°C, preferably less than 10°C, lower than the condensation temperature of the feed gas stream. Preferably, the temperature of the liquid stream at the bottom of the column is
25 substantially equal to the condensation temperature of the gaseous mixture introduced into the column, to reduce to a minimum condensation of the acrolein and its degradation. In most cases it is lower than 100°C.

The condensation temperature of the gaseous mixture originating from the catalytic oxidation of propylene and containing approximately 10 to 15 % by weight of acrolein and 20 to 30 % by weight of water is between
5 70 and 90°C, at a pressure of approximately 1.2×10^5 Pa.

The cooling column preferably operates at a pressure of 10^5 to 3×10^5 Pa.

The residence time of acrolein in the cooling column is preferably from 5 to 10 minutes. At this
10 residence time the risk of degradation is limited while the impurities are removed efficiently using the process of the invention.

According to the process of the invention a portion of the liquid stream accumulated in the bottom
15 of the cooling column is preferably removed, optionally cooled, and recycled as circulating cold liquid stream in the cooling column. A compound which inhibits the polymerization of acrolein and of acrylic acid may be added to the recycled liquid stream. The polymerization
20 inhibitor may be chosen from hydroquinone and phenothiazine and its derivatives.

The recycled stream generally contains organic acids, including acrylic acid, and less than
2 %, preferably less than 1.5 %, by weight of acrolein
25 and at least 90 % by weight of water.

The recycled liquid stream may be introduced into the cooling column in one or more places. It is preferably injected at the top of the column to spray

the gaseous mixture to be treated. More particularly, the recycled liquid is cooled to between 15 and 45°C by a cooler using industrial water or a cooler using water containing glycol.

5 According to an optional stage of the process of the invention, another portion of the liquid stream accumulated at the bottom of the cooling column may be removed to undergo a stripping operation to recycle to the bottom of the column a portion of the acrolein
10 entrained in gaseous form. The liquid stream removed for this purpose is preferably heated to a temperature of 90 to 120°C before the stripping.

 The stripping operation is carried out by circulating the liquid stream removed in a column,
15 countercurrentwise to an inert gas, preferably nitrogen, the liquid being introduced at the top of the column and the gas being introduced into the bottom part of the column. The temperature of the stripping gas is advantageously from 130 to 160°C, preferably
20 approximately 150°C, in order to eliminate any risk of degradation of the acrolein and to improve stripping efficiency.

 During the operation acrolein present in the liquid stream is extracted by the stripping gas, with
25 the result that a gaseous stream is recovered containing acrolein, a small portion of water and non-condensables. Most of the acids and water remain in the liquid phase.

The pressure in the stripping column is preferably higher than the pressure in the cooling column to allow the gaseous stream produced by stripping to be reinjected into the cooling column with the feed gas stream. It should be noted that the pressure in the stripping column must not be too high otherwise stripping efficiency is impaired.

Return of the gaseous stream produced by the stripping operation may be carried out by mixing the stripped stream with the feed gas stream or, preferably, by injection of the stripped stream into the cooling column, approximately at the same height as the feed gas stream entry.

In the second stage of the process according to the invention, the gas leaving the top of the cooling column is partly condensed, preferably in a surface condenser. Separation of the condensed liquid phase provides a gas which is depleted in impurities, especially in water and in acids, and containing acrolein and non-condensables.

The gaseous effluent is advantageously condensed once or several times in succession to give a gaseous fraction containing acrolein, the temperature of the gas leaving the last condenser being lower than 20°C. Condensation of the gaseous effluent is preferably carried out in the presence of an acrolein polymerization inhibitor.

At the end of the condensation operation(s)

the gaseous fraction containing acrolein may be recovered as purified gas, which may be used directly in the synthesis of MTPA.

According to an optional additional stage,
5 the gaseous fraction may preferably be subjected to an absorption operation by circulation countercurrentwise to water, to further remove additional residual acids. This operation may be carried out in an absorption column. The water circulates countercurrentwise to the
10 gaseous fraction at a temperature that is lower than the temperature of the fraction, with a mass flow rate such that the ratio of the mass flow rate of water to the mass flow rate of the gaseous fraction is between 0.005:1 and 0.05:1, preferably between 0.01:1 and
15 0.05:1. A purified gas is thus obtained in which the water content is lower than 2 % by weight and preferably lower than or equal to 1 % by weight, and the acid content is lower than 100 ppm.

The ratio ranges of water mass flow rate to
20 the mass flow rate of the gaseous fraction are expressed for an absorption performed in a column comprising 5 to 10 theoretical plates. The use of a column which has a number of plates outside the values referred to is possible. In this case a person skilled
25 in the art is capable of adapting the water and gaseous fraction flow rates accordingly.

Also, in order to increase the efficiency of absorption in water, and hence to conform to the

desired water and acid contents in the purified gas, it is preferred that the gas should be as cold as possible. Consequently, it is preferred to supercool the gas in the condensation operation.

5 A compound of the type described above which inhibits the polymerization of acrolein may be added to the water for absorption. However, during the absorption operation, dissolving chemical species in the water has the effect of reheating the gas leaving
10 the absorption column.

 A water content in the purified acrolein-containing gas of lower than 2 % by weight, and a total acid content of lower than 100 ppm, are values which are preferred for the use of the gas as starting
15 material for the direct synthesis of MTPA.

 Another embodiment of the present invention is a plant intended for implementing the process of the invention as described above. This plant includes a cooling column supplied with a feed gas stream by means
20 for feeding gas, a condensation unit provided with means for feeding gas from the top of the cooling column and, optionally, a stripping column fed at the top by means for feeding liquid from the bottom of the cooling column and means for recirculating the gas from
25 the top of the stripping column to the means for feeding gas or to the lower part of the cooling column.

 The plant preferably additionally includes means for circulating liquid from the bottom of the

cooling column to one or more points of the upper part of the cooling column, after the liquid has been cooled by means of a heat exchanger.

When the plant includes a stripping column, a
5 preheater is preferably installed upstream of the column to raise the temperature of the liquid to be stripped.

An example of an embodiment of the invention will now be described with reference to Figures 1, 2
10 and 3 of the attached drawings, which show, respectively, three embodiments of a plant in accordance with the invention for purification of a gaseous stream containing acrolein.

The plant shown in Figure 1 is intended for
15 the purification of a gaseous mixture originating from a unit 30 for the gas-phase oxidation of propylene and for producing a gaseous stream containing purified acrolein which feeds an MTPA synthesis unit 40. The plant essentially comprises a cooling column 1, a
20 condensation unit 3 made up of two separate condensers 3A and 3B, a cooler 7, a heater 14 and a stripping column 4, the top of which is connected via a gas delivery conduit 6 to the bottom part of the cooling column 1.

25 The plant operates in the following manner.

The feed gas stream produced at 30 is first cooled to a temperature, for example, of 100 to 200°C in a heat exchanger 31.

The cooled feed gas is then introduced via a feed conduit 2 into the lower part of the cooling column 1. The latter may be a cooling column of any known type, for example a packed column, it being possible for the packing to be chicanes or trays.

Feeding with the gaseous stream to be purified is preferably performed in the lower quarter of the column, whereas a fraction of the condensed liquid is introduced at the top of the column. The latter advantageously operates at a pressure of 10^5 to 3×10^5 Pa.

The cooling of the gas in the column 1 produces at the top of the column gases whose temperature is usually between 30 and 60°C and preferably between 50 and 60°C, while a liquid stream whose temperature corresponds to the condensation temperature of the feed gas stream accumulates at the bottom of the column.

The sizing of the column 1 is carried out according to the knowledge of a person skilled in the art as a function of these operating parameters. The height thus depends on the heat load to be removed and the difference in temperature between the condensed liquid fed at the top of the column and the liquid stream deposited at the bottom and the diameter, on the speed of the gases in the column.

The gases from the top of the column 1 are conveyed towards the condensation unit 3 which, in the

embodiment shown in Figure 1, comprises two separate condensers 3A and 3B in series. In fact, the condensation unit 3 may be made up of a variable number of condensers; a single condenser may even be
5 sufficient. In general, the choice of the number of condensers depends on the difference in temperature between the gases from the top of column 1 and the gas which feeds the MTPA synthesis unit, and on the area of the heat exchange units. A surface condenser, or
10 condensers, is/are preferably employed.

Gas cooled in the condenser 3A and separated off on leaving the latter, depleted in impurities such as water and acrylic acid, is conveyed towards the entry of the condenser 3B, while the condensed liquid
15 containing water and particularly acrylic acid, separated off on leaving the condenser 3A, is removed via a conduit 12A. On leaving the condenser 3B, gas separated off, further depleted in impurities such as water and acrylic acid, is conveyed in a conduit 16
20 towards the unit 40, while the liquid condensed in 3B, containing water and particularly acrylic acid, is removed via a conduit 12B.

To cool the gases from the top of the column 1 from their column top temperature (30 to 60°C,
25 preferably 50°C to 60°C) to a temperature that is lower than 20°C, it is preferable that the condenser 3A should operate with a conventional cooling liquid such as industrial water, and that the condenser 3B should

operate with a more complex cooling liquid such as brine (water and calcium chloride) or else water containing glycol.

Although polymerization of acrolein is not
5 very fast at the low temperature of the condensers, it is preferable to reduce the residence time of acrolein in the condensers to a minimum. To this end it is advantageous to employ vertical tubular condensers.

According to an alternative form, not shown
10 in Figure 1, the condensers 3A and 3B may be equipped with means for introducing a compound which inhibits the polymerization of acrolein, such as hydroquinone.

In the embodiment shown in Figure 1, the conduits 12A and 12B removing the liquids produced
15 during the condensation are connected to the upper part of the column 1, at a point 15, in order to introduce condensed liquid, typically containing water and acrylic acid. The point 15 is preferably situated in the upper quarter of the column. Circulation of the
20 condensed liquid, towards the bottom of the cooling column 1, in intimate contact with the gaseous stream rising in the column, provides some of the cooling of the latter.

The liquid stream accumulated at the bottom
25 of the cooling column 1 essentially contains impurities such as water, acrylic acid and heavy condensibles, but also contains a portion of acrolein from the feed gas stream. Some condensed liquid is therefore removed

from the bottom of the column 1 in a liquid delivery conduit 5, connected to the top of the stripping column 4.

In the embodiment shown in Figure 1 a portion
5 of this condensed liquid stream is removed at a point 9 of the conduit 5, via an abstraction conduit 8. This conduit feeds the entry of a cooler 7 to cool the bottom liquid from its initial temperature to a temperature of 15 to 45°C. The liquid cooled in 7 is
10 reinjected into the cooling column 1 via a conduit 10 at a point 11. Point 11 is preferably situated in the upper part of the column 1, preferably in the upper quarter. Point 11 is advantageously close to the abovementioned point 15. The cooler 7 may be chosen
15 from any of the known types of heat exchangers.

The other portion of the liquid stream removed at the bottom of the column 1 is conveyed via the conduit 5 to the stripping column 4. The stripping column is a packed column of any known type. A column
20 containing packing or a column containing trays may be employed. The conduit 5 injects the liquid to be stripped into the upper part of the stripping column 4. An "inert gas", such as nitrogen, injected into the lower part of the column 4 via the conduit 22 is
25 employed as stripping gas. This gas is preferably preheated to a temperature of 130 to 160°C, preferably approximately 150°C, to improve the stripping efficiency.

In the embodiment shown in Figure 1 a heat exchanger 14 is installed at an intermediate point of the conduit 5 upstream of the column 4 and downstream of the point 9. This exchanger functions as a heater to
5 raise the temperature of the liquid mixture to be stripped to a value of the order of 90 to 120°C, with the aim of improving the efficiency of the stripping stage.

The inert stripping gas desorbs acrolein from
10 the liquid mixture as it rises in the stripping column 4, with the result that a gaseous fraction containing water, non-condensables and acrolein is recovered at the top of this column. This gaseous fraction is reinjected into the lower part of the cooling column 1,
15 via the gas delivery conduit 6, preferably into the bottom quarter of the column 1. The injection of gas via the conduit 6 is preferably performed at the same height as the injection of the feed gas stream via the conduit 2.

20 In an alternative form, the conduit 6 may be connected to the conduit 2 upstream of the entry of the cooling column 1, in order to mix the gaseous fraction produced by the stripping with the gaseous stream to be purified.

25 To permit the gases recovered at the top of the stripping column 4 to return into the cooling column 1, the pressure in the column 4 is advantageously kept higher than the pressure prevailing

in the column 1. The pressure in the column 4 is preferably slightly higher than the pressure prevailing in the column 1.

The cooling column 1 is endowed with a system
5 for drawing off liquid from the bottom of the column, intended to keep the level of liquid constant at the bottom of the column 1. Any known means for drawing off liquid may be employed, particularly means 12 for controlling the liquid level at the bottom of the
10 column 1 and a valve 13 as shown in Figure 1. Opening of the valve 13 enables the surplus of liquid accumulated at the bottom to be removed via the conduit 5, towards the stripping column 4 or the cooler 7.

The plant shown in Figure 2 is an alternative
15 form of the plant shown in Figure 1, from which it differs in the following ways.

The gas delivery conduit 16 is connected to a liquid/gas absorption column 17. This column is a packed column which may be of any known type,
20 especially a column containing packing or a column containing trays.

Conduit 16 allows gas depleted in impurities, originating from the condensation unit 3, to be injected into the lower part of the column 17, while
25 the latter is fed in its upper part via a water delivery conduit 18. The water travelling countercurrentwise absorbs the last traces of acid, usually acrylic acid. This water, which is collected

at the bottom of the column 17, may be reinjected into the upper part of the column 1, as cooling liquid. This water, laden with traces of acid, is preferably conveyed via the conduit for removing liquid 23 towards
5 a point 24 of the column 1, advantageously situated above the point 15. The temperature of the water introduced into the column 17 is advantageously lower than 20°C.

Water delivered by the conduit 18 preferably
10 contains a compound which inhibits the polymerization of acrolein. The compound is added to the water upstream of the absorption column 17.

The plant shown in Figure 3 is an alternative form of the plant shown in Figure 2, in which the
15 layout of the units is slightly different.

Thus the plant includes a column 25 enclosing a cooling column 19 in its lower part, a condensation unit 3 in its intermediate part and an absorption column 20 in its upper part.

20 The cooling column 19 is a cooling column which is identical with the cooling column 1, the top of which is in direct communication with the entry of the condensation unit 3. The gaseous effluent resulting from the fractionation in the cooling column 19 is thus
25 conveyed directly into the condensation unit 3, which may be made up of one or more condensers in series. The liquid condensed in the condensation unit 3 travels freely, descending into the lower part of the column

25, countercurrentwise to the feed gas stream, and accumulates in the bottom with the liquid stream condensed in the cooling column.

The gaseous fraction depleted in impurities leaving the condensation unit 3 enters directly into the upper part of the column 25, where the absorption column 20 is placed. The latter is similar in type to the absorption column 17, except that the injection of the gas no longer takes place via a gas delivery conduit.

The upper part of the absorption column 20 is fed with water via a water delivery conduit 18 connected to the upper part of the unit 25.

The water travels countercurrentwise to the gas rising in the absorption column and picks up the residual acidic impurities present in this gas. It then flows freely back down in the intermediate part and the lower part of the column 25 and accumulates at the bottom with the other liquid fractions.

The column 25 is advantageously endowed with means enabling a back-pressure to be created between the cooling column 19 and the upper section. A hydraulic ram may be installed for this purpose.

Each of the plants described above makes it possible to perform the purification of a mixture containing acrolein, water and acids such as acrylic acid, to supply a gaseous stream containing purified acrolein. Preferably, a purified gas is thus produced

which has a water content lower than 2 % by weight and a total acid content lower than 100 ppm, without fouling the equipment and while limiting the degradation of acrolein.

5 The following examples illustrate the present invention.

EXAMPLE 1

A plant of the type shown in Figure 1, including a condenser as condensation unit, is employed
10 for purifying a gaseous mixture originating from a reactor for vapour-phase oxidation of propylene to acrolein. The composition of the mixture is as follows.

(The portions are shown in relation to the weight of the mixture.)

15	Non-condensable gases (nitrogen, oxygen, propylene, carbon oxides)	63.3 %
	Water	21.0 %
	Acrolein	12.3 %
	Acrylic acid	1.4 %
20	Other (acetaldehyde, allyl alcohol, formaldehyde etc.)	2.0 %

The gaseous mixture to be purified is introduced at a temperature of 180°C at the bottom of a cooling column having 10 perforated trays, below the
25 final tray, at a mass flow rate of 20 kg/h, at a pressure of 1.2×10^5 Pa.

The liquid at the bottom of the column is maintained at a temperature of 70.3°C, while the

condensation temperature of the feed gas stream is 74.7°C (the difference being equal to 4.3°C). The feed gas contains 1.3 % by weight of acrolein.

The recycling of liquid from the bottom to the top of the column takes place at a flow rate of 80 kg/h, the heat exchanger supplying a recycled liquid at a temperature of 30°C.

Another portion of the liquid from the bottom of the column is conveyed into the nitrogen stripping column, filled with random bulk packing, operating at a temperature of 90°C with a stripping nitrogen mass flow rate of 0.6 kg/h. A liquid containing not more than 100 ppm of acrolein is recovered from it.

At the exit of the cooling column the top gases are cooled to 2°C in the condenser. The gaseous fraction recovered at the exit of the condenser is analysed to determine its acid and water contents.

The determination of the content of the organic acids is performed by ionic chromatography after bubbling the gas through water.

Water content determination is performed by the Karl Fischer method after bubbling the gas through n-propanol.

It is determined that the purified gas contains 96 ppm of acids and 0.34 % of water, relative to the total weight of the said gas.

EXAMPLE 2

A plant of the type shown in Figure 2,

including the various units of the plant of Example 1 and in addition an absorption column with six perforated trays is employed for purifying the same mixture as that in Example 1.

5 The operating parameters of Example 1 are retained, except that the condenser cools the gases from the top of the cooling column to 4°C.

 The gaseous fraction recovered on leaving the condenser is introduced at the bottom of the absorption
10 column at a flow rate of 16.2 kg/h. A stream of water circulates in this absorption column, introduced at 4°C above the first tray of the column, at a flow rate of 0.2 kg/h.

 As in Example 1, it is determined that the
15 gaseous stream which leaves the absorption column contains 0.43 % of water and less than 10 ppm of acids, relative to the total weight of the gaseous stream.

CLAIMS

1. Process for the purification of acrolein present in a feed gas stream, which process comprises, in a first stage, fractionating the feed gas stream
5 into a gaseous effluent and a liquid stream in a cooling column operating such that the temperature of the liquid stream present at the bottom of the column is lower than or equal to the condensation temperature of the feed gas stream, the difference in temperature
10 not exceeding 20°C and then, in a second stage, condensing the gaseous effluent at a temperature that is lower than 20°C to give a liquid fraction and a purified gaseous fraction.

2. Process according to claim 1, wherein
15 the feed gas stream originates from the gas-phase oxidation of propylene to acrolein.

3. Process according to claim 1, wherein the temperature of the liquid stream at the bottom of the cooling column is lower than or equal to the
20 condensation temperature of the feed gas stream, the difference not exceeding 10°C.

4. Process according to claim 1, wherein the feed gas stream is introduced into the cooling column at a temperature of 100 to 200°C, the gas
25 temperature at the top of the column being from 30 to 60°C and the temperature of the liquid stream being close to the condensation temperature of the feed gas stream.

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5. Process according to claim 1, wherein a portion of the liquid stream at the bottom of the cooling column is removed, optionally cooled, and recycled as circulating liquid stream in the cooling column.

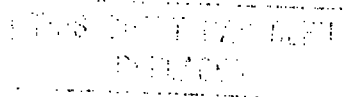
6. Process according to claim 5, wherein the temperature of the recycled liquid stream, before circulation in the cooling column, is from 15 to 45°C.

7. Process according to claim 1, wherein the purified gaseous fraction is absorbed by countercurrentwise circulation of the gaseous fraction with water at a mass flow rate of water such that the ratio of the mass flow rate of water to the mass flow rate of the gaseous fraction is between 0.005:1 and 0.05:1, the water temperature being lower than the temperature of the gaseous fraction.

8. Process according to claim 1, wherein a portion of the liquid stream at the bottom of the cooling column is stripped using a stripping gas to provide a stripped gaseous stream containing acrolein, which is introduced into the cooling column with the feed gas stream.

9. Process according to claim 8, wherein stripping takes place by heating the liquid stream to a temperature of 90 to 120°C and introducing it into a column where it is circulated countercurrentwise to the stripping gas.

10. Process according to claim 9, wherein



the temperature of the stripping gas is from 130 to 160°C.

11. Plant for purification of acrolein present in a feed gas stream, which plant comprises a cooling column (1) having means (2) for supplying a feed gas stream and a condensation unit (3,3A,3B) provided with means for feeding gas from the top of the cooling column (1).

12. Plant according to claim 11, wherein the feed gas stream originates from the gas-phase oxidation of propylene to acrolein.

13. Plant according to claim 11, comprising means (8,10) for circulating liquid from the bottom of the cooling column (1) to a point (11) at the upper part of the column (1), and optionally means (7) for cooling the circulating liquid.

14. Plant according to claim 11, wherein the cooling column (1) is equipped with means (12) for controlling the level of liquid at the bottom of the column (1), by which a draw-off valve (13) is servo-controlled.

15. Plant according to claim 11, additionally comprising a stripping column (4) fed at the top by means (5) for feeding liquid from the bottom of the cooling column (1) and means (6) for circulating gas from the top of the stripping column (4) to the means (2) or to the lower part of the cooling column (1).

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16. Plant according to claim 15, comprising a heat exchanger (14) installed at an intermediate location of the feeding means (5), upstream of the stripping column (4).

5 17. Plant according to claim 11, comprising a liquid/gas absorption column (17) fed in its lower part by means (16) for feeding gas leaving the condensation unit (3) and in its upper part by a water delivery conduit (18).

10 18. Plant according to claim 11, comprising means (12A,12B) for introduction of liquid condensed in the condensation unit (3,3A,3B) into the upper part of the cooling column (1), at a point (15) of the latter close to a point (11) at the upper part of the column
15 (1).

19. Plant according to of claim 11, wherein the condensation unit (3) is placed in the upper part of a cooling column (19), inside a single column (25).

20 20. Plant according to claim 11, wherein the condensation unit (3) is placed in the upper part of a cooling column (19) and an absorption unit (20) is placed above the condensation unit (3), inside a single column (25).

ABSTRACT

PROCESS AND PLANT FOR PURIFICATION
OF A GAS STREAM CONTAINING ACROLEIN

In a first stage a feed gas stream containing
5 acrolein originating for example from the gas-phase
oxidation of propylene to acrolein, is fractionated
into a gaseous effluent and a liquid stream in a
cooling column operating such that the temperature of
the liquid stream at the bottom of the column is lower
10 than or equal to the condensation temperature of the
feed gas stream, the difference in temperature not
exceeding 20°C and, in a second stage, the gaseous
effluent is condensed at a temperature that is lower
than 20°C, to give a liquid fraction and a purified
15 gaseous fraction.

FIGURE 1



FIG. 1

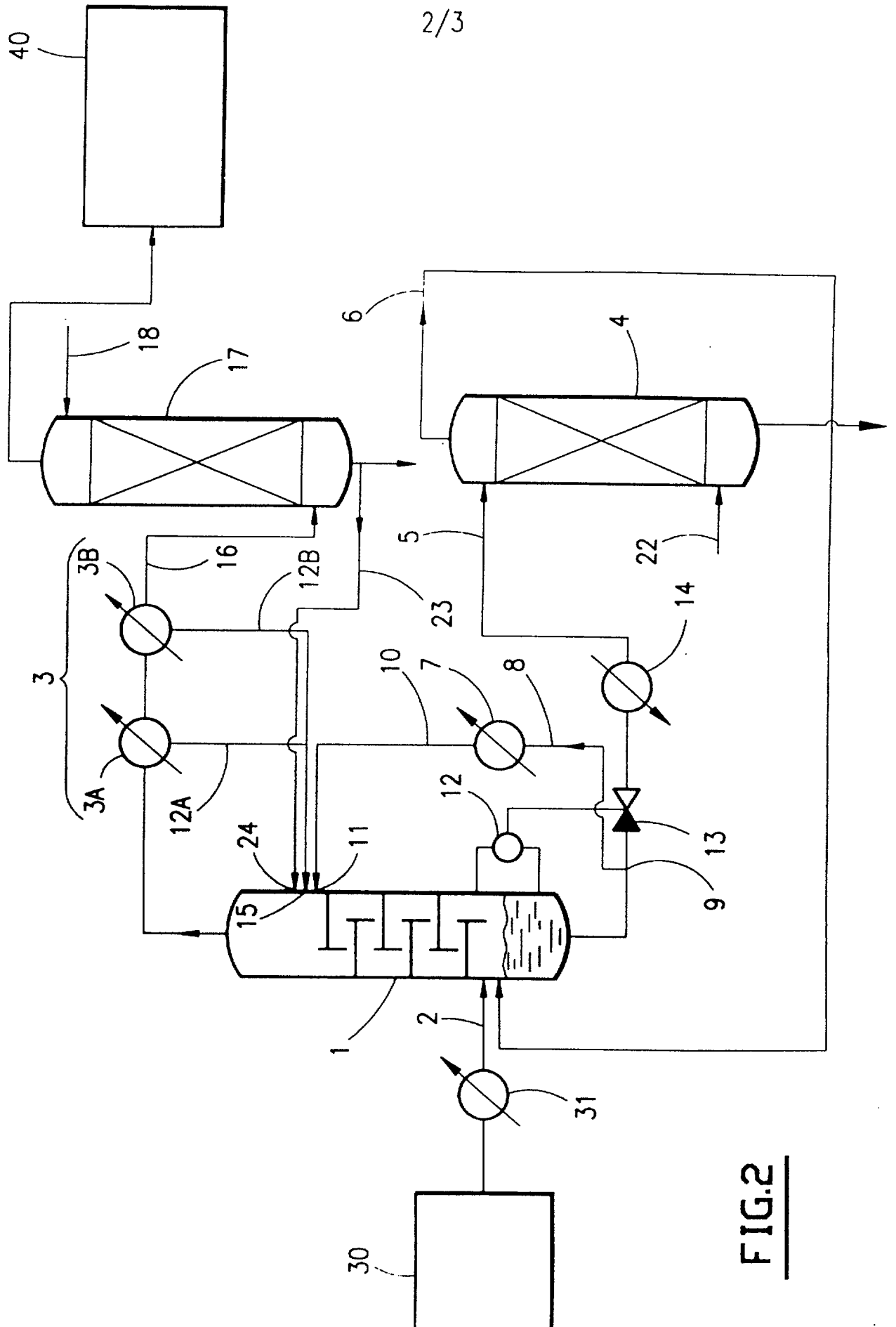
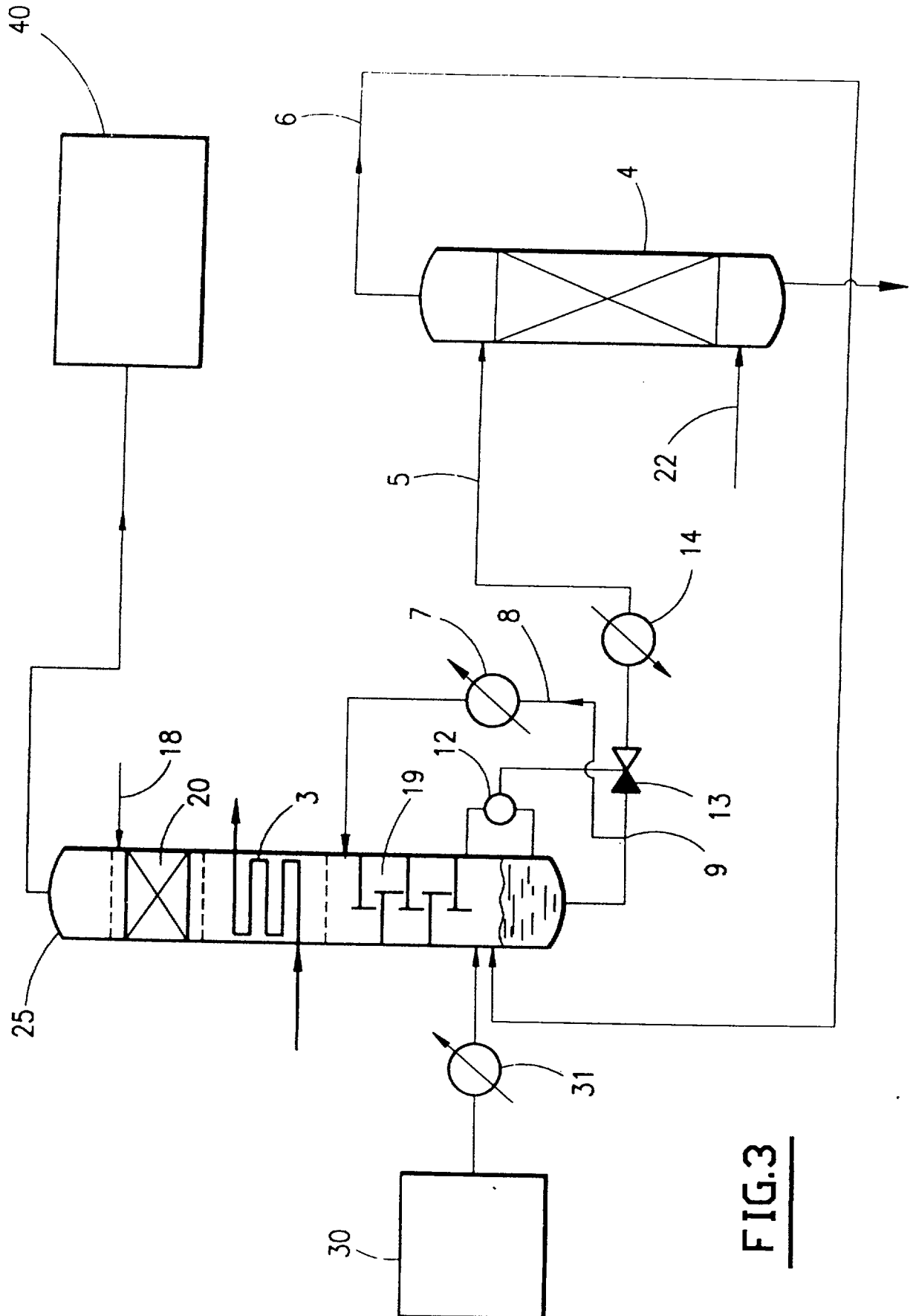
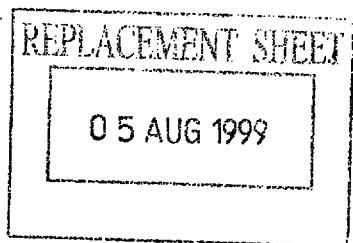


FIG. 2





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CLAIMS

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1. Process for the purification of acrolein present in a feed gas stream comprising acrolein and water, acids and inert gases, which process comprises, in a first stage, fractionating the feed gas stream into a gaseous effluent and a liquid stream in a cooling column operating such that the temperature of the liquid stream present at the bottom of the column is lower than or equal to the condensation temperature of the feed gas stream, the difference in temperature not exceeding 20°C and then, in a second stage, condensing the gaseous effluent at a temperature that is lower than 20°C to give a liquid fraction and a purified gaseous fraction.

2. Process according to claim 1, wherein the feed gas stream originates from the gas-phase oxidation of propylene to acrolein.

3. Process according to claim 1, wherein the temperature of the liquid stream at the bottom of the cooling column is lower than or equal to the condensation temperature of the feed gas stream, the difference not exceeding 10°C.

4. Process according to claim 1, wherein the feed gas stream is introduced into the cooling column at a temperature of 100 to 200°C, the gas temperature at the top of the column being from 30 to 60°C and the temperature of the liquid stream being close to the condensation temperature of the feed gas stream.

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the temperature of the stripping gas is from 130 to 160°C.

5 11. Plant when used for the purification of acrolein present in a feed gas stream comprising acrolein and water, acids and inert gases, which plant comprises a cooling column (1) having means (2) for supplying a feed gas stream and a condensation unit (3,3A,3B) provided with means for feeding gas from the top of the cooling column (1).

10 12. Plant according to claim 11, wherein the feed gas stream originates from the gas-phase oxidation of propylene to acrolein.

15 13. Plant according to claim 11, comprising means (8,10) for circulating liquid from the bottom of the cooling column (1) to a point (11) at the upper part of the column (1), and optionally means (7) for cooling the circulating liquid.

20 14. Plant according to claim 11, wherein the cooling column (1) is equipped with means (12) for controlling the level of liquid at the bottom of the column (1), by which a draw-off valve (13) is servo-controlled.

25 15. Plant according to claim 11, additionally comprising a stripping column (4) fed at the top by means (5) for feeding liquid from the bottom of the cooling column (1) and means (6) for circulating gas from the top of the stripping column (4) to the means (2) or to the lower part of the cooling column (1).