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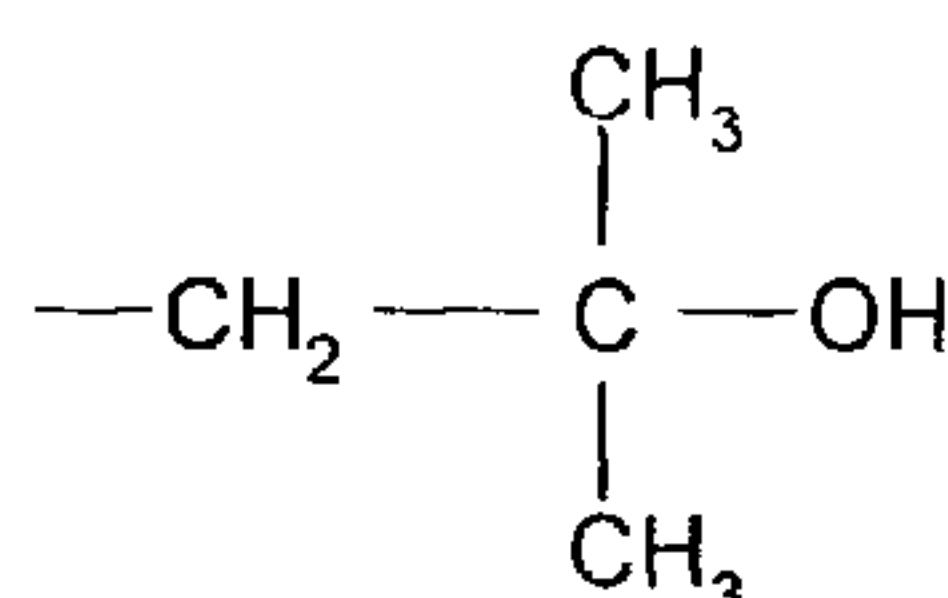
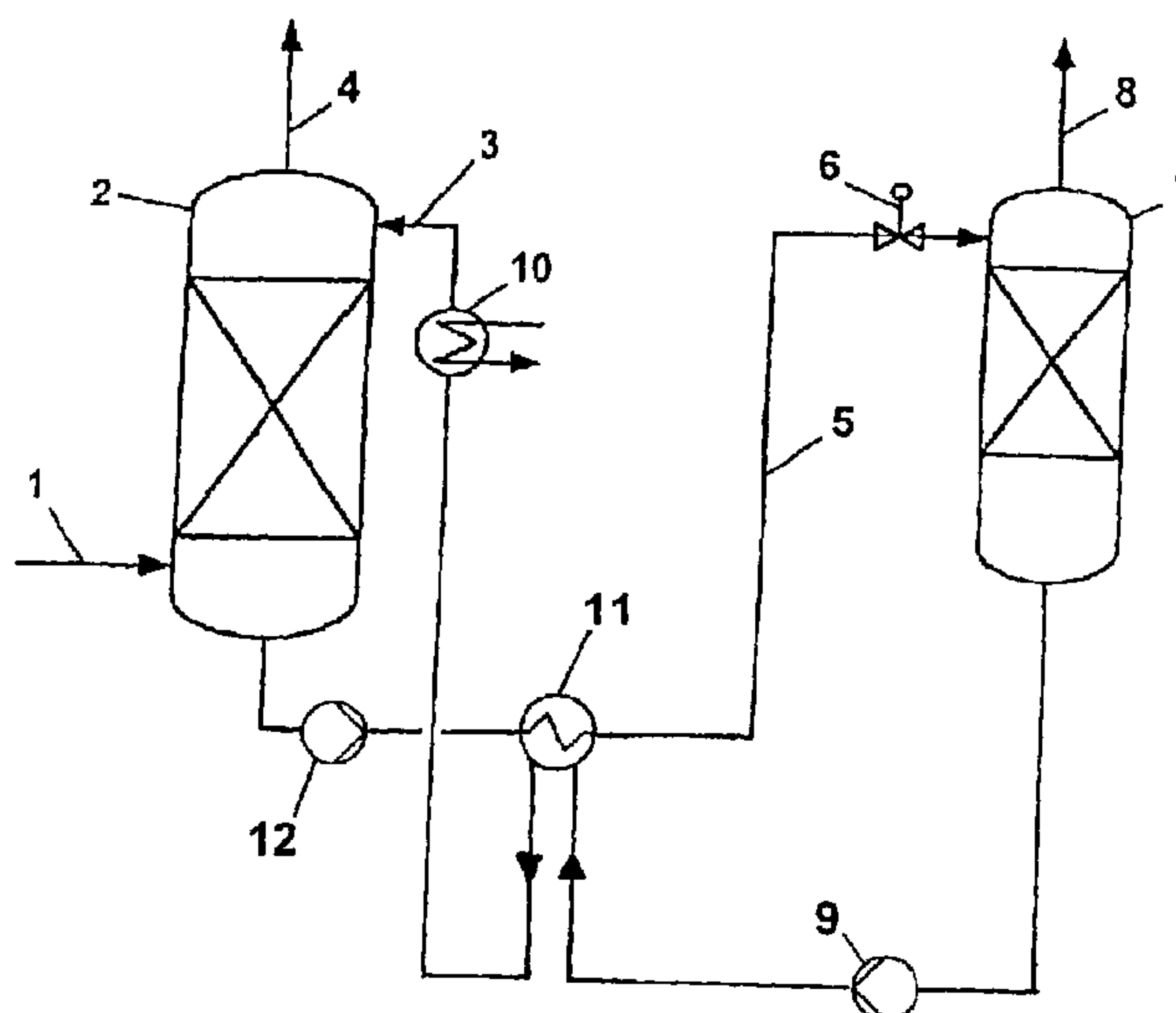
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(54) Title: ABSORBENT AND METHOD FOR THE ELIMINATION OF CARBON DIOXIDE FROM GAS FLOWS



(I)

(57) Abrégé/Abstract:

Disclosed is an absorbent for eliminating carbon dioxide from gas flows. Said absorbent contains an aqueous solution of an amine of formula (I) HNR_2 , wherein one or both radicals R represent (II) while the other radical R represents hydrogen. The inventive absorbent is characterized by particularly great resistance to oxidation.



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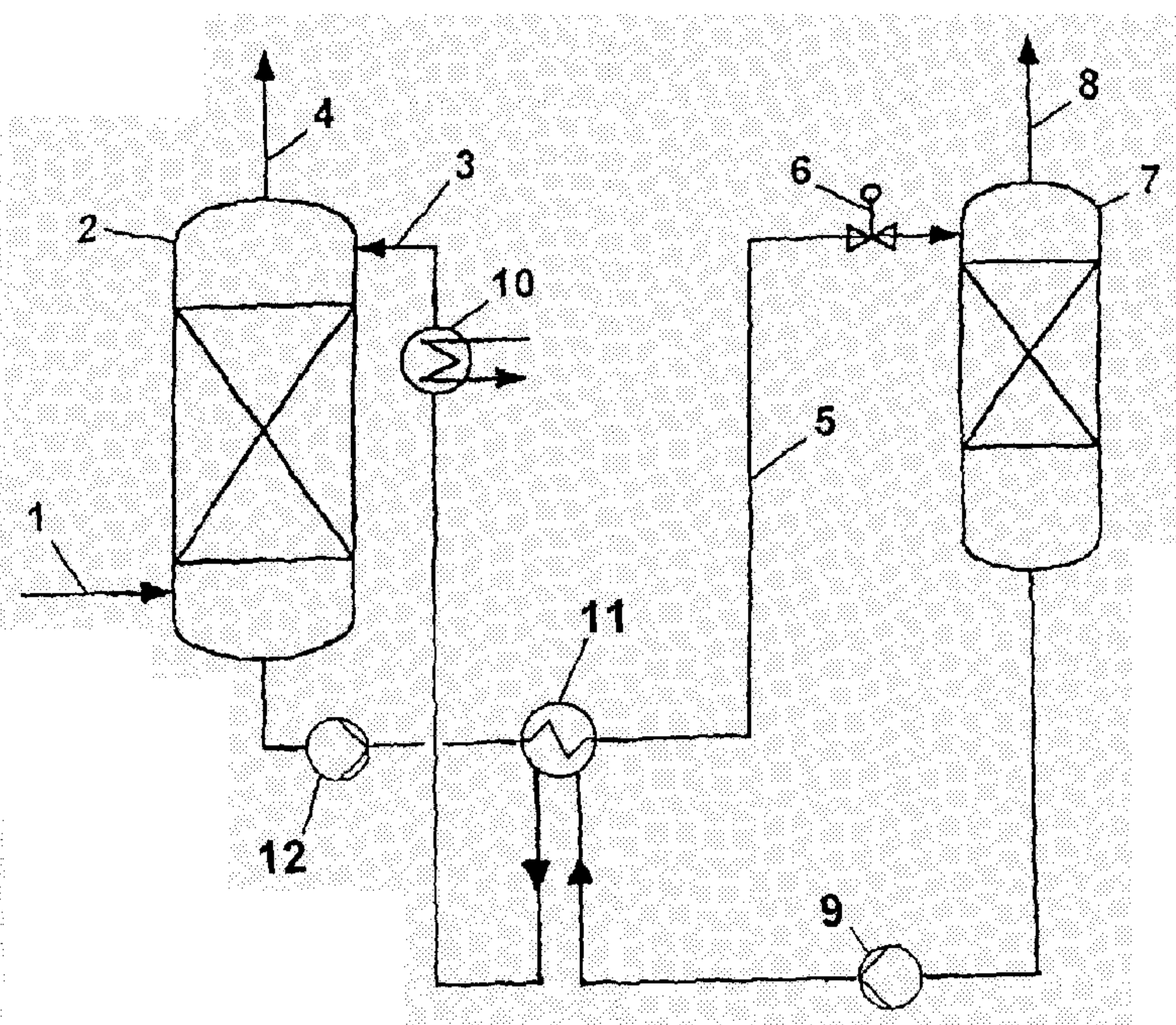
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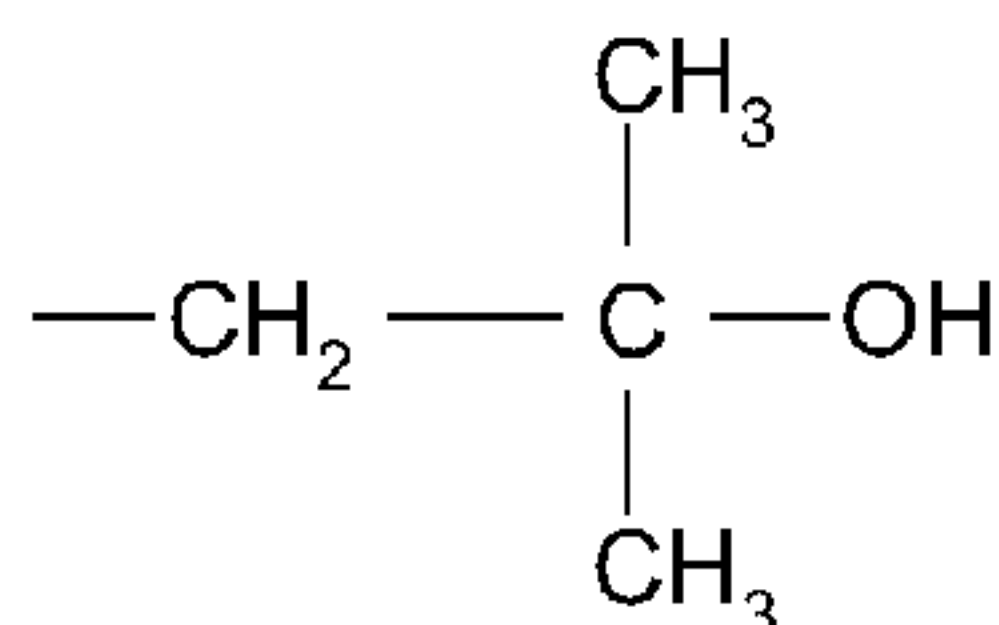
(54) Title: ABSORBENT AND METHOD FOR THE ELIMINATION OF CARBON DIOXIDE FROM GAS FLOWS

(54) Bezeichnung: ABSORPTIONSMITTEL UND VERFAHREN ZUM ENTFERNEN VON KOHLENDIOXID AUS GASSTRÖMEN



(57) Abstract: Disclosed is an absorbent for eliminating carbon dioxide from gas flows. Said absorbent contains an aqueous solution of an amine of formula (I) HNR_2 , wherein one or both radicals R represent (II) while the other radical R represents hydrogen. The inventive absorbent is characterized by particularly great resistance to oxidation.

(57) Zusammenfassung: Beschrieben wird ein Absorptionsmittel zum Entfernen von Kohlendioxid aus Gasströmen, enthaltend eine wässrige Lösung eines Amins der Formel (I) worin ein oder beide Reste R für stehen und der andere Rest R für Wasserstoff steht. Das Absorptionsmittel zeichnet sich durch besondere Oxidationsbeständigkeit aus.



(I)

WO 2007/045679 A1



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Zur Erklärung der Zweibuchstaben-Codes und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

ABSORBENT AND METHOD FOR THE ELIMINATION OF CARBON DIOXIDE FROM GAS FLOWS

Description

- 5 The present invention relates to an absorption medium for removing carbon dioxide from gas streams and to a method using the absorption medium.

10 In numerous processes in the chemical industry, fluid streams occur which comprise acid gases such as, for example, CO₂, H₂S, SO₂, CS₂, HCN, COS, NO_x, disulfides or mercaptans as impurities. These fluid streams can be, for example, gas streams, such as natural gas, synthesis gas, refinery gas, or reaction gases which are produced in the oxidation of organic materials, such as, for example, organic wastes, coal, natural gas or mineral oil, or in the composting of waste materials comprising organic substances.

- 15 The removal of acid gases is of particular importance for various reasons. For example, the content of sulfur compounds of natural gas must be reduced by suitable treatment measures directly at the natural gas source, since the sulfur compounds, together with the water frequently entrained by the natural gas, also form acids which have a corrosive action. For transport of natural gas in a pipeline, therefore preset limit values
20 of sulfur impurities must be maintained. The acid gases must be removed from the reaction gases produced in the oxidation of organic materials in order to prevent the emission of gases which can damage the natural environment or affect the climate.

On an industrial scale, frequently aqueous solutions of organic bases, e.g.
25 alkanolamines, are used as absorption media. When acid gases are dissolved, ionic products are formed from the base and the acid gas components. The absorption medium can be regenerated by expansion to a lower pressure or by stripping, the acid gases again being released and/or stripped off by steam. After the regeneration process, the absorption medium can be reused.

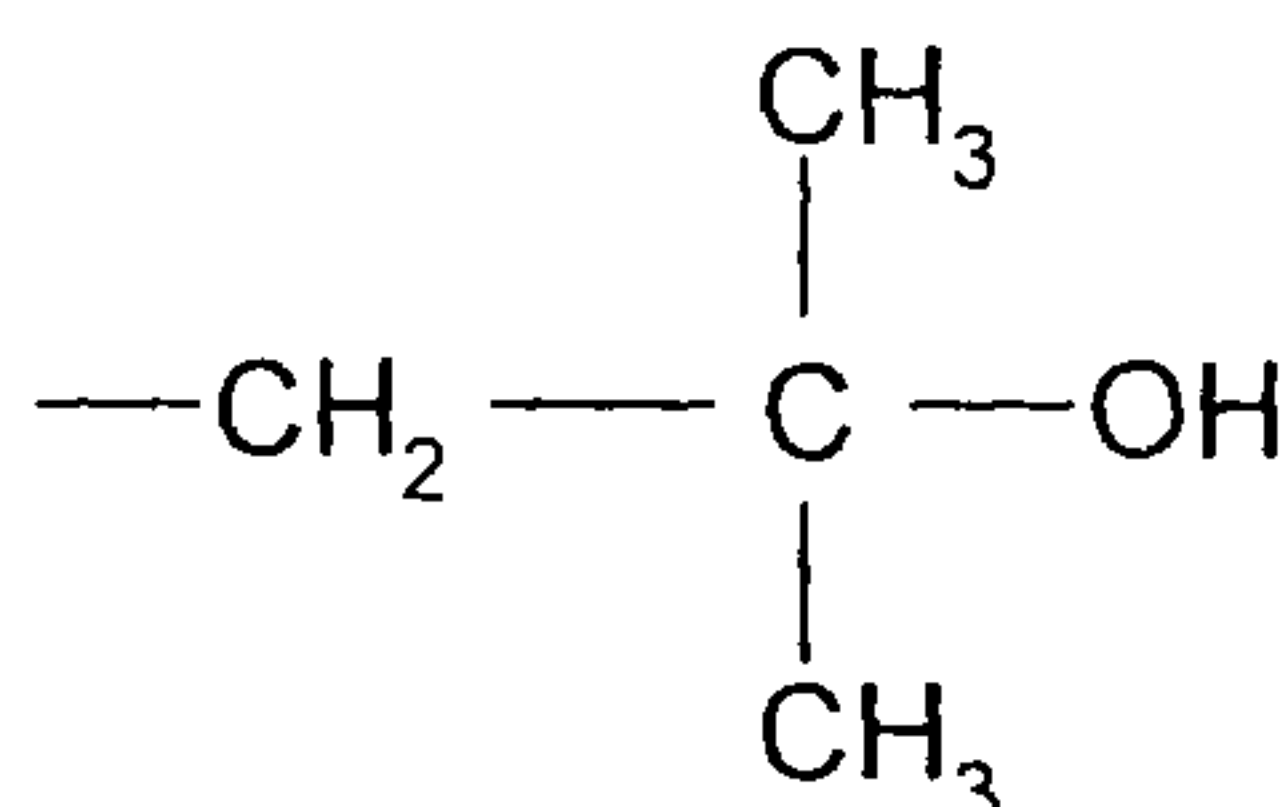
- 30 The known absorption media are very highly suitable for deacidifying hydrocarbons streams such as natural gas. Certain problems occur in the treatment of oxygen-containing fluids, e.g. flue gases. In this case the absorption capacity of the absorption medium worsens over the long term and is not completely recovered in the
35 regeneration. The presence of molecular oxygen is thought to be responsible for oxidative decomposition of the amines present in the absorption medium.

The object underlying the invention is to specify an absorption medium and a method for deacidifying fluid streams, the absorption capacity of the absorption medium being
40 retained in the long term.

The object is achieved by an absorption medium which comprises an aqueous solution of an amine of the formula I



where one or both radicals R are



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and the other radical R is hydrogen.

In one embodiment, the compound of the formula I is 1-amino-2-methylpropan-2-ol, (CAS 2854-16-2; hereinafter: 1A2MP). In another embodiment, it is amino-di-tert-butyl
15 alcohol. It has been found that both compounds exhibit surprisingly high oxygen stability.

The first step of the decomposition induced by molecular oxygen is presumably the abstraction of a hydrogen atom by the carbon atom in the α -position to the amino
20 group. Amines in which the α -carbon atoms solely bear substituents different from hydrogen are very resistant to oxidative decomposition. In 1A2MP, the carbon atom in the α -position to the amino group is a primary carbon atom, i.e. it bears two hydrogen atoms. Surprisingly, this structure, however, has been found to be highly resistant to the action of molecular oxygen. The stability to oxygen-induced decomposition is
25 particularly critical for flue gas scrubbers, so that the novel absorption medium here is a considerable improvement.

The inventive absorption medium, in addition to the amine of the formula I, can comprise one or more further amines, in order, for example, to optimize the loading
30 capacity of the absorption medium with carbon dioxide or other acid gas components, the mass-transfer rate for carbon dioxide or other acid gas components, and/or other factors. The other amine or amines customarily comprise 4 to 12 carbon atoms.

The absorption medium is usually present in the form of an aqueous solution having a
35 total amine content of generally 10 to 65% by weight, preferably 25 to 60% by weight. To the amine of the formula I is generally apportioned at least 1% by weight, preferably

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at least 5% by weight, based on the absorption medium.

In addition to water, the solutions can comprise physical solvents which are, for example, selected from cyclotetramethylene sulfone (sulfolane) and derivatives thereof, aliphatic acid amides (acetylmorpholine, N-formylmorpholine), N-alkylated pyrrolidones and corresponding piperidones, such as N-methylpyrrolidone (NMP), propylene carbonate, methanol, dialkyl ethers of polyethylene glycols and mixtures thereof.

The inventive absorption medium can comprise further functional components, such as stabilizers, in particular antioxidants, see, e.g., DE 102004011427. Addition of acids, such as phosphoric acid, formic acid or sulfonic acids, can be suitable for reducing the energy required for regeneration of the loaded absorption medium.

Suitable amines which can be present in the inventive absorption medium in addition to the amine of the formula I are selected, e.g., from

- (A) tertiary amines (this is taken to mean including tertiary monoamines or polyamines solely having tertiary amino groups);
 - (B) primary amines, where the amino group is bound to a tertiary carbon atom;
 - (C) secondary amines, where the amino group is bound to at least one secondary or tertiary carbon atom;
- and mixtures thereof.

Preferred amines of this type are selected from

- (A) tertiary amines having three hydroxyalkyl groups on the nitrogen atom, where the amino group is separated from the hydroxyl groups by at least two carbon atoms, such as triethanolamine (TEA);

tertiary amines having one or two hydroxyalkyl groups and two or one unsubstituted alkyl groups on the nitrogen atom, where the amino group is separated from the hydroxyl group(s) by at least two carbon atoms,

such as diethylethanolamine (DEEA), methyldiethanolamine (MDEA), 3-dimethyl-amino-1-propanol (DIMAP), dimethylethanolamine (DMEA), methyldiisopropanol-amine (MDIPA);

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diamines having two tertiary amino groups;

such as N,N,N',N'-tetramethylethylenediamine, N,N-diethyl-N',N'-dimethyl-ethylenediamine, N,N,N',N'-tetraethylethylenediamine, N,N,N',N'-tetramethyl-1,3-propanediamine and N,N,N',N'-tetraethyl-1,3-propanediamine and also bis(dimethylaminoethyl)ether.

- (B) primary amines having at least one hydroxyl group, where the amino group is bound to a tertiary carbon atom and is separated from the hydroxyl group by at least two carbon atoms,

such as 2-amino-2-methyl-1-propanol (AMP), 3-amino-3-methyl-2-pentanol, 2,3-dimethyl-3-amino-1-butanol, 2-amino-2-ethyl-1-butanol, 2-amino-2-methyl-3-pentanol, 2-amino-2-methyl-1-butanol, 3-amino-3-methyl-1-butanol, 3-amino-3-methyl-2-butanol, 2-amino-2,3-dimethyl-3-butanol, 2-amino-2,3-dimethyl-1-butanol and 2-amino-2-methyl-1-pentanol, of which 2-amino-2-methyl-1-propanol is preferred;

- (C) secondary amines having at least one hydroxyl group, where the amino group is bound to at least one secondary or tertiary carbon atom and is separated from the hydroxyl group by at least two carbon atoms,

such as 2-(isopropylamino)ethanol, 2(sec-butylamino)ethanol, 2-piperidine-ethanol;

and mixtures thereof.

Preferred absorption media comprise

- (i) 1 to 30% by weight, preferably 5 to 25% by weight, of amine of the formula I and
(ii) 10 to 60% by weight, preferably 15 to 50% by weight, of one or more amines (A) to (C), in each case based on the total weight of the absorption medium, with the proviso that the maximum total amine content of the absorption medium is 65% by weight.

In other embodiments, the inventive absorption medium, in addition to the amine of the formula I, comprises at least one amine which is selected from

- (D) primary or secondary alkanolamines;

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(E) alkylenediamines;

(F) polyalkylenepolyamines;

5 (G) amines of the general formula



where R^1 is C_1 - C_6 -alkyl and R^2 is C_2 - C_6 -alkylene;

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(H) cyclic amines having a 5-, 6- or 7-membered saturated ring which comprises an NH group and if appropriate a further heteroatom, in particular an oxygen or nitrogen atom;

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and mixtures thereof.

Particularly suitable amines of this type are selected from

20 (D) monoethanolamine (MEA), diethanolamine (DEA), diisopropylamine (DIPA), aminoethoxyethanol (AEE);

(E) hexamethylenediamine;

25 (F) diethylenetriamine; triethylenetetramine, 3,3-iminobispropylamines; preferably diethylenetriamine;

(G) 3-methylaminopropylamine (MAPA);

30 (H) piperazine, 2-methylpiperazine, N-methylpiperazine, N-ethylpiperazine, N-aminoethylpiperazine, homopiperazine, piperidine, morpholine;

and mixtures thereof.

Preferred absorption media comprise

35

(i) 20 to 60% by weight, preferably 25 to 50% by weight, of amine of the formula I and

40 (ii) 1 to 25% by weight, preferably 3 to 20% by weight, of one or more amines (D) to (H), in each case based on the total weight of the absorption medium, with the proviso that the maximum total amine content of the absorption medium is 65%

by weight.

The invention also relates to a method for removing carbon dioxide from a gas stream, the gas stream being brought into contact with a liquid absorption medium which
5 comprises an aqueous solution of an amine of the formula I.

In addition to carbon dioxide, other acid gases and/or their precursor compounds, such as, e.g., H_2S , SO_2 , CS_2 , HCN , COS , NO_x , disulfides or mercaptans, are partially or
10 completely removed from the gas stream.

The method is particularly suitable for gas streams in which the partial pressure of the carbon dioxide in the gas stream is less than 500 mbar, preferably less than 200 mbar, usually 20 to 150 mbar.

15 The gas stream is preferably a gas stream which is formed in the following manner:

- a) oxidation of organic substances, e.g. (flue gases),
- b) composting and storage of waste materials comprising organic substances, or
- 20 c) bacterial decomposition of organic substances.

The oxidation can be carried out with appearance of flame, i.e. as customary combustion, or as oxidation without flame appearance, e.g. in the form of a catalytic oxidation or partial oxidation. Organic substances which are subjected to combustion
25 are customarily fossil fuels such as coal, natural gas, mineral oil, gasoline, diesel, raffinates or kerosene, biodiesel or waste materials having a content of organic substances. Starting materials of the catalytic (partial) oxidation are, e.g., methanol or methane which can be reacted to form formic acid or formaldehyde.

30 Waste materials which are subjected to oxidation, composting or storage, are typically domestic refuse, plastic wastes or packaging refuse.

The combustion of organic substances usually proceeds in customary incineration plants with air. Composting and storage of waste materials comprising organic
35 substances generally proceeds in refuse landfills. The exhaust gas or the exhaust air of such plants can advantageously be treated by the inventive method.

As organic substances for bacterial decomposition, use is customarily made of stable manure, straw, liquid manure, sewage sludge, fermentation residues and the like.
40 Bacterial decomposition proceeds, e.g., in customary biogas plants. The exhaust air of

such plants can advantageously be treated by the inventive method.

The method is also suitable for treating the exhaust gases of fuel cells or chemical synthesis plants which make use of a (partial) oxidation of organic substances.

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In addition, the inventive method can of course also be used to treat unburnt fossil gases, such as natural gas, e.g. coal seam gases, i.e. gases produced in the extraction of coal; which are collected and compressed.

10 Generally, these gas streams, under standard conditions, comprise less than 50 mg/m³ of sulfur dioxide.

The starting gases can either have the pressure which roughly corresponds to the pressure of the ambient air, i.e. atmospheric pressure or a pressure which deviates
15 from atmospheric pressure by up to 1 bar.

Suitable apparatuses for carrying out the inventive method comprise at least one scrubbing column, e.g. random packing columns, ordered packing columns and tray columns, and/or other absorbers such as membrane contactors, radial stream
20 scrubbers, jet scrubbers, Venturi scrubbers and rotary spray scrubbers. Treatment of the gas stream by the absorption medium preferably proceeds in a scrubbing column in countercurrent flow. The gas stream is generally fed into the lower region and the absorption medium into the upper region of the column.

25 Suitable columns for carrying out the inventive method are also scrubbing columns made of plastic, such as polyolefins or polytetrafluoroethylene, or scrubbing columns whose inner surface is partly or completely lined with plastic or rubber. In addition, membrane contactors having a plastic housing are suitable.

30 The temperature of the absorption medium is generally about 30 to 70°C in the absorption step, then when a column is used, for example 30 to 60°C at the top of the column and 40 to 70°C at the bottom of the column. A product gas (byproduct gas) low in acid gas components, i.e. depleted in these components, and an absorption medium loaded with acid gas components are obtained.

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The carbon dioxide can be liberated from the absorption medium loaded with the acid gas components in a regeneration step, a regenerated absorption medium being obtained. In the regeneration step the loading of the absorption medium is decreased and the resultant regenerated absorption medium is preferably subsequently recycled
40 to the absorption step.

Generally, the loaded absorption medium is regenerated by

- a) heating, e. g. to 70 to 120°C,
- 5 b) expanding,
- c) stripping with an inert fluid

or a combination of two or all of these measures.

- 10 Generally, the loaded absorption medium is heated for regeneration and the liberated carbon dioxide is separated off, e.g. in a desorption column. Before the regenerated absorption medium is reintroduced into the absorber, it is cooled to a suitable absorption temperature. In order to utilize the energy present in the hot regenerated absorption medium, it is preferred to preheat the loaded absorption medium from the
- 15 absorber by heat exchange with the hot regenerated absorption medium. The heat exchange brings the loaded absorption medium to a higher temperature, so that in the regeneration step a lower energy usage is required. Via the heat exchange, a partial regeneration of the loaded absorption medium can already be performed with liberation of carbon dioxide. The resultant gas-liquid mixed phase stream is passed into a phase
- 20 separation vessel from which the carbon dioxide is taken off; the liquid phase is passed into the desorption column for complete regeneration of the absorption medium.

- Frequently, the carbon dioxide liberated in the desorption column is subsequently compressed and fed, e.g., to a pressure tank or to sequestration. In these cases, it can
- 25 be advantageous to carry out the regeneration of the absorption medium at a relatively high pressure, e.g. 2 to 10 bar, preferably 2.5 to 5 bar. The loaded absorption medium for this is compressed by means of a pump to the regeneration pressure and introduced into the desorption column. The carbon dioxide occurs in this manner at a higher pressure level. The pressure difference from the pressure level of the pressure
- 30 tank is less and under some circumstances a compression stage can be saved. A higher pressure during regeneration necessitates a higher regeneration temperature. At a higher regeneration temperature, a lower residual loading of the absorption medium can be achieved. The regeneration temperature is generally only limited by the thermal stability of the absorption medium.

- 35 Before the inventive absorption medium treatment, the flue gas is preferably subjected to a scrubbing using an aqueous liquid, in particular water, in order to cool the flue gas and moisten it (quench it). In the scrubbing, dusts or gaseous impurities such as sulfur dioxide can also be removed.

The invention will be described in more detail with reference to the accompanying drawings and the examples hereinafter.

Fig. 1 is a diagrammatic representation of a plant suitable for carrying out the inventive method.

According to fig. 1, a suitably pretreated carbon dioxide-containing combustion gas, in an absorber 2, is brought, via a feed line 1, into contact in countercurrent flow with the regenerated absorption medium which is fed via the absorption medium line 3. The absorption medium removes carbon dioxide by absorption from the combustion gas; via an exhaust gas line 7 a pure gas low in carbon dioxide is produced. The absorber 2 can have, above the absorption medium inlet, backwash trays or backwash sections which are preferably equipped with ordered packings (not shown), where, using water or condensate, entrained absorption medium is separated off from the CO₂-depleted gas. The liquid on the backwash tray is recycled in a suitable manner via an external cooler.

Via an absorption medium line 5, a pump 12, a solvent-solvent heat exchanger 11 in which the acid gas-loaded absorption medium is heated by the heat from the regenerated absorption medium coming from the bottom of the desorption column 7, and a throttle valve 6, the carbon dioxide-loaded absorption medium is passed to a desorption column 7. In the lower part of the desorption column 7 the loaded absorption medium is heated by means of a heater (not shown) and regenerated. The carbon dioxide liberated leaves the desorption column 7 via the exhaust gas line 8. The desorption column 7 can, above the absorption medium inlet, have backwash trays or backwash sections which are preferably equipped with ordered packings (not shown), where entrained absorption medium is separated off from the liberated CO₂ using water or condensate. In the line 8, a heat exchanger having a top distributor or condenser can be provided. The regenerated absorption medium is subsequently fed by means of a pump 9 via the solvent-solvent heat exchanger 11, in which the regenerated absorption medium heats up the acid gas-loaded absorption medium and is itself cooled, and back to the heat exchanger 10 of the absorption column 2. To prevent the accumulation of absorbed substances which are not expelled or are only incompletely expelled in the regeneration, or of decomposition products in the absorption medium, a substream of the absorption medium taken off from the desorption column 7 can be fed to an evaporator in which low-volatility byproducts and decomposition products are produced as residue and the pure absorption medium is taken off as vapors. The condensed vapors are fed back to the absorption medium circuit. Expediently, a base, such as potassium hydroxide, can be added to the substream which, e.g. together with sulfate ions or chloride ions, forms low-volatility

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salts which are taken off from the system together with the evaporator residue.

Examples

- 5 The resistance of various amines to the action of oxygen was determined as follows:

In a 100 ml flask, 5 l(S.T.P.) air/h were bubbled into about 60 ml of amine at 120°C for 6 days via a frit at atmospheric pressure. The vapor space above the solution was made inert by 10 l(S.T.P.) of N₂/h. The flask was equipped with a reflux condenser so
10 that matter stripped out was substantially condensed and recycled.

Samples of the fresh solution and the solution treated for 6 days were taken and analyzed by GC.

GC method: 30 m RTX-5 Amine, (0.32 mm, 1.5 µm), 50°C - 3 min - 7°C/min - 280°C -
15 20 min

Sample 1:

2-Amino-2-methylpropan-1-ol (comparison)

Concentration of the original solution: 93.5%

20 Concentration after 6 days of experiment: 93.4%

Sample 2:

1-Amino-2-methylpropan-2-ol

Concentration of the original solution: 99.8%

25 Concentration after 6 days of experiment: 98.2%

Sample 3:

Monoethanolamine (comparison)

Concentration of the original solution: 100%

30 Concentration after 6 days of experiment: 32.8%

Sample 4:

Methyldiethanolamine (comparison)

Concentration of the original solution: 99.3%

35 Concentration after 6 days of experiment: 72.4%

Sample 5:

Methylmonoethanolamine (comparison)

Concentration of the original solution: 99.6%

40 Concentration after 6 days of experiment: 75.5%

Patent claims

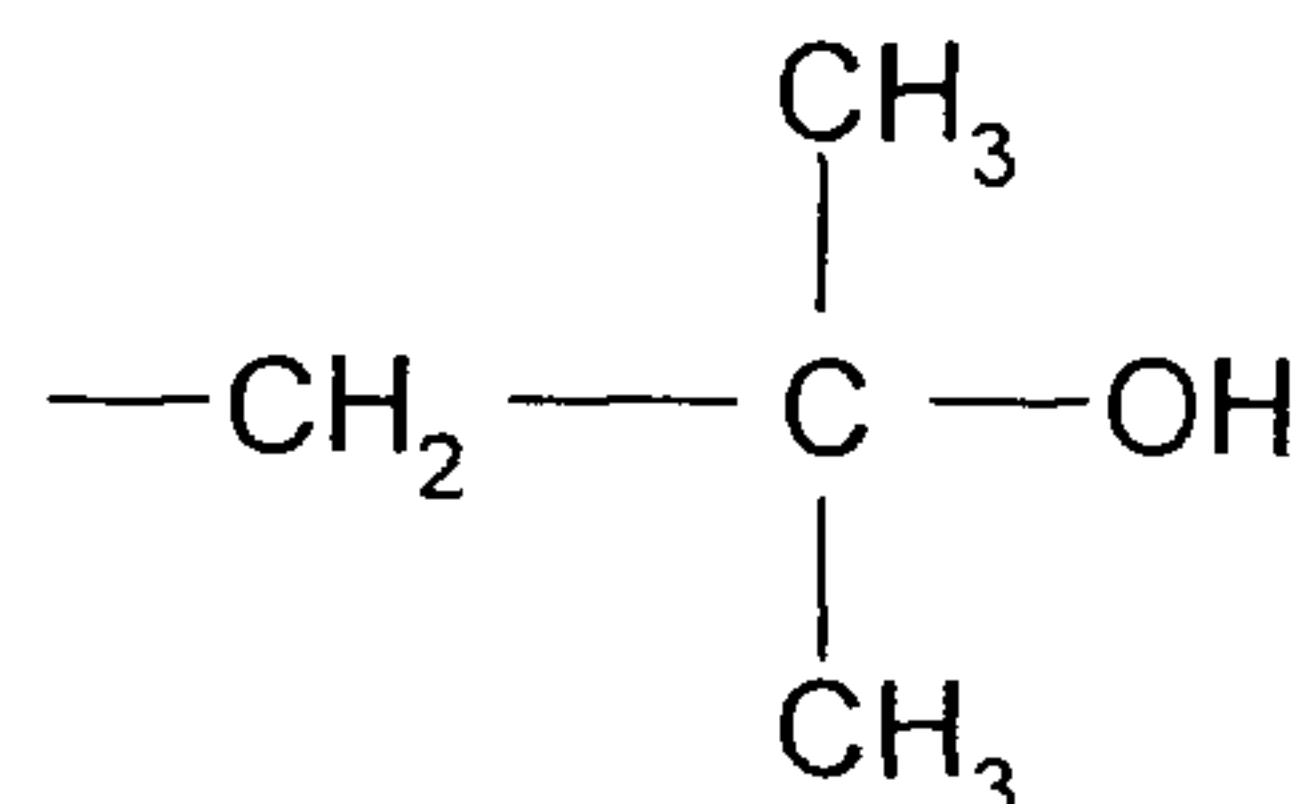
1. An absorption medium for removing carbon dioxide from gas streams which comprises an aqueous solution of an amine of the formula I

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where one or both radicals R are

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and the other radical R is hydrogen.

2. The absorption medium according to claim 1, in addition comprising at least one amine which is selected from

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- (A) tertiary amines;
 - (B) primary amines, where the amino group is bound to a tertiary carbon atom;
 - (C) secondary amines, where the amino group is bound to at least one secondary or tertiary carbon atom;
- and mixtures thereof.

20

3. The absorption medium according to claim 2, comprising at least one amine which is selected from
- (A) tertiary amines having one or two hydroxyalkyl groups and two or one unsubstituted alkyl groups on the nitrogen atom, where the amino group is separated from the hydroxyl group(s) by at least two carbon atoms;
 - (B) primary amines having at least one hydroxyl group, where the amino group is bound to a tertiary carbon atom and is separated from the hydroxyl group by at least two carbon atoms;
 - (C) secondary amines having at least one hydroxyl group, where the amino group is bound to at least one secondary or tertiary carbon atom and is separated from the hydroxyl group by at least two carbon atoms;
- and mixtures thereof.

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4. The absorption medium according to claim 2 or 3, comprising 1 to 30% by weight of amine of the formula I and 10 to 60% by weight of one or more amines (A) to (C), based on the total weight of the absorption medium, with the proviso that the maximum total amine content of the absorption medium is 65% by weight.

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5. The absorption medium according to claim 1, in addition comprising at least one amine which is selected from

- 5 (D) primary or secondary alkanolamines;
(E) alkylenediamines;
(F) polyalkylenepolyamines;
(G) amines of the general formula



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where R^1 is C_1 - C_6 -alkyl and R^2 is C_2 - C_6 -alkylene;

- (H) cyclic amines having a 5-, 6- or 7-membered saturated ring which comprises an NH group and if appropriate a further heteroatom, in particular an oxygen or nitrogen atom;
15 and mixtures thereof.

6. The absorption medium according to claim 4, comprising at least one amine which is selected from

- 20 (D) monoethanolamine, diethanolamine, methylethanolamine;
(E) hexamethylenediamine;
(F) diethylenetriamine;
(G) 3-methylaminopropylamine;
(H) piperazine, 2-methylpiperazine, N-methylpiperazine, N-ethylpiperazine, N-aminoethylpiperazine, homopiperazine, piperidine, morpholine;
25 and mixtures thereof.

7. The absorption medium according to claim 5 or 6, comprising 20 to 60% by weight of amine of the formula I and 1 to 25% by weight of one or more amines (D) to (H), based on the total weight of the absorption medium, with the proviso
30 that the maximum total amine content of the absorption medium is 65% by weight.

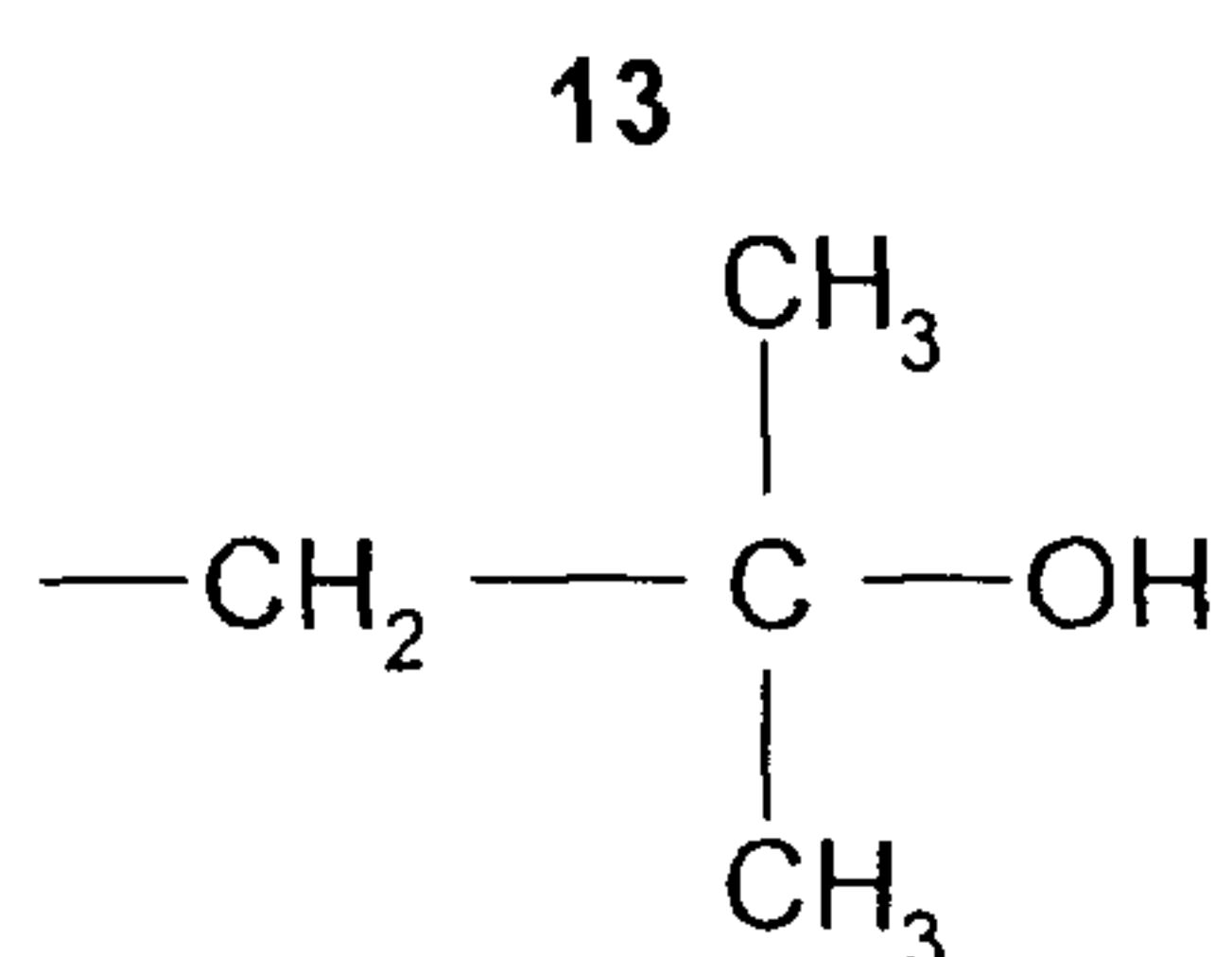
8. A method for removing carbon dioxide from a gas stream, the gas stream being brought into contact with a liquid absorption medium which comprises an
35 aqueous solution of an amine of the formula I,



(I)

where one or both radicals R are

40



and the other radical R is hydrogen.

- 5 9. The method according to claim 8, the partial pressure of the carbon dioxide in the gas stream being less than 500 mbar.
10. The method according to claim 8 or 9, the gas stream originating from
- 10 a) the oxidation of organic substances,
 b) the composting or storage of waste materials comprising organic substances, or
 c) the bacterial decomposition of organic substances.
- 15 11. The method according to one of claims 8 to 10, the loaded absorption medium being regenerated by
- a) heating,
 b) expanding,
20 c) stripping with an inert fluid

or a combination of two or all of these measures.

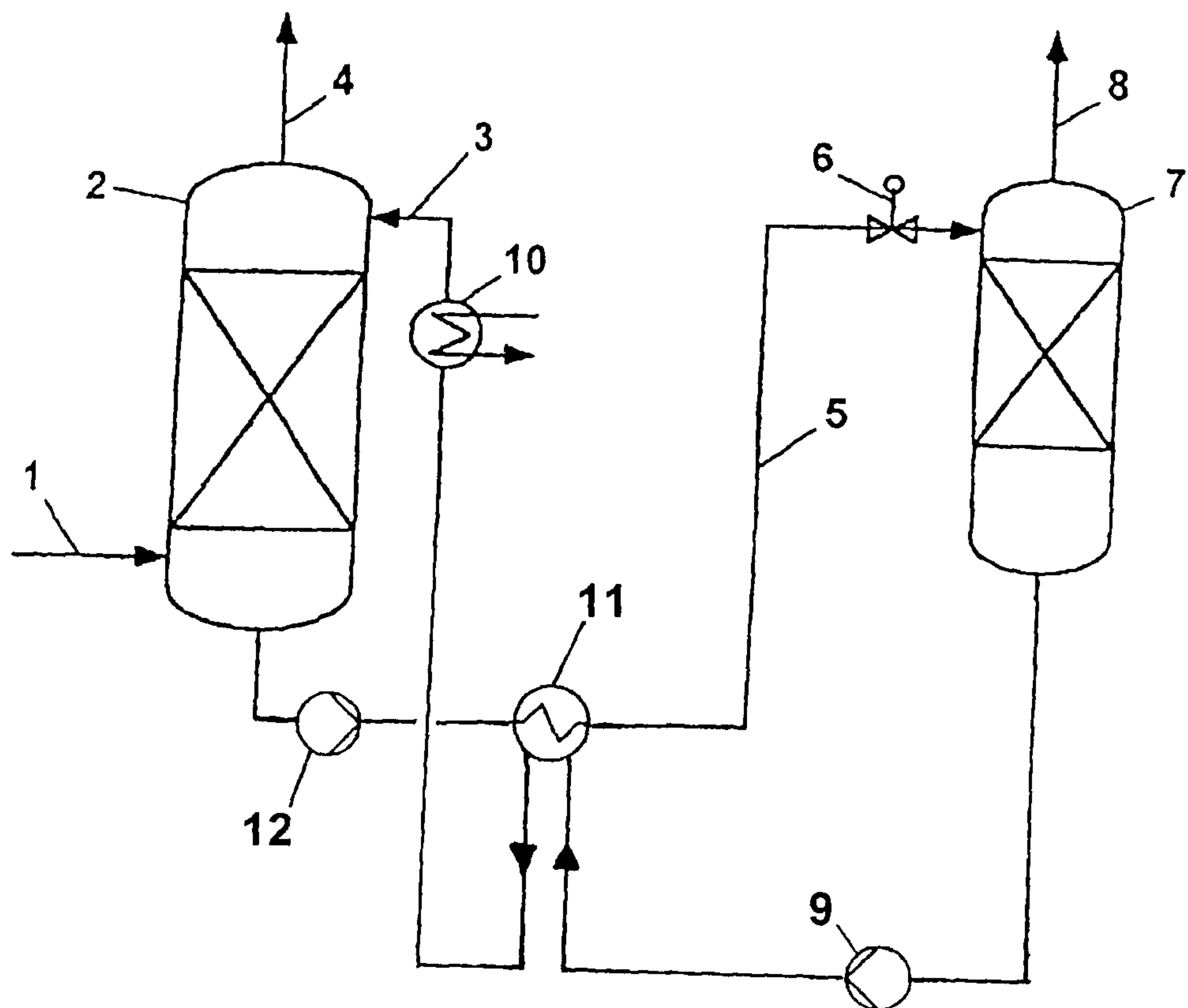
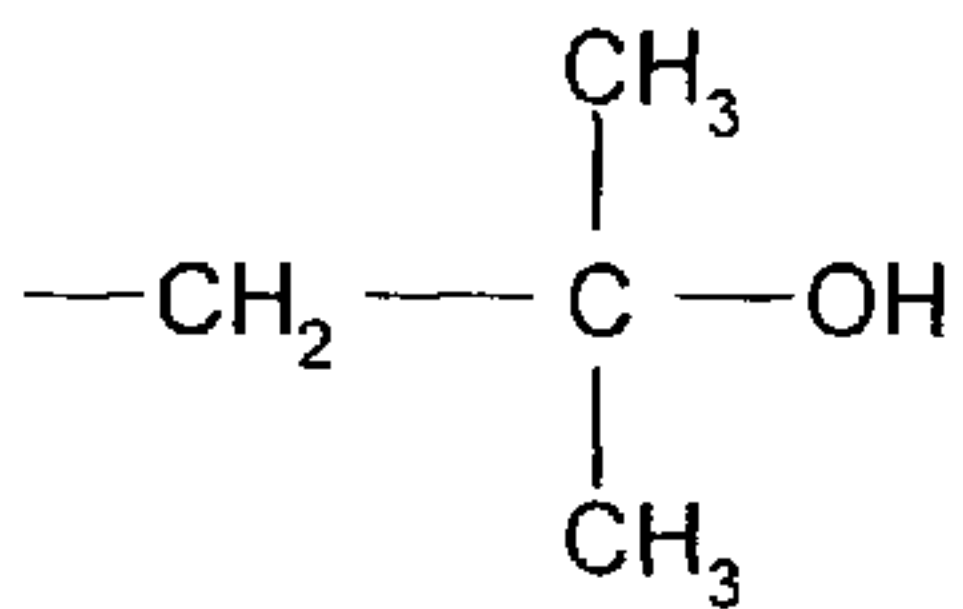
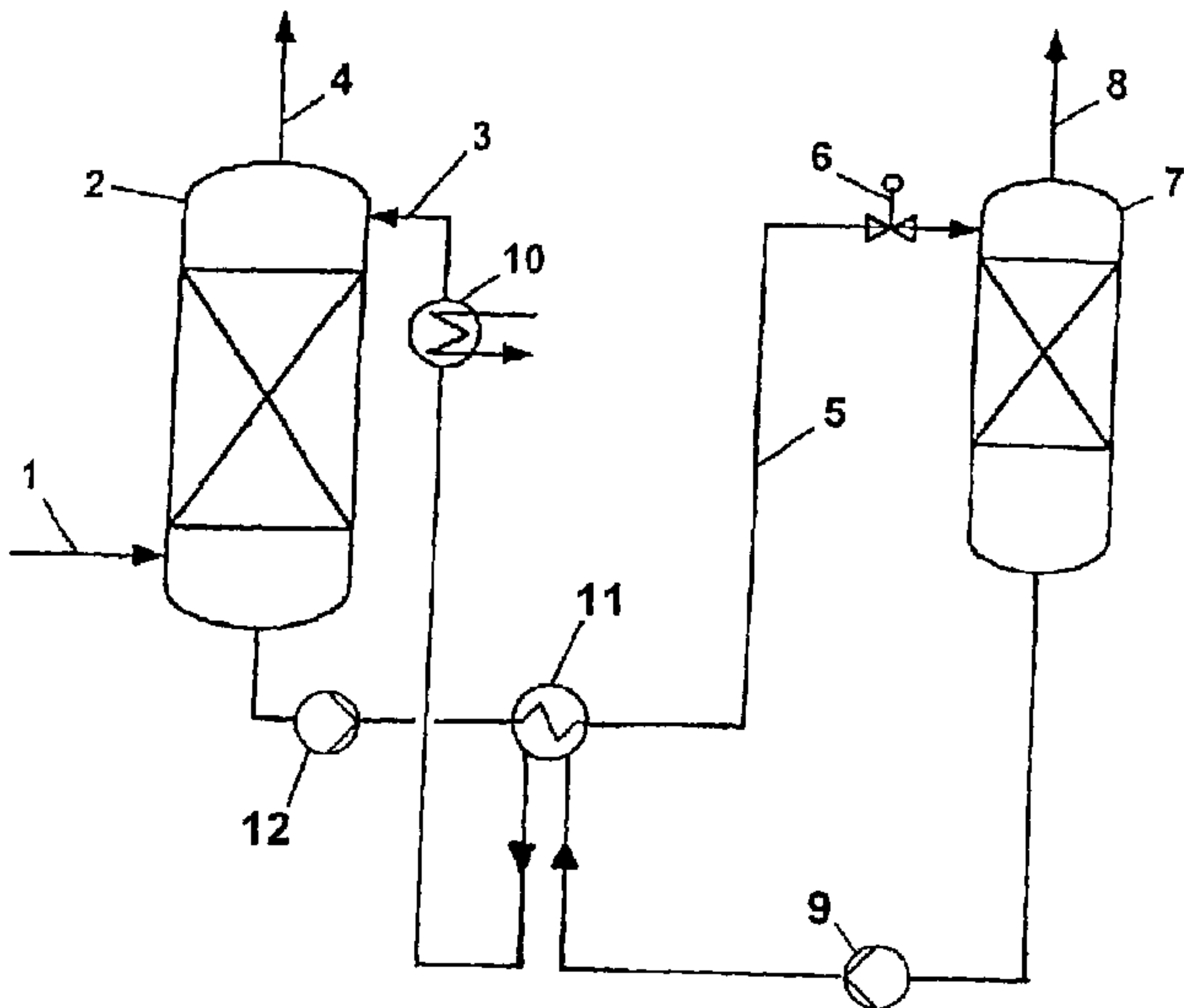


Figure 1



(I)