

Process for the energy-efficient production of alkali metal alkoxides

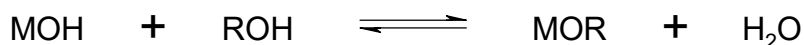
The present invention relates to a process for producing sodium and/or potassium alkoxides in countercurrent by reactive rectification. Alcohol is reacted in countercurrent with the respective alkali metal hydroxide. The vapours comprising alcohol and water are separated in at least two serially arranged rectification columns. The energy of the vapour obtained in the second rectification is utilized for operating the first rectification. This specific energy integration coupled with establishing a certain pressure difference in the two rectification stages makes it possible to cover a particularly large proportion of the energy required for the rectification through heating steam and to minimize the use of electricity.

1. Background of the invention

The production of alkali metal alkoxides is an important industrial process.

Alkali metal alkoxides are used as strong bases in the synthesis of numerous chemicals, for example in the production of pharmaceutical or agrochemical active ingredients. Alkali metal alkoxides are also used as catalysts in transesterification and amidation reactions.

Alkali metal alkoxides (MOR) are produced by reactive distillation of alkali metal hydroxides (MOH) and alcohols (ROH) in a countercurrent distillation column, wherein the water of reaction formed according to the following reaction <1> is removed with the distillate.



Such a process principle, by which aqueous alkali metal hydroxide solution and gaseous methanol are run in countercurrent in a rectification column, is disclosed for example in US 2,877,274 A. This process is described again in generally unchanged form in WO 01/42178 A1.

Similar processes, which additionally employ an entraining agent such as for example benzene, are disclosed in GB 377,631 A and US 1,910,331 A. This entraining agent is used to separate water and the water-soluble alcohol. In both patents the condensate is subjected to a phase separation to separate off the water of reaction.

Correspondingly, DE 96 89 03 C describes a process for continuous production of alkali metal alkoxides in a reaction column, wherein the water-alcohol mixture withdrawn at the top of the column is condensed and then subjected to a phase separation. The aqueous phase is discarded and the alcoholic phase is returned to the top of the column together with the fresh alcohol. EP 0 299 577 A2 describes a similar process, wherein the water in the condensate is separated off with the aid of a membrane.

The most industrially important alkali metal alkoxides are those of sodium and potassium, especially the methoxides and ethoxides. Their synthesis is frequently described in the prior art, for example in EP 1 997 794 A1.

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The syntheses of alkali metal alkoxides by reactive rectification described in the prior art typically afford vapours comprising the employed alcohol and water. It is advantageous for economic reasons to reuse the alcohol comprised in the vapours as a reactant in the reactive distillation. The vapours are therefore typically supplied to a rectification column and the alcohol present therein is separated off (described for example in GB 737 453 A and US 4,566,947 A). The thus recovered alcohol is then supplied to the reactive distillation as a reactant for example. Alternatively or in addition a portion of the alcohol vapour may be utilized for heating the rectification column (described in WO 2010/097318 A1). However, this requires that the vapour be compressed in order to achieve the temperature level required for heating the rectification column. The vapour is cooled between the compression stages, wherein a multistage compression is thermodynamically advantageous and an intermediate cooling ensures that the maximum allowable temperature of the compressor is not exceeded.

Heat integration within the rectification stage for efficient utilization of employed energy is described in a different context in Ott, J., Gronemann, V., Pontzen, F., Fiedler, E., Grossmann, G., Kersebohm, D.B., Weiss, G. and Witte, C. (2012). Methanol. in Ullmann's Encyclopedia of Industrial Chemistry, (Ed.). (doi:10.1002/14356007.a16_465.pub3). Paragraph 5.4 of this citation discloses the workup of crude methanol obtained in conventional synthesis processes by rectification using a plurality of rectification columns. It generally proposes utilizing the heat of condensation of the vapour obtained at the rectification column at relatively high pressure for heating the rectification column at relatively low pressure. However, this citation discloses nothing about advantageous energy integration in the separation of water-methanol vapours produced in the reactive rectification of alkali metal alkoxides.

In the production of alkali metal alkoxides it is desirable to cover the highest possible proportion of the energy altogether required for operation from lower-energy energy sources such as heating steam, instead of having to resort to higher-energy sources such as electrical current. This requirement is achieved on an industrial scale specifically in integrated plants (chemistry parks, technology parks) in which heating steam is generated and remains unutilized.

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It was accordingly the object of the present invention to provide an improved process for production of alkoxides of sodium and potassium by reactive distillation. Said process should especially allow energy-efficient utilization of the heat liberated during compression and cooling of the vapours. It should also cover a highest possible proportion of the energy requirement through heating steam as an external energy source and feature a lowest possible electricity demand.

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2. Brief summary of the invention

The present invention accordingly relates to a process for producing at least one alkali metal alkoxide of formula M_AOR , wherein R is a C_1 to C_6 hydrocarbon radical, preferably methyl or ethyl, and wherein M_A is selected from sodium, potassium, and wherein M_A is preferably sodium, wherein:

(a1) a reactant stream S_{AE1} comprising ROH is reacted with a reactant stream S_{AE2} comprising $MAOH$ in countercurrent at a pressure p_{3A} and a temperature T_{3A} in a reactive rectification column RR_A to afford a crude product RP_A comprising $MAOR$, water, ROH, $MAOH$,

wherein a bottoms product stream S_{AP} comprising ROH and $MAOR$ is withdrawn at the lower end of RR_A and a vapour stream S_{AB} comprising water and ROH is withdrawn at the upper end of RR_A ,

(a2) and optionally, simultaneously with and spatially separate from step (a1), a reactant stream S_{BE1} comprising ROH is reacted with a reactant stream S_{BE2} comprising M_BOH in countercurrent at a pressure p_{3B} and a temperature T_{3B} in a reactive rectification column RR_B to afford a crude product RP_B comprising M_BOR , water, ROH, M_BOH , wherein M_B is selected from sodium, potassium, and wherein M_B is preferably potassium,

wherein a bottoms product stream S_{BP} comprising ROH and M_BOR is withdrawn at the lower end of RR_B and a vapour stream S_{BB} comprising water and ROH is withdrawn at the upper end of RR_B ,

(b) the vapour stream S_{AB} and, if step (a2) is performed, the vapour stream S_{BB} , in admixture with S_{AB} or separately from S_{AB} , is passed into a first rectification column RD_1 , to obtain a mixture G_{RD1} comprising water and ROH in the first rectification column RD_1 ,

(c) the mixture G_{RD1} is in the first rectification column RD_1 at a pressure p_1 and a temperature T_1 separated into an ROH-comprising vapour stream S_{RDB1} at the upper end of RD_1 and a bottoms stream S_{RDS1} comprising water and ROH at the lower end of RD_1 ,

(d) the bottoms stream S_{RDS1} is completely or partially passed into a second rectification column RD_2 ,

to obtain a mixture G_{RD2} comprising water and ROH in the second rectification column RD_2 ,

(e) the mixture G_{RD2} is at a pressure p_2 and a temperature T_2 separated into an ROH-comprising vapour stream S_{RDB2} at the top of RD_2 and a bottoms stream S_{RDS2} comprising water and optionally ROH at the lower end of RD_2 ,

characterized

in that $p_2 > p_1$, $p_2 > p_{3A}$ and, in the cases in which step (a2) is performed, $p_2 > p_{3B}$, and wherein preferably also $p_{3A} > p_1$ and, in the cases in which step (a2) is performed, in addition preferably also $p_{3B} > p_1$,

and in that (f) energy from S_{RDB2} is transferred to the mixture G_{RD1} in the first rectification column RD_1 .

3. Figures

3.1 Figure 1

Figure 1 shows a process not according to the invention for producing alkali metal alkoxides with a corresponding interconnection of the rectification columns. Employed are a reactive rectification column ("reactive rectification column" is hereinbelow abbreviated to "reaction column") $RR_A <3A>$ at a pressure p_{3A} and two rectification columns $RD_1 <1>$ and $RD_2 <2>$ at the pressures p_1 and p_2 respectively. Here, $p_1 > p_{3A} > p_2$.

In $RR_A <3A>$ NaOH (stream $S_{AE2} <3A02>$) is reacted with methanol (stream $S_{AE1} <3A01>$) to afford a crude product $RP_A <3A07>$ comprising water, methanol, NaOH and sodium methoxide. At the lower end of $RR_A <3A>$ a methanol-sodium methoxide mixture $S_{AP} <3A04>$ is withdrawn. The bottoms evaporator $VS_{3A} <3A06>$ at the lower end of the reaction column $RR_A <3A>$ is used to adjust the concentration of the methoxide solution to the desired value in the resulting mixture $S_{AP^*} <3A08>$. There may additionally be attached at the bottom of the reaction column $RR_A <3A>$ a further evaporator, especially for startup of the reaction column $RR_A <3A>$ (not shown).

At the top of $RR_A <3A>$ a methanol-water mixture is withdrawn as vapour stream $S_{AB} <3A03>$. $S_{AB} <3A03>$ is supplied to the first water/methanol column $RD_1 <1>$, wherein optionally $S_{AB} <3A03>$ is at the top of the reaction column $RR_A <3A>$ partially condensed in the condenser $K_{RRA} <3A05>$ and recycled in liquid form as reflux to the top of $RR_A <3A>$. At least a portion of the vapour $S_{AB} <3A03>$ is then passed through a compressor $VD_{31} <10>$, thus increasing the pressure of the vapour $S_{AB} <3A03>$ from p_{3A} to the pressure p_1 .

A methanol/water mixture $G_{RD1} <108>$ is thus obtained in the first rectification column $RD_1 <1>$. Methanol is distillatively recovered as vapour $S_{RDB1} <101>$ in this first water/methanol column $RD_1 <1>$. The methanol recovered as vapour stream $S_{RDB1} <101>$ is at the withdrawal point $<109>$ at the top of $RD_1 <1>$ discharged therefrom and partially at the top of the rectification column $RD_1 <1>$ condensed in the condenser $K_{RD1} <102>$ and recycled in liquid form as reflux to the top of $RD_1 <1>$. The remaining portion of the methanol recovered as vapour $S_{RDB1} <101>$ is for example via a

throttle D_{13} <11> decompressed to the pressure p_3 and introduced into RR_A <3A> as methanol stream S_{AE1} <3A01>.

At the lower end (another term for “lower end of a rectification column” is “bottom of a rectification column”) of RD_1 <1> a bottoms stream S_{RDS1} <103> comprising water and methanol is discharged at the withdrawal point <110>. A first portion S_{RDS11} <104> of the stream S_{RDS1} <103> is supplied to a second water/methanol column RD_2 <2>, a second portion S_{RDS12} <105> of the stream S_{RDS1} <103> is via a bottoms evaporator VS_{RD1} <106> recycled to RD_1 <1>. S_{RDS11} <104> is for example via a throttle D_{12} <12> decompressed to the pressure p_2 before it is introduced into RD_2 <2>.

A methanol/water mixture G_{RD2} <206> is thus obtained in the second rectification column RD_2 <2>. In the rectification column RD_2 <2> residues of methanol from S_{RDS11} <104> are separated from the water and distillatively recovered as vapour stream S_{RDB2} <201> at the top of RD_2 <2>. The methanol recovered as vapour stream S_{RDB2} <201> is at the withdrawal point <208> at the top of RD_2 <2> discharged therefrom and partially at the top of the rectification column RD_2 <2> condensed in the condenser K_{RD2} <203> and recycled in liquid form as reflux to the top of RD_2 <2>. The remaining portion of the methanol recovered as vapour S_{RDB2} <201> is passed through a compressor VD_{23} <13>, thus compressed to the pressure p_3 and, together with the vapour S_{RDB1} <101> from RD_1 <1> decompressed to the pressure p_3 , introduced as methanol stream S_{AE1} <3A01> into RR_A <3A>.

At the lower end of RD_2 <2> a bottoms stream S_{RDS2} <202> comprising water and optionally methanol is discharged at the withdrawal point <207>. S_{RDS2} <202> is partially heated via a bottoms evaporator VS_{RD2} <204> and recycled into RD_2 <2>.

For the heating of the portion of the bottoms stream S_{RDS2} <202> which is recycled via VS_{RD2} <204> into RD_2 <2>, the energy liberated upon condensation of S_{RDB1} <101> in the condenser K_{RD1} <102> at the top of the rectification column RD_1 <1> is utilized. Said energy is supplied to VS_{RD2} <204>, as indicated by the dashed arrow <4>. The heat transfer may be effected indirectly, i.e. using a heat transfer medium distinct from S_{RDB1} <101> and S_{RDS2} <202>, or else directly, i.e. through contacting of S_{RDB1} <101> with S_{RDS2} <202> in the condenser K_{RD1} <102> or bottoms evaporator VS_{RD2} <204>. In the case of direct contacting it is sufficient to employ only the condenser K_{RD1} <102> and omit the bottoms evaporator VS_{RD2} <204> or to employ only the bottoms evaporator VS_{RD2} <204> and omit the condenser K_{RD1} <102>, and then in each case to pass both streams S_{RDB1} <101> with S_{RDS2} <202> through the condenser K_{RD1} <102> or the bottoms evaporator VS_{RD2} <204> such that energy, preferably heat, is transferred from S_{RDB1} <101> to S_{RDS2} <202>.

3.2 Figure 2

Figure 2 shows a further process not according to the invention for producing alkali metal alkoxides. This differs from the process shown in figure 1 in terms of the pressures in the respective column. In the embodiment shown in figure 1 $p_1 > p_{3A} > p_2$, while in the embodiment shown in figure 2 $p_1 > p_2 > p_{3A}$. This different pressure regime makes the compressor VD_{23} <13> unnecessary, and a throttle D_{23} <14> for example is attached. The throttle D_{23} <14> decompresses the vapour stream S_{RDB2} <201> from p_2 to the pressure p_{3A} , while in the embodiment according to figure 1 the compressor VD_{23} <13> increases it from p_2 to the pressure p_{3A} .

3.3 Figure 3

Figure 3 shows one embodiment of the process according to the invention for producing alkali metal alkoxides with a corresponding interconnection of the reactive rectification and rectification columns. Employed here, similarly to the embodiments described in figures 1 and 2, are a reactive rectification column RR_A <3A> at a pressure p_{3A} and two rectification columns RD_1 <1> and RD_2 <2> having the pressures p_1 and p_2 respectively. Here, $p_2 > p_1 > p_{3A}$. The setup shown in figure 3 corresponds to the setup shown in figure 2 with the following exceptions:

1. Arranged at the rectification column RD_1 <1> next to the bottoms evaporator VS_{RD1} <106> is an intermediate evaporator VZ_{RD1} <107> which may be used to supply energy to the mixture G_{RD1} <108> in RD_1 <1>. To this end the mixture G_{RD1} <108> is at a withdrawal point <111> discharged from the rectification column RD_1 <1> as stream S_{RDX1} <112>. S_{RDX1} <112> is heated in VZ_{RD1} <107> and recycled into the rectification column RD_1 <1>.
2. The throttle D_{12} <12> is on account of the different pressures in the rectification columns RD_1 <1> and RD_2 <2> ($p_2 > p_1$) replaced by a pump P <15>. The reason for this difference is that the pressure of S_{RDS11} <104>, when this stream is passed into RD_2 <2>, is according to the invention increased to p_2 .
3. In an optional embodiment additional methanol as stream S_{XE1} <205> is via the reflux at the rectification column RD_2 <2> added thereto.
4. The energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the intermediate evaporator VZ_{RD1} <107> transferred to S_{RDX1} <112> and, after reintroduction of S_{RDX1} <112> into RD_1 <1>, transferred from S_{RDX1} <112> to the mixture G_{RD1} <108> present in RD_1 <1>. Alternatively or in addition, energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the bottoms evaporator VS_{RD1} <106> transferred to the portion S_{RDS12} <105> of the stream S_{RDS1} <103>. Once S_{RDS12} <105> is recycled into RD_1 <1>, it transfers the energy to the mixture G_{RD1} <108> present in RD_1 <1>. The energy flow is shown by the dashed arrow <4>.

In the case of direct contacting it is sufficient to employ only the condenser K_{RD2} <203> and omit the bottoms evaporator VS_{RD1} <106> or to employ only the bottoms evaporator VS_{RD1} <106> and omit the condenser K_{RD2} <203>, and then in each case to pass both streams S_{RDB2} <201> with

S_{RDS12} <105> through the condenser K_{RD2} <203> or the bottoms evaporator VS_{RD1} <106> such that energy, preferably heat, is transferred from S_{RDB2} <201> to S_{RDS12} <105>.

3.4 Figure 4

- 5 Figure 4 shows a further embodiment of the process according to the invention for producing alkali metal alkoxides with a corresponding interconnection of the rectification columns. Employed here, similarly to the embodiments described in figures 1 and 2, are a reactive rectification column RR_A <3A> at a pressure p_{3A} and two rectification columns RD_1 <1> and RD_2 <2> having the pressures p_1 and p_2 respectively. Here, $p_2 > p_{3A} > p_1$. The setup shown in figure 4 corresponds to the setup
10 shown in figure 3 with the exception that the pressure $p_{3A} > p_1$. This allows the compressor VD_{31} <10> to be omitted, while the throttle D_{13} <11> is replaced by the compressor VD_{13} <16>.

3.5 Figure 5

- Figure 5 shows a further embodiment of the process according to the invention. Said embodiment
15 employs two reactive rectification columns (RR_A <3A> at a pressure p_{3A} and RR_B <3B> at a pressure p_{3B}) and two rectification columns (RD_1 <1> and RD_2 <2> having the pressures p_1 and p_2 respectively). Here, $p_2 > p_1 > p_{3A}$ and $p_1 > p_{3B}$.

- In RR_A <3A> NaOH (stream S_{AE2} <3A02>) is reacted with methanol (stream S_{AE1} <3A01>) to afford
20 a crude product RP_A <3A07> comprising water, methanol, NaOH and sodium methoxide.

- At the lower end of RR_A <3A> a methanol-sodium methoxide mixture S_{AP} <3A04> is withdrawn. The bottoms evaporator VS_{3A} <3A06> at the bottom of the column RR_A <3A> is used to adjust the concentration of the methoxide solution to the desired value in the resulting mixture S_{AP^*} <3A08>.
25 There may additionally be attached at the bottom of the column RR_A <3A> a further evaporator, especially for startup of the column RR_A <3A> (not shown).

- At the top of RR_A <3A> a methanol-water mixture is withdrawn as vapour S_{AB} <3A03>. S_{AB} <3A03> is supplied to the first water/methanol column RD_1 <1>, wherein optionally S_{AB} <3A03> is at the top of the reaction column RR_A <3A> partially condensed in the condenser K_{RRA} <3A05> and recycled
30 in liquid form as reflux to the top of RR_A <3A>.

- Simultaneously with the reaction in RR_A <3A>, in RR_B <3B> KOH (stream S_{BE2} <3B02>) is reacted with methanol (stream S_{BE1} <3B01>) to afford a crude product RP_B <3B07> comprising water,
35 methanol, KOH and potassium methoxide.

At the lower end of RR_B <3B> a methanol-potassium methoxide mixture S_{BP} <3B04> is withdrawn. The bottoms evaporator VS_{3B} <3B06> at the lower end of the column RR_B <3B> is used to adjust the concentration of the methoxide solution to the desired value in the resulting mixture S_{BP^*}

<3B08>. There may additionally be attached at the lower end of the column **RR_B** <3B> a further evaporator, especially for startup of the reaction column **RR_B** <3B> (not shown).

At the top of **RR_B** <3B> a methanol-water mixture is removed as vapour **S_{BB}** <3B03>, wherein
 5 optionally **S_{BB}** <3B03> is at the top of the reaction column **RR_B** <3B> partially condensed in the condenser **K_{RRB}** <3B05> and recycled in liquid form as reflux to the top of **RR_B** <3B>.

At least a portion of the vapours **S_{AB}** <3A03> and **S_{BB}** <3B03> is combined and the combined
 10 vapour is then passed through a compressor **VD₃₁** <10> which increases the pressure of the combined vapours to the pressure **p₁**. The combined vapours are then passed into the first rectification column **RD₁** <1> which then affords a methanol/water mixture **G_{RD1}** <108>.

In **RD₁** <1> methanol is distillatively recovered overhead as vapour **S_{RDB1}** <101>. The methanol
 15 recovered as vapour stream **S_{RDB1}** <101> is at the withdrawal point <109> at the top of **RD₁** <1> discharged therefrom and partially at the top of the rectification column **RD₁** <1> condensed in the condenser **K_{RD1}** <102> and recycled in liquid form as reflux to the top of **RD₁** <1>. The remaining portion of the methanol recovered as vapour **S_{RDB1}** <101> is for example via a throttle **D₁₃** <11> decompressed to the pressure **p_{3A}/p_{3B}** and introduced into the two reaction columns **RR_A** <3A> and **RR_B** <3B> as methanol stream **S_{AE1}** <3A01> and **S_{BE1}** <3B01> respectively.

20 Arranged at the rectification column **RD₁** <1> next to the bottoms evaporator **VS_{RD1}** <106> is an intermediate evaporator **VZ_{RD1}** <107> which may be used to supply energy to the mixture **G_{RD1}** <108> in **RD₁** <1>. To this end the mixture **G_{RD1}** <108> is at a withdrawal point <111> discharged from the rectification column **RD₁** <1> as stream **S_{RDX1}** <112>. **S_{RDX1}** <112> is heated in **VZ_{RD1}**
 25 <107> and recycled into the rectification column **RD₁** <1>.

At the lower end of **RD₁** <1> a bottoms stream **S_{RDS1}** <103> comprising water and methanol is
 discharged at the withdrawal point <110>. A first portion **S_{RDS11}** <104> of the stream **S_{RDS1}** <103> is
 30 supplied to a second water/methanol column **RD₂** <2>, a second portion **S_{RDS12}** <105> of the stream **S_{RDS1}** <103> is via a bottoms evaporator **VS_{RD1}** <106> recycled to **RD₁** <1>. The pressure of **S_{RDS11}** <104> is via a pump **P** <15> increased to the pressure **p₂** before this stream is introduced into **RD₂** <2>.

A methanol/water mixture **G_{RD2}** <206> is thus obtained in the second rectification column **RD₂** <2>.
 35 In the rectification column **RD₂** <2> residues of methanol from **S_{RDS11}** <104> are separated from the water and distillatively recovered as vapour stream **S_{RDB2}** <201> at the top of **RD₂** <2>. The methanol recovered as vapour stream **S_{RDB2}** <201> is at the withdrawal point <208> at the top of **RD₂** <2> discharged therefrom and partially at the top of the rectification column **RD₂** <2> condensed in the condenser **K_{RD2}** <203> and recycled in liquid form as reflux to the top of **RD₂** <2>.

Optionally, additional methanol as stream S_{XE1} <205> is via the reflux at the rectification column RD_2 <2> added thereto.

The remaining portion of the methanol recovered as vapour S_{RDB2} <201> not supplied to the condenser K_{RD2} <203> is passed through throttle D_{23} <14>, thus decompressed to the pressure p_{3A} or p_{3B} and, together with the vapour S_{RDB1} <101> from RD_1 <1> decompressed to the pressure p_{3A} or p_{3B} , introduced into RR_A <3A> and RR_B <3B> as methanol stream S_{AE1} <3A01> and S_{BE1} <3B01> respectively.

At the lower end of RD_2 <2> a bottoms stream S_{RDS2} <202> comprising water and optionally methanol is discharged at the withdrawal point <207>. S_{RDS2} <202> is partially heated via a bottoms evaporator VS_{RD2} <204> and recycled into RD_2 <2>.

The energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the intermediate evaporator VZ_{RD1} <107> transferred to S_{RDX1} <112> and, after reintroduction of S_{RDX1} <112> into RD_1 <1>, transferred from S_{RDX1} <112> to the mixture G_{RD1} <108> present in RD_1 <1>. Alternatively or in addition, energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the bottoms evaporator VS_{RD1} <106> transferred to the portion S_{RDS12} <105> of the stream S_{RDS1} <103>. Once S_{RDS12} <105> is recycled into RD_1 <1>, it transfers the energy to the mixture G_{RD1} <108> present in RD_1 <1>. The energy flow is shown by the dashed arrow <4>.

3.6 Figure 6

Figure 6 shows a further embodiment of the process according to the invention for producing alkali metal alkoxides with a corresponding interconnection of the rectification columns. Employed here, similarly to the embodiment described in figure 5, are two reactive rectification columns RR_A <3A> and RR_B <3B> at a pressure p_{3A} and p_{3B} respectively and two rectification columns RD_1 <1> and RD_2 <2> having the pressures p_1 and p_2 respectively. In contrast to the embodiment according to figure 5, $p_2 > p_{3A} > p_1$ and $p_2 > p_{3B} > p_1$. The setup shown in figure 6 corresponds to the setup shown in figure 5 with the exception that the pressure $p_{3A} > p_1$ and the pressure $p_{3B} > p_1$, thus allowing the compressor VD_{31} <10> to be omitted and the throttle D_{13} <11> to be replaced by the compressor VD_{13} <16>.

3.7 Figure 7

Figure 7 shows an embodiment of the process according to the invention which corresponds to that described in figure 5. Here, $p_2 > p_1 > p_{3A}$ and $p_1 > p_{3B}$. It shows the embodiment in which the energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is directly via the bottoms evaporator VS_{RD1} <106> transferred to the portion S_{RDS12} <105> of the stream S_{RDS1} <103> which is recycled into RD_1 <1>. The transfer is effected directly since the vapour S_{RDB2} <201> is contacted with S_{RDS12} <105> in VS_{RD1} <106>, thus allowing energy transfer from S_{RDB2} <201> to S_{RDS12} <105> without for example interposing a further heat transfer medium. The

condenser K_{RD2} <203> shown in figure 5 may be omitted, and the stream of fresh methanol S_{XE1} <205> is here introduced directly into the column RD_2 <2>.

3.8 Figure 8

- 5 Figure 8 shows an embodiment of the process according to the invention which corresponds to that described in figure 6. Here, $p_2 > p_{3A} > p_1$ and $p_2 > p_{3B} > p_1$. It shows the embodiment in which the energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is directly via the bottoms evaporator VS_{RD1} <106> transferred to the portion S_{RDS12} <105> of the stream S_{RDS1} <103> which is recycled into RD_1 <1>. The transfer is effected directly since the vapour S_{RDB2} <201> is contacted with S_{RDS12} <105> in VS_{RD1} <106>, thus allowing energy transfer from S_{RDB2} <201> to S_{RDS12} <105> without for example interposing a further heat transfer medium. The condenser K_{RD2} <203> shown in figure 6 may be omitted, and the stream of fresh methanol S_{XE1} <205> is here introduced directly into the rectification column RD_2 <2>.

3.9 Figure 9

- 15 Figure 9 shows an embodiment of the process according to the invention which corresponds to that described in figure 5. Here, $p_2 > p_1 > p_{3A}$ and $p_1 > p_{3B}$. It shows the embodiment in which the energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the intermediate evaporator VZ_{RD1} <107> transferred to the water/methanol mixture G_{RD1} <108> from RD_1 <1>. G_{RD1} <108> is withdrawn from the rectification column RD_1 <1> at a withdrawal point <111> as stream S_{RDX1} <112>. S_{RDB2} <201> transfers energy to S_{RDX1} <112> in VZ_{RD1} <107>, especially by heating of S_{RDX1} <112>. S_{RDX1} <112> is then recycled into the rectification column RD_1 <1>.
- 20 In RD_1 <1> the stream S_{RDX1} <112> then transfers the energy to the mixture G_{RD1} <108> present in RD_1 <1>. The transfer of energy from S_{RDB2} <201> to S_{RDX1} <112> is effected directly since the vapour S_{RDB2} <201> is contacted with S_{RDX1} <112> from RD_1 <1> in VZ_{RD1} <107>, thus allowing energy transfer from S_{RDB2} <201> to S_{RDX1} <112> from RD_1 <1> without for example interposing a further heat transfer medium. The condenser K_{RD2} <203> shown in figure 5 may be omitted, and the stream of fresh methanol S_{XE1} <205> is here introduced directly into the rectification column RD_2 <2>.
- 25 30

3.10 Figure 10

- 35 Figure 10 shows an embodiment of the process according to the invention which corresponds to that described in figure 6. Here, $p_2 > p_{3A} > p_1$ and $p_2 > p_{3B} > p_1$. It shows the embodiment in which the energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is via the intermediate evaporator VZ_{RD1} <107> transferred to the water/methanol mixture G_{RD1} <108> from RD_1 <1>. G_{RD1} <108> is withdrawn from the rectification column RD_1 <1> at a withdrawal point

<111> as stream S_{RDX1} <112>. S_{RDB2} <201> transfers energy to S_{RDX1} <112> in VZ_{RD1} <107>, especially by heating of S_{RDX1} <112>. S_{RDX1} <112> is then recycled into the rectification column RD_1 <1>.

- 5 In RD_1 <1> the stream S_{RDX1} <112> then transfers the energy to the mixture G_{RD1} <108> present in RD_1 <1>. The transfer of energy from S_{RDB2} <201> to S_{RDX1} <112> is effected directly since the vapour S_{RDB2} <201> is contacted with S_{RDX1} <112> from RD_1 <1> in VZ_{RD1} <107>, thus allowing energy transfer from S_{RDB2} <201> to S_{RDX1} <112> from RD_1 <1> without for example interposing a further heat transfer medium. The condenser K_{RD2} <203> shown in figure 6 may be omitted, and
10 the stream of fresh methanol S_{XE1} <205> is here introduced directly into the rectification column RD_2 <2>.

3.11 Figure 11

- Figure 11 shows an embodiment of the process according to the invention which shows a
15 combination of the embodiments according to figures 7 and 9. Here, $p_2 > p_1 > p_{3A}$ and $p_1 > p_{3B}$. It shows the embodiment in which the energy liberated upon condensation of the vapour S_{RDB2} <201> at the top of RD_2 <2> is directly via the bottoms evaporator VS_{RD1} <106> transferred to the portion S_{RDS12} <105> of the stream S_{RDS1} <103> which is recycled into RD_1 <1>. The transfer is effected directly since the vapour S_{RDB2} <201> is contacted with S_{RDS12} <105> in VS_{RD1} <106>, thus
20 allowing energy transfer from S_{RDB2} <201> to S_{RDS12} <105> without for example interposing a further heat transfer medium.

- After this heat transfer further energy from the vapour S_{RDB2} <201> is transferred via the intermediate evaporator VZ_{RD1} to the water/methanol mixture G_{RD1} <108> from RD_1 <1>. G_{RD1}
25 <108> is withdrawn from the rectification column RD_1 <1> at a withdrawal point <111> as stream S_{RDX1} <112>. S_{RDB2} <201> transfers energy to S_{RDX1} <112> in VZ_{RD1} <107>, especially by heating of S_{RDX1} <112>. S_{RDX1} <112> is then recycled into the rectification column RD_1 <1>.

- The transfer of energy from S_{RDB2} <201> to S_{RDX1} <112> is effected directly since the vapour S_{RDB2}
30 <201> is contacted with S_{RDX1} <112> from RD_1 <1> in VZ_{RD1} <107>, thus allowing energy transfer from S_{RDB2} <201> to S_{RDX1} <112> from RD_1 <1> without for example interposing a further heat transfer medium.

- In RD_1 <1> the streams S_{RDX1} <112> and S_{RDS12} <105> then transfer the energy absorbed by S_{RDB2}
35 <201> to the mixture G_{RD1} <108> present in RD_1 .

3.12 Figure 12

Figure 12 shows an embodiment of the process according to the invention which corresponds to a combination of the embodiments of figures 8 and 10. Here, $p_2 > p_{3A} > p_1$ and $p_2 > p_{3B} > p_1$. It

shows an embodiment in which the energy liberated upon condensation of the vapour $S_{RDB2} <201>$ at the top of $RD_2 <2>$ is directly via the bottoms evaporator $VS_{RD1} <106>$ transferred to the portion $S_{RDS12} <105>$ of the stream $S_{RDS1} <103>$ which is recycled into $RD_1 <1>$. The transfer is effected directly since the vapour $S_{RDB2} <201>$ is contacted with $S_{RDS12} <105>$ in $VS_{RD1} <106>$, thus allowing energy transfer from $S_{RDB2} <201>$ to $S_{RDS12} <105>$ without for example interposing a further heat transfer medium.

After this heat transfer further energy from the vapour $S_{RDB2} <201>$ is transferred via the intermediate evaporator VZ_{RD1} to the water/methanol mixture $G_{RD1} <108>$ from $RD_1 <1>$. $G_{RD1} <108>$ is withdrawn from the rectification column $RD_1 <1>$ after withdrawal point $<111>$ as stream $S_{RDX1} <112>$. $S_{RDB2} <201>$ transfers energy to $S_{RDX1} <112>$ in $VZ_{RD1} <107>$, especially by heating of $S_{RDX1} <112>$. $S_{RDX1} <112>$ is then recycled into the rectification column $RD_1 <1>$.

The transfer of energy from $S_{RDB2} <201>$ to $S_{RDX1} <112>$ is effected directly since the vapour $S_{RDB2} <201>$ is contacted with $S_{RDX1} <112>$ from $RD_1 <1>$ in $VZ_{RD1} <107>$, thus allowing energy transfer from $S_{RDB2} <201>$ to $S_{RDX1} <112>$ from $RD_1 <1>$ without for example interposing a further heat transfer medium.

In $RD_1 <1>$ the streams $S_{RDX1} <112>$ and $S_{RDS12} <105>$ then transfer the energy absorbed by $S_{RDB2} <201>$ to the mixture $G_{RD1} <108>$ present in $RD_1 <1>$.

4. Detailed description of the invention

4.1 Step (a1) of the process according to the invention

In step (a1) of the process according to the invention for producing at least one alkali metal alkoxide of formula M_AOR a reactant stream S_{AE1} comprising ROH is reacted with a reactant stream S_{AE2} comprising $MAOH$ in countercurrent at a pressure p_{3A} and a temperature T_{3A} in a reactive rectification column RR_A to afford a crude product RP_A comprising M_AOR , water, ROH, $MAOH$.

According to the invention, a "reactive rectification column" is a rectification column in which the reaction according to step (a1) or step (a2) of the process according to the invention proceeds at least in some parts. It may also be abbreviated to "reaction column".

In step (a1) of the process according to the invention a bottoms product stream S_{AP} comprising ROH and M_AOR is withdrawn at the lower end of RR_A . A vapour stream S_{AB} comprising water and ROH is withdrawn at the upper end of RR_A .

"Vapour stream" means that the respective stream is a gaseous stream.

In the process according to the invention, R is a C₁-C₆ hydrocarbon radical, preferably selected from the group consisting of methyl, ethyl, *n*-propyl, *iso*-propyl, *n*-butyl, *sec*-butyl, *iso*-butyl, *tert*-butyl, isomers of pentyl, such as *n*-pentyl, more preferably selected from the group consisting of methyl, ethyl, *n*-propyl, *iso*-propyl, yet more preferably selected from the group consisting of
 5 methyl, ethyl. R is particularly preferably methyl and ROH is accordingly methanol.

M_A is selected from sodium, potassium, and preferably sodium.

The reactant stream **S_{AE1}** comprises ROH. In a preferred embodiment the mass fraction of ROH in
 10 **S_{AE1}**, based on the total mass of the reactant stream **S_{AE1}**, is $\geq 95\%$ by weight, yet more preferably $\geq 99\%$ by weight, wherein **S_{AE1}** otherwise comprises especially water.

The alcohol ROH used as reactant stream **S_{AE1}** in step (a1) of the process according to the invention can also be a commercially available alcohol having a mass fraction of alcohol, based on
 15 the total mass of the reactant stream **S_{AE1}**, of more than 99.8% by weight and a mass fraction of water, based on the total mass of the reactant stream **S_{AE1}**, of up to 0.2% by weight.

The reactant stream **S_{AE1}** is preferably introduced in vapour form.

20 The reactant stream **S_{AE2}** comprises M_AOH. In a preferred embodiment **S_{AE2}** comprises not only M_AOH but also at least one further compound selected from water, ROH. **S_{AE2}** more preferably also comprises water in addition to M_AOH, in which case **S_{AE2}** is an aqueous solution of M_AOH.

When the reactant stream **S_{AE2}** comprises M_AOH and water, the mass fraction of M_AOH, based on
 25 the total weight of the reactant stream **S_{AE2}**, is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight, most preferably 50% by weight.

When the reactant stream **S_{AE2}** comprises M_AOH and ROH, the mass fraction of M_AOH, based on
 30 the total weight of the reactant stream **S_{AE2}**, is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight.

In the particular case in which the reactant stream **S_{AE2}** comprises both water and ROH in addition
 35 to M_AOH, it is particularly preferable when the mass fraction of M_AOH, based on the total weight of the reactant stream **S_{AE2}**, is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight.

Step (a1) of the process according to the invention is performed in a reactive rectification column (or "reaction column") **RR_A**.

Step (a2) of the process according to the invention is performed in a reactive rectification column
5 (or "reaction column") **RR_B**.

The reaction column **RR_A/RR_B** preferably contains internals. Suitable internals are, for example, trays, structured packings or unstructured packings. When the reaction column **RR_A/RR_B** contains trays, bubble cap trays, valve trays, tunnel trays, Thormann trays, cross-slit bubble cap trays or
10 sieve trays are suitable. When the reaction column **RR_A/RR_B** contains trays, it is preferable to choose trays where not more than 5% by weight, more preferably less than 1% by weight, of the liquid trickles through the respective trays. The constructional measures required to minimize trickle-through of the liquid are familiar to those skilled in the art. In the case of valve trays, particularly tightly closing valve designs are selected for example. Reducing the number of valves
15 also makes it possible to increase the vapour velocity in the tray openings to twice the value typically established. When using sieve trays it is particularly advantageous to reduce the diameter of the tray openings while maintaining or even increasing the number of openings.

When using structured or unstructured packings, structured packings are preferred in terms of
20 uniform distribution of the liquid. In this embodiment it is further preferable when in all parts of the column cross section corresponding to more than 2% of the total column cross section the average ratio of liquid stream to vapour stream is not exceeded by more than 15%, more preferably by more than 3%, in respect of the liquid. This minimized liquid amount makes it possible for the capillary effect at the wire meshes to eliminate local peaks of liquid trickling density.

25 For columns comprising unstructured packings, especially comprising random packings, and for columns comprising structured packings, the desired characteristics of the liquid distribution may be achieved when the liquid trickling density in the edge region of the column cross section adjacent to the column shell, corresponding to about 2% to 5% of the total column cross section, is
30 reduced compared to the other cross-sectional regions by up to 100%, preferably by 5% to 15%. This can easily be achieved by, for example, targeted distribution of the drip points of the liquid distributors or the holes thereof.

The process according to the invention may be carried out either continuously or discontinuously. It
35 is preferably carried out continuously.

According to the invention "reaction of a reactant stream **S_{AE1}** comprising ROH with a reactant stream **S_{AE2}** comprising MAOH in countercurrent" is achieved, in particular, as a result of the feed point for at least a portion of the reactant stream **S_{AE1}** comprising ROH in step (a1) being located
40 on the reaction column **RR_A** below the feed point of the reactant stream **S_{AE2}** comprising MAOH.

The reaction column **RR_A** preferably comprises at least 2, in particular 15 to 40, theoretical plates between the feed point of the reactant stream **S_{AE1}** and the feed point of the reactant stream **S_{AE2}**.

- 5 The reaction column **RR_A** is preferably operated as a pure stripping column. Accordingly the reactant stream **S_{AE1}** comprising ROH is especially supplied in vaporous form in the lower region of the reaction column **RR_A**. Step (a1) of the process according to the invention also comprises the case where a portion of the reactant stream **S_{AE1}** comprising ROH is added in vapour form below the feed point of the reactant stream **S_{AE2}** comprising MAOH but nevertheless at the upper end or in
- 10 the region of the upper end of the reaction column **RR_A**. This makes it possible to reduce the dimensions in the lower region of the reaction column **RR_A**. When a portion of the reactant stream **S_{AE1}** comprising ROH, in particular methanol, is added especially in vaporous form at the upper end or in the region of the upper end of the reaction column **RR_A**, only a fraction of 10% to 70% by weight, preferably of 30% to 50% by weight (in each case based on the total amount of the alcohol
- 15 ROH employed in step (a1) as **S_{AE1}**) is supplied at the lower end of the reaction column **RR_A** and the remaining fraction is added in vaporous form in a single stream or divided into a plurality of substreams preferably 1 to 10 theoretical trays, particularly preferably 1 to 3 theoretical trays, below the feed point of the reactant stream **S_{AE2}** comprising MAOH.
- 20 In the reaction column **RR_A**, the reactant stream **S_{AE1}** comprising ROH is then reacted with the reactant stream **S_{AE2}** comprising MAOH according to the reaction <1> described hereinabove to afford MAOR and H₂O, where these products are present in admixture with the reactants ROH and MAOH since an equilibrium reaction is concerned. Accordingly a crude product **RP_A** which contains not only the products MAOR and water but also ROH and MAOH is obtained in the reaction column
- 25 **RR_A** in step (a1) of the process according to the invention.

The bottoms product stream **S_{AP}** comprising ROH and MAOR is then obtained and withdrawn at the lower end of **RR_A**.

- 30 A water-containing alcohol stream, presently referred to as "vapour stream **S_{AB}** comprising water and ROH", is withdrawn at the upper end of **RR_A**, preferably at the column top of **RR_A**.

- The amount of the alcohol ROH comprised by the reactant stream **S_{AE1}** is preferably chosen such that said alcohol simultaneously serves as a solvent for the alkali metal alkoxide MAOR obtained in
- 35 the bottoms product stream **S_{AP}**. The amount of the alcohol ROH in the reactant stream **S_{AE1}** is preferably chosen to achieve in the bottom of the reaction column **RR_A** the desired concentration of the alkali metal alkoxide solution which is withdrawn as a bottoms product stream **S_{AP}** comprising ROH and MAOR.

In a preferred embodiment of the process according to the invention, and especially in cases in which S_{AE2} also comprises water in addition to M_{AOH} , the ratio of the total weight (masses; unit: kg) of alcohol ROH used as reactant stream S_{AE1} in step (a1) to the total weight (masses; unit: kg) of M_{AOH} used as reactant stream S_{AE2} in step (a1) is from 1: 1 to 50: 1, more preferably 5: 1 to 48: 1, yet more preferably 9: 1 to 35: 1, yet still more preferably 10: 1 to 30: 1, yet still more preferably 13: 1 to 22: 1, most preferably 14: 1.

The reaction column RR_A is operated with or without, preferably without, reflux.

- 10 "Without reflux" means that the vapour stream S_{AB} withdrawn at the upper end of RR_A comprising water and ROH is completely supplied to the first rectification column RD_1 according to step (b). The vapour stream S_{AB} comprising water and ROH is preferably supplied to the first rectification column RD_1 in vaporous form.
- 15 "With reflux" means that the vapour stream S_{AB} withdrawn at the upper end of the respective column, the reaction column RR_A in step (a1), comprising water and ROH is not completely discharged, i.e. is not completely supplied to the first rectification column RD_1 in step (b), but rather is at least partially, preferably partially, recycled to the respective column, i.e. the reaction column RR_A in step (a1), as reflux. In the cases where such a reflux is established, the reflux ratio is
- 20 preferably 0.05 to 0.99, more preferably 0.1 to 0.9, yet more preferably 0.11 to 0.34, particularly preferably 0.14 to 0.27 and very particularly preferably 0.17 to 0.24. A reflux may be established by attaching to the top of the respective column, the reaction column RR_A in step (a1), a condenser K_{RRA} in which the vapour stream S_{AB} is at least partially condensed and sent back to the respective column, the reaction column RR_A in step (a1). Generally and in the context of the present invention,
- 25 a reflux ratio is to be understood as meaning the ratio of the mass flow (kg/h) recycled to the respective column in liquid form (reflux) to the mass flow (kg/h) discharged from the respective column in liquid form (distillate) or gaseous form (vapour).

- In the embodiment in which a reflux is established on the reaction column RR_A , the alcohol M_{AOH} employed in step (a1) as reactant stream S_{AE2} may also be at least partially, preferably partially, mixed with the reflux stream and the resulting mixture thus be supplied to step (a1).

- Step (a1) of the process according to the invention is in particular performed at a temperature T_{3A} in the range from 25°C to 200°C, preferably in the range from 45°C to 150°C, more preferably in the range from 47°C to 120°C, more preferably in the range from 60°C to 110°C.

Step (a1) of the process according to the invention is in particular performed at a pressure p_{3A} of 0.5 bar to 40 bar, preferably in the range from 0.75 bar to 5 bar, more preferably in the range from 1 bar to 2 bar, more preferably in the range from 1 bar to 1.8 bar, yet more preferably at 1.1 bar to

1.6 bar. It is an essential feature of the invention that when establishing the pressure p_{3A} : $p_2 > p_{3A}$. It is especially also the case that $p_{3A} > p_1$.

5 The reaction column **RR_A** comprises in a preferred embodiment at least one evaporator which is in particular selected from intermediate evaporators **VZ_{3A}** and bottoms evaporators **VS_{3A}**. The reaction column **RR_A** particularly preferably comprises at least one bottoms evaporator **VS_{3A}**. Evaporators are special embodiments of heat exchangers **WT**.

10 Condensers **K** are likewise special embodiments of heat exchangers **WT**. Typical condensers are known to those skilled in the art. These are preferably employed as liquefiers at the top of rectification columns and reaction columns. In the direct energy transfer from the top stream of one column to the bottoms or intermediate stream of another column a condenser of one column may simultaneously be employed as an evaporator of the other column (as shown in the examples).

15 According to the invention "intermediate evaporators" **VZ** (for example **VZ_{3A}** in **RR_A**, **VZ_{3B}** in **RR_B**, **VZ_{RD1}** in **RD₁**, **VZ_{RD2}** in **RD₂**) are to be understood as meaning evaporators arranged above the bottom of the respective column, in particular above the bottom of the reaction column **RR_A/RR_B** or above the bottom of the rectification column **RD₁** or **RD₂**. They in particular evaporate crude product **RP_A/RP_B** or **S_{RDX1Z}** as sidestream.

20 According to the invention "bottoms evaporators" **VS** (for example **VS_{3A}** in **RR_A**, **VS_{3B}** in **RR_B**, **VS_{RD1}** in **RD₁**, **VS_{RD2}** in **RD₂**) are to be understood as meaning evaporators which heat the bottom of the respective column, in particular the bottom of the reaction column **RR_A/RR_B** or rectification column **RD₁** or **RD₂**. They evaporate bottoms product stream (for example **S_{AP}/S_{BP}** or **S_{RDX1S}**).

25 An evaporator is typically arranged outside the respective reaction column or rectification column. The mixture to be evaporated in the evaporator is withdrawn from the column via a takeoff or "withdrawal point" and supplied to the at least one evaporator. The evaporated mixture is recycled back into the respective column, optionally with a residual proportion of liquid, via a feed or "feed point". When the evaporator is an intermediate evaporator, the takeoff by means of which the respective mixture is withdrawn and supplied to the evaporator is a sidestream takeoff and the feed by means of which the evaporated respective mixture is sent back to the column is a sidestream feed. When the evaporator is a bottoms evaporator, i.e. heats the column bottom, at least a portion of the bottom takeoff stream is evaporated and recycled back into the respective column in the region of the column bottom.

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However, it is alternatively also possible for example on a suitable tray when using an intermediate evaporator or in the bottom of the respective column to provide tubes which are traversed by the relevant heating medium. In this case, the evaporation occurs on the tray or in the bottom of the column. However, it is preferable to arrange the evaporator outside the respective column.

Suitable evaporators employable as intermediate evaporators and bottoms evaporators include for example natural circulation evaporators, forced circulation evaporators, forced circulation evaporators with decompression, steam boilers, falling film evaporators or thin film evaporators. Heat exchangers for evaporation typically employed in the case of natural circulation evaporators and forced circulation evaporators are shell and tube or plate apparatuses. When using a shell and tube exchanger the heating medium may flow either through the tubes with the mixture to be evaporated flowing around the tubes or else the heating medium may flow around the tubes with the mixture to be evaporated flowing through the tubes. In the case of a falling-film evaporator, the mixture to be evaporated is typically introduced as a thin film on the inside of a tube and the tube is heated externally. In contrast to a falling-film evaporator, a thin-film evaporator additionally comprises a rotor with wipers which distributes the liquid to be evaporated on the inner wall of the tube to form a thin film.

In addition to the recited evaporator types it is however also possible to employ any desired further evaporator type known to those skilled in the art and suitable for use on a rectification column.

When the reaction column **RR_A**/reaction column **RR_B** has an intermediate evaporator **VZ_{3A}** or **VZ_{3B}** it is preferable when the respective intermediate evaporator is arranged in the stripping region of the reaction column **RR_A** in the region of the feed point of the reactant stream **S_{AE1}** or in the case of the reaction column **RR_B** in the region of the feed point of the reactant stream **S_{BE1}**. This makes it possible to introduce a predominant portion of the heat energy via the intermediate evaporator **VZ_{3A}/VZ_{3B}**. It is thus possible for example to introduce more than 80% of the energy via the intermediate evaporator. According to the invention the intermediate evaporator **VZ_{3A}/VZ_{3B}** is preferably arranged and/or configured such that it introduces more than 50%, in particular more than 75%, of the total energy required for the reactive rectification.

When the reaction column **RR_A**/reaction column **RR_B** has an intermediate evaporator **VZ_{3A}** or **VZ_{3B}** it is additionally advantageous when the intermediate evaporator is arranged such that the reaction column **RR_A/RR_B** has 1 to 50 theoretical trays below the intermediate evaporator and has 1 to 200 theoretical trays above the intermediate evaporator. It is especially preferred when the reaction column **RR_A/RR_B** then has 2 to 10 theoretical trays below the intermediate evaporator and has 20 to 50 theoretical trays above the intermediate evaporator.

When the reaction column **RR_A**/reaction column **RR_B** has an intermediate evaporator **VZ_{3A}/VZ_{3B}** it is also advantageous when the sidestream takeoff (i.e. the "withdrawal point **E_{RRA}**" on the reaction column **RR_A**/the "withdrawal point **E_{RRB}**" on the reaction column **RR_B**) by means of which the crude product **RP_A/RP_B** is supplied to the intermediate evaporator **VZ_{3A}/VZ_{3B}** and the sidestream feed (i.e. the "withdrawal point **Z_{RRA}**" on the reaction column **RR_A**/the "withdrawal point **Z_{RRB}**" on the reaction column **RR_B**) by means of which the evaporated crude product **RP_A/RP_B** from the intermediate evaporator **VZ_{3A}/VZ_{3B}** is sent back to the respective reaction column **RR_A/RR_B** are

positioned between the same plates of the reaction column **RR_A**/reaction column **RR_B**. However, it is also possible for the sidestream takeoff and sidestream feed to be arranged at different heights.

In a preferred embodiment when using an intermediate evaporator **VZ_{3A}/VZ_{3B}** in **RR_A/RR_B** the diameter of the reaction column **RR_A/RR_B** above the intermediate evaporator **RR_A/RR_B** is greater than the diameter of the reaction column **RR_A/RR_B** below the intermediate evaporator **VZ_{3A}/VZ_{3B}**. This has the advantage of allowing capital expenditure savings.

In such an intermediate evaporator **VZ_{3A}/VZ_{3B}** liquid crude product **RP_A** comprising M_AOR, water, ROH, M_AOH present in the reaction column **RR_A** or liquid crude product **RP_B** comprising M_BOR, water, ROH, M_BOH present in the reaction column **RR_B** may be converted into the gaseous state or, if already in the gaseous state, heated further, thus improving the efficiency of the reaction according to step (a1)/(a2) in the process according to the invention.

Arranging one or more intermediate evaporators **VZ_{3A}** in the upper region of the reaction column **RR_A** or one or more intermediate evaporators **VZ_{3B}** in the upper region of the reaction column **RR_B** makes it possible to reduce the dimensions in the lower region of the reaction column **RR_A/RR_B**. In the embodiment having at least one, preferably two or more, intermediate evaporators **VZ_{3A}/VZ_{3B}**, it is also possible to supply substreams of the ROH in liquid form in the upper region of the reaction column **RR_A/RR_B**.

According to the invention bottoms evaporators are arranged at the bottom of the reaction column **RR_A/RR_B** and are then referred to as "**VS_{3A}**" and "**VS_{3B}**". Bottoms product stream **S_{AP}/S_{BP}** present in the reaction column **RR_A/RR_B** may be passed into such a bottoms evaporator and ROH at least partially removed therefrom to obtain a bottoms product stream **S_{AP}*** having an elevated mass fraction of M_AOR compared to **S_{AP}**/to obtain a bottoms product stream **S_{BP}*** having an elevated mass fraction of M_BOR compared to **S_{BP}**.

In step (a1) of the process according to the invention a bottoms product stream **S_{AP}** comprising ROH and M_AOR is withdrawn at the lower end of the reaction column **RR_A**.

It is preferable when the reaction column **RR_A** comprises at least one bottoms evaporator **VS_{3A}** through which the bottoms product stream **S_{AP}** is then at least partially passed to at least partially remove ROH, thus affording a bottoms product stream **S_{AP}*** having an elevated mass fraction of M_AOR compared to **S_{AP}**.

The mass fraction of M_AOR in the bottoms product stream **S_{AP}*** is especially elevated compared to the mass fraction of M_AOR in the bottoms product stream **S_{AP}** by at least 1%, preferably by $\geq 2\%$, more preferably by $\geq 5\%$, yet more preferably by $\geq 10\%$, yet still more preferably by $\geq 20\%$, yet still

more preferably by $\geq 30\%$, yet still more preferably by $\geq 40\%$, yet still more preferably by $\geq 50\%$, yet still more preferably by $\geq 100\%$, yet still more preferably by $\geq 150\%$.

- It is preferable when $\mathbf{S_{AP}}$ or, if at least one bottoms evaporator $\mathbf{VS_{3A}}$ through which the bottoms product stream $\mathbf{S_{AP}}$ is at least partially passed to at least partially remove ROH is used, $\mathbf{S_{AP^*}}$ has a mass fraction of $\mathbf{M_{AOR}}$ in ROH in the range from 1% to 50% by weight, preferably 5% to 32% by weight, more preferably 15% to 32% by weight, most preferably 30% to 32% by weight, in each case based on the total mass of $\mathbf{S_{AP}/S_{AP^*}}$.
- 5
- 10 The mass fraction of residual water in $\mathbf{S_{AP}/S_{AP^*}}$ is preferably $< 1\%$ by weight, preferably $< 0.1\%$ by weight, more preferably $< 0.01\%$ by weight, based on the total mass of $\mathbf{S_{AP}/S_{AP^*}}$.

- The mass fraction of reactant $\mathbf{M_{AOH}}$ in $\mathbf{S_{AP}/S_{AP^*}}$ is preferably $< 1\%$ by weight, preferably $< 0.1\%$ by weight, more preferably $< 0.01\%$ by weight, based on the total mass of $\mathbf{S_{AP}/S_{AP^*}}$.
- 15

4.2 Step (a2) of the process according to the invention (optional)

- According to the invention step (a2) is performed or else not performed. In the optional step (a2), which proceeds simultaneously with and spatially separately from step (a1) of the process
- 20 according to the invention, a reactant stream $\mathbf{S_{BE1}}$ comprising ROH is reacted with a reactant stream $\mathbf{S_{BE2}}$ comprising $\mathbf{M_{BOH}}$ in countercurrent at a pressure $\mathbf{p_{3B}}$ and a temperature $\mathbf{T_{3B}}$ in a reactive rectification column $\mathbf{RR_B}$ to afford a crude product $\mathbf{RP_B}$ comprising $\mathbf{M_{BOR}}$, water, ROH, $\mathbf{M_{BOH}}$.
- 25 In the optional step (a2) of the process according to the invention a bottoms product stream $\mathbf{S_{BP}}$ comprising ROH and $\mathbf{M_{BOR}}$ is withdrawn at the lower end of $\mathbf{RR_B}$. A vapour stream $\mathbf{S_{BB}}$ comprising water and ROH is withdrawn at the top end of $\mathbf{RR_B}$.
- $\mathbf{M_B}$ is selected from sodium, potassium, and preferably potassium.
- 30
- The reactant stream $\mathbf{S_{BE1}}$ comprises ROH. In a preferred embodiment the mass fraction of ROH in $\mathbf{S_{BE1}}$, based on the total mass of the reactant stream $\mathbf{S_{BE1}}$, is $\geq 95\%$ by weight, yet more preferably $\geq 99\%$ by weight, wherein $\mathbf{S_{BE1}}$ otherwise comprises especially water.
- 35 The alcohol ROH used as reactant stream $\mathbf{S_{BE1}}$ in the optional step (a2) of the process according to the invention can also be a commercial alcohol having a mass fraction of alcohol, based on the total mass of the reactant stream $\mathbf{S_{BE1}}$, of more than 99.8% by weight and a mass fraction of water, based on the total mass of the reactant stream $\mathbf{S_{BE1}}$, of up to 0.2% by weight.

The reactant stream S_{BE1} is preferably introduced in vapour form.

The reactant stream S_{BE2} comprises M_BOH . In a preferred embodiment S_{BE2} comprises not only M_BOH but also at least one further compound selected from water, ROH. It is yet more preferable when S_{BE2} comprises water in addition to M_BOH , thus rendering S_{BE2} an aqueous solution of M_BOH .

When the reactant stream S_{BE2} comprises M_BOH and water, the mass fraction of M_BOH , based on the total weight of the reactant stream S_{BE2} , is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight, most preferably 50% by weight.

When the reactant stream S_{BE2} comprises M_BOH and ROH, the mass fraction of M_BOH , based on the total weight of the reactant stream S_{BE2} , is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight.

In the particular case in which the reactant stream S_{BE2} comprises both water and ROH in addition to M_BOH , it is particularly preferable when the mass fraction of M_BOH , based on the total weight of the reactant stream S_{BE2} , is especially in the range from 10% to 55% by weight, preferably from 15% to 54% by weight, more preferably from 30% to 53% by weight and particularly preferably from 45% to 52% by weight.

Step (a2) of the process according to the invention is performed in a reactive rectification column (or "reaction column") RR_B . Preferred embodiments of the reaction column RR_B are described in section 4.1.

According to the invention "reaction of a reactant stream S_{BE1} comprising ROH with a reactant stream S_{BE2} comprising M_BOH in countercurrent" is especially achieved as a result of the feed point for at least a portion of the reactant stream S_{BE1} comprising ROH in the optional step (a2) being arranged below the feed point for the reactant stream S_{BE2} comprising M_BOH on the reaction column RR_B .

The reaction column RR_B preferably comprises at least 2, in particular 15 to 40, theoretical plates between the feed point of the reactant stream S_{BE1} and the feed point of the reactant stream S_{BE2} .

The reaction column RR_B is preferably operated as a pure stripping column. Accordingly the reactant stream S_{BE1} comprising ROH is especially supplied in vaporous form in the lower region of the reaction column RR_B . The optional step (a2) of the process according to the invention also encompasses the case of part of the reactant stream S_{BE1} comprising ROH being introduced in

vapour form below the feed point of the reactant stream **S_{BE2}** comprising aqueous sodium hydroxide solution **M_BOH** but nevertheless at the upper end or in the region of the upper end of the reaction column **RR_B**. This makes it possible to reduce the dimensions in the lower region of the reaction column **RR_B**. When a portion of the reactant stream **S_{BE1}** comprising ROH, in particular methanol, is added especially in vaporous form at the upper end or in the region of the upper end of the reaction column **RR_B** only a fraction of in particular 10% to 70% by weight, preferably of 30% to 50% by weight (in each case based on the total amount of the alcohol ROH employed in the optional step (a2)) is supplied at the lower end of the reaction column **RR_B** and the remaining fraction is added in vaporous form in a single stream or divided into a plurality of substreams, preferably 1 to 10 theoretical trays, particularly preferably 1 to 3 theoretical trays, below the feed point of the reactant stream **S_{BE2}** comprising **M_BOH**.

In the reaction column **RR_B** the reactant stream **S_{BE1}** comprising ROH is then reacted with the reactant stream **S_{BE2}** comprising **M_BOH** according to the reaction <1> described hereinabove to afford **M_BOR** and **H₂O**, where these products are present in admixture with the reactants ROH and **M_BOH** since an equilibrium reaction is concerned. Accordingly a crude product **RP_B** which contains not only the products **M_BOR** and water but also ROH and **M_BOH** is obtained in the reaction column **RR_B** in optional step (a2) of the process according to the invention.

The bottoms product stream **S_{BP}** comprising ROH and **M_BOR** is then obtained and withdrawn at the lower end of **RR_B**.

A water-containing alcohol stream, presently referred to as "vapour stream **S_{BB}** comprising water and ROH", is withdrawn at the upper end of **RR_B**, preferably at the top of **RR_B**.

This vapour stream **S_{BB}** comprising water and ROH is supplied to step (b) of the process according to the invention. Said stream is mixed with **S_{AB}** before being supplied to step (b) of the process according to the invention or is not, i.e. is supplied to step (b) of the process according to the invention separately from **S_{AB}**. Vapour stream **S_{BB}** is preferably mixed with **S_{AB}** and the resulting mixed vapour stream is then introduced into step (b) of the process according to the invention.

The amount of the alcohol ROH comprised by the reactant stream **S_{BE1}** is preferably selected so that it simultaneously serves as solvent for the alkali metal alkoxide **M_BOR** present in the bottoms product stream **S_{BP}**. The amount of the alcohol ROH in the reactant stream **S_{BE1}** is preferably chosen to achieve in the bottom of the reaction column the desired concentration of the alkali metal alkoxide solution which is withdrawn as a bottoms product stream **S_{BP}** comprising ROH and **M_BOR**.

In a preferred embodiment of optional step (a2) of the process according to the invention, and especially in the cases where **S_{BE2}** also comprises water in addition to **M_BOH**, the ratio of the total weight (masses; units: kg) of alcohol ROH employed in step (a2) as reactant stream **S_{BE1}** to the

total weight (masses; unit: kg) of M_BOH employed in step (a2) as reactant stream S_{BE2} is 1: 1 to 50: 1, more preferably 5: 1 to 48: 1, yet more preferably 9: 1 to 35: 1, yet still more preferably 10: 1 to 30: 1, yet still more preferably 13: 1 to 22: 1, most preferably 14: 1.

- 5 The reaction column RR_B is operated with or without, preferably without, reflux.

"Without reflux" means that the vapour stream S_{BB} withdrawn at the upper end of RR_B comprising water and ROH is completely supplied to the rectification column RD_1 according to step (b). The vapour stream S_{BB} comprising water and ROH is preferably supplied to the rectification column
10 RD_1 in vaporous form.

"With reflux" means that the vapour stream S_{BB} withdrawn at the upper end of the respective column, the reaction column RR_B in step (a2), comprising water and ROH is not completely discharged, i.e. is not completely supplied to the first rectification column RD_1 in step (b), but rather
15 is at least partially, preferably partially, recycled to the respective column, i.e. the reaction column RR_B in step (a2), as reflux. In the cases where such a reflux is established the reflux ratio is preferably 0.05 to 0.99, more preferably 0.1 to 0.9, yet more preferably 0.11 to 0.34, particularly preferably 0.14 to 0.27 and very particularly preferably 0.17 to 0.24. A reflux may be established by attaching at the top of the respective column, the reaction column RR_B in step (a2), a condenser
20 K_{RRB} in which the vapour stream S_{BB} is at least partially condensed and sent back to the respective column, the reaction column RR_B in step (a2).

In the embodiment in which a reflux is established on the reaction column RR_B the alcohol M_BOH employed in optional step (a2) as reactant stream S_{BE2} may also be at least partially, preferably
25 partially, mixed with the reflux stream and the resulting mixture thus be supplied to step (a2).

Optional step (a2) of the process according to the invention is in particular performed at a temperature T_{3B} in the range from 25°C to 200°C, preferably in the range from 45°C to 150°C, more preferably in the range from 47°C to 120°C, more preferably in the range from 60°C to 110°C.

30

Optional step (a2) of the process according to the invention is in particular performed at a pressure p_{3B} of 0.5 bar to 40 bar, preferably in the range from 0.75 bar to 5 bar, more preferably in the range from 1 bar to 2 bar, more preferably in the range from 1 bar to 1.8 bar, yet more preferably at 1.1 bar to 1.6 bar. It is an essential feature of the invention that when establishing the pressure p_{3B} : p_2
35 $> p_{3B}$. It is especially also the case that $p_{3B} > p_1$.

In a preferred embodiment the reaction column RR_B comprises at least one evaporator which is in particular selected from intermediate evaporators VZ_B and bottoms evaporators VS_B . The reaction column RR_B particularly preferably comprises at least one bottoms evaporator VS_{3B} .

In optional step (a2) of the process according to the invention a bottoms product stream S_{BP} comprising ROH and M_BOR is withdrawn at the lower end of the reaction column RR_B .

5 It is preferable when the reaction column RR_B comprises at least one bottoms evaporator VS_{3B} through which the bottoms product stream S_{BP} is then at least partially passed to at least partially remove ROH, thus affording a bottoms product stream S_{BP^*} having an elevated mass fraction of M_BOR compared to S_{BP} .

10 The mass fraction of M_BOR in the bottoms product stream S_{BP^*} is especially elevated compared to the mass fraction of M_BOR in the bottoms product stream S_{BP} by at least 1%, preferably by $\geq 2\%$, more preferably by $\geq 5\%$, yet more preferably by $\geq 10\%$, yet still more preferably by $\geq 20\%$, yet still more preferably by $\geq 30\%$, yet still more preferably by $\geq 40\%$, yet still more preferably by $\geq 50\%$, yet still more preferably by $\geq 100\%$, yet still more preferably by $\geq 150\%$.

15 It is preferable when S_{BP} or, if at least one bottoms evaporator VS_{3B} through which the bottoms product stream S_{BP} is at least partially passed to at least partially remove ROH, S_{BP^*} has a mass fraction of M_BOR in ROH in the range from 1% to 50% by weight, preferably 5% to 32% by weight, more preferably 10% to 32% by weight, most preferably 15% to 30% by weight, in each case based on the total mass of S_{BP}/S_{BP^*} .

20

The mass fraction of residual water in S_{BP}/S_{BP^*} is preferably $< 1\%$ by weight, preferably $< 0.1\%$ by weight, more preferably $< 0.01\%$ by weight, based on the total mass of S_{BP}/S_{BP^*} .

25 The mass fraction of reactant M_BOH in S_{BP}/S_{BP^*} is preferably $< 1\%$ by weight, preferably $< 0.1\%$ by weight, more preferably $< 0.01\%$ by weight, based on the total mass of S_{BP}/S_{BP^*} .

30 In the embodiments of the present process in which step (a2) is also performed, it is preferable when the bottoms product stream S_{AP} is at least partially passed through a bottoms evaporator VS_{3A} and ROH is at least partially removed from S_{AP} to afford a bottoms product stream S_{AP^*} having an elevated mass fraction of M_AOR compared to S_{AP} and/or, preferably and, the bottoms product stream S_{BP} is at least partially passed through a bottoms evaporator VS_{3B} and ROH is at least partially removed from S_{BP} to afford a bottoms product stream S_{BP^*} having an elevated mass fraction of M_BOR compared to S_{BP} .

35 In the embodiments of the present invention in which it is performed, step (a2) of the process according to the invention is performed simultaneously with and spatially separate from step (a1). Spatial separation is ensured by performing steps (a1) and (a2) in the two reaction columns RR_A and RR_B .

In an advantageous embodiment of the invention the reaction columns **RR_A** and **RR_B** are accommodated in one column shell, where the column is at least partially subdivided by at least one dividing wall. Such a column having at least one dividing wall will according to the invention be referred to as "**DWC**". Such dividing wall columns are familiar to those skilled in the art and are described for example in US 2,295,256, EP 0 122 367 A2, EP 0 126 288 A2, WO 2010/097318 A1 and I. Dejanović, Lj. Matijašević, Ž. Olujić, *Chemical Engineering and Processing* **2010**, *49*, 559-580. In the dividing wall columns suitable for the process according to the invention, the dividing walls preferably extend to the column floor and, in particular, preferably span at least a quarter, more preferably at least a third, yet more preferably at least half, yet more preferably at least two thirds, yet still more preferably at least three quarters, of the column by height. They divide the columns into at least two reaction spaces in which spatially separate reactions may be carried out. The reaction spaces provided by the at least one dividing wall may be of identical or different sizes.

In this embodiment the bottoms product streams **S_{AP}** and **S_{BP}** may be separately withdrawn in the respective regions separated by the dividing wall and preferably passed through the bottoms evaporator **VS_{3A}/ VS_{3B}** attached for each reaction space formed by the at least one reaction wall in which ROH is at least partially removed from **S_{AP}/S_{BP}** to afford **S_{AP*}/S_{BP*}**.

4.3 Step (b) of the process according to the invention

In step (b) of the process according to the invention the vapour stream **S_{AB}** and, if step (a2) is performed, the vapour stream **S_{BB}**, in admixture with **S_{AB}** or separately from **S_{AB}**, is passed into a first rectification column **RD₁**, to obtain a mixture **G_{RD1}** comprising water and ROH in the rectification column **RD₁**.

In the optional embodiment of the process according to the invention in which step (a2) is performed, the vapour stream **S_{BB}** is preferably mixed with **S_{AB}** and the obtained mixed vapour **S_{ABB}** then introduced into a rectification column **RD₁**.

In one embodiment of the present invention (when $p_{3A} < p_1/p_{3B} < p_1$) the vapour stream **S_{AB}** and, in the cases where the optional step (a2) is performed, the vapour stream **S_{BB}** may be compressed before they are passed into the rectification column **RD₁**. This may be effected via a compressor **VD₃₁**. However, in the embodiments of the present invention in which $p_{3A} > p_1$ and $p_{3B} > p_1$ the provision of a compressor **VD₃₁** is not necessary, and it is then possible to save on the provision thereof and thus the electrical energy required therefor.

It will be appreciated that even in the embodiments in which the optional step (a2) is performed, and **S_{BB}** is introduced into the rectification column **RD₁** separately from **S_{AB}**, **S_{AB}** and **S_{BB}** undergo

mixing in the rectification column **RD₁** with the result that a mixture **G_{RD1}** comprising water and ROH is always obtained in the first rectification column **RD₁** after performance of step (b).

Any desired rectification column known to those skilled in the art may be employed as rectification column **RD₁** in step (b) of the process according to the invention. The rectification column **RD₁** preferably contains internals. Suitable internals are, for example, trays, unstructured packings or structured packings. As trays, use is normally made of bubble cap trays, sieve trays, valve trays, tunnel trays or slit trays. Unstructured packings are generally beds of random packing elements. Random packing elements normally used are Raschig rings, Pall rings, Berl saddles or Intalox® saddles. Structured packings are for example marketed under the trade name Mellapack® from Sulzer. Apart from the internals mentioned, further suitable internals are known to a person skilled in the art and can likewise be used.

Preferred internals have a low specific pressure drop per theoretical plate. Structured packings and random packing elements have, for example, a significantly lower pressure drop per theoretical plate than trays. This has the advantage that the pressure drop in the rectification column remains as low as possible and thus the mechanical power of the compressor and the temperature of the alcohol/water mixture **G_{RD1}** to be evaporated remains low.

When the rectification column **RD₁** contains structured packings or unstructured packings these may be divided or in the form of an uninterrupted packing. However, typically at least two packings are provided, one packing above the feed point of the vapour stream **S_{AB}**/the feed points of the two vapour streams **S_{AB}** and **S_{BB}** and one packing below the feed point of the vapour stream **S_{AB}**/the feed points of the two vapour streams **S_{AB}** and **S_{BB}**/the feed point of the mixed vapour **S_{ABB}**. If an unstructured packing is used, for example a random packing, the random packing elements are typically disposed on a suitable sieve tray or mesh tray.

At the end of step (b) of the process according to the invention, a mixture **G_{RD1}** comprising water and ROH is finally obtained in the rectification column **RD₁**. The composition of the mixture **G_{RD1}** results in particular from the composition of the vapour stream **S_{AB}** and, if step (a2) is performed, partly from the composition of the two vapour streams **S_{AB}** and **S_{BB}** in particular.

4.4 Step (c) of the process according to the invention

In step (c) of the process according to the invention the mixture **G_{RD1}** comprising water and ROH is in the first rectification column **RD₁** at a pressure **p₁** and a temperature **T₁** separated into an ROH-comprising vapour stream **S_{RDB1}** at the upper end (= top) of **RD₁** and a bottoms stream **S_{RDS1}** comprising water and ROH at the lower end (= bottom) of **RD₁**.

With the exception of the proviso that $p_2 > p_1$, the pressure p_1 in RD_1 may be chosen by those skilled in the art according to their knowledge of the art. It is preferably in the range between 1 bar and 20 bar, preferably 1 bar and 15 bar, more preferably 1 to 10 bar, yet more preferably 1.00 to 3.00 bar, yet more preferably 1.00 to 2.00 bar, yet more preferably 1.10 to 1.50 bar, wherein,
 5 simultaneously, $p_2 > p_1$.

The temperature T_1 in RD_1 may be chosen by those skilled in the art according to their knowledge of the art. It is preferably in the range from 40°C to 220°C, preferably from 60°C to 190°C.

10 In a preferred embodiment, $p_{3A} > p_1$ and, in the cases where step (a2) is performed, in addition $p_{3B} > p_1$. As a result of this established pressure the total energy demand of the process is surprisingly minimized compared to the embodiments where $p_{3A} < p_1/p_{3B} < p_1$.

The separation according to step (c) of the process according to the invention is a distillative
 15 separation of the alcohol/water mixture G_{RD1} as is known to those skilled in the art.

At the lower end (also: "bottom") of the rectification column RD_1 a bottoms stream S_{RDS1} still comprising alcohol ROH is obtained. S_{RDS1} comprises ROH in a mass fraction of in particular 0.005% to 95% by weight, preferably 25% to 95% by weight, based on the total mass of S_{RDS1} .
 20 S_{RDS1} preferably comprises essentially water in addition to the alcohol ROH.

In a preferred embodiment of the invention S_{RDB1} is at least partially employed as reactant stream S_{AE1} in the reactive rectification column RR_A and, if step (a2) is performed, alternatively or in addition employed as reactant stream S_{BE1} in the reactive rectification column RR_B .
 25

Also obtained at the top of the rectification column RD_1 is the vapour stream S_{RDB1} comprising ROH. The preferred mass fraction of ROH in this vapour stream S_{RDB1} is $\geq 99\%$ by weight, preferably $\geq 99.6\%$ by weight, more preferably $\geq 99.9\%$ by weight, in each case based on the total mass of S_{RDB1} , wherein the remainder is especially water.
 30

In step (c) the vapour S_{AB} or S_{AB} and S_{BB} obtained in step (a1) or step (a1) and (a2) is subjected to distillative separation. These vapours comprise essentially the alcohol ROH and water. In particular, S_{AB} or S_{AB} and S_{BB} are each a water/alcohol mixture in which the mass fraction of ROH is preferably in the range $> 80\%$ by weight, more preferably $> 85\%$ by weight, yet more preferably $> 90\%$ by weight (based on the total mass of S_{AB} or S_{AB} and S_{BB}). Thus in particular G_{RD1} too is an alcohol/water mixture in which the mass fraction of ROH is preferably in the range $> 80\%$ by weight, more preferably $> 95\%$ by weight, yet more preferably $> 90\%$ by weight (based on the total mass of G_{RD1}).
 35

4.5 Step (d) of the process according to the invention

In step (d) of the process according to the invention the bottoms stream S_{RDS1} is completely or partially, preferably partially, passed into a second rectification column RD_2 .

5 This affords a mixture G_{RD2} comprising water and ROH in the second rectification column RD_2 .

In the embodiment of the present invention in which S_{RDS1} is partially passed into RD_2 , this is especially performed such that a first portion S_{RDS11} of the bottoms stream S_{RDS1} discharged from the first rectification column RD_1 is passed into a second rectification column RD_2 , and a second
10 portion S_{RDS12} of the bottoms stream S_{RDS1} discharged from the first rectification column RD_1 is recycled into the first rectification column RD_1 . It is yet more preferable when energy is transferred to S_{RDS12} , yet still more preferable when S_{RDS12} is heated. Once S_{RDS12} has been recycled to RD_1 it undergoes mixing in RD_1 with G_{RD1} and thus provides energy for separating G_{RD1} according to step (c).
15

In this preferred embodiment of step (d) of the process according to the invention it is yet more preferable when the ratio of the masses (in kg) of S_{RDS11} to S_{RDS12} are in the range 9: 1 to 1:9, yet more preferably 4:1 to 1: 4, yet more preferably 7: 3 to 3: 7, yet more preferably 3: 2 to 2: 3, yet more preferably 1: 1.
20

In this preferred embodiment of step (d) of the process according to the invention it is possible to supply energy to the stream S_{RDS12} . In a preferred embodiment this is effected when the stream S_{RDS12} is passed through a bottoms evaporator VS_{RD1} in which energy is transferred from S_{RDB2} or another heat transfer medium to S_{RDS12} . This energy transfer may advantageously be undertaken
25 when S_{RDS12} and S_{RDB2}/S_{RDS12} and the heat transfer medium are passed through a bottoms evaporator VS_{RD1} . After the recycling of S_{RDS12} into the reaction column RR_A , S_{RDS12} then transfers the energy to G_{RD1} .

Any desired rectification column known to those skilled in the art may be employed as rectification
30 column RD_2 in step (d) of the process according to the invention. The rectification column RD_2 preferably contains internals. Suitable internals are, for example, trays, unstructured packings or structured packings. As trays, use is normally made of bubble cap trays, sieve trays, valve trays, tunnel trays or slit trays. Unstructured packings are generally beds of random packing elements. Random packing elements normally used are Raschig rings, Pall rings, Berl saddles or Intalox®
35 saddles. Structured packings are for example marketed under the trade name Mellapack® from Sulzer. Apart from the internals mentioned, further suitable internals are known to a person skilled in the art and can likewise be used.

Preferred internals have a low specific pressure drop per theoretical plate. Structured packings and
40 random packing elements have, for example, a significantly lower pressure drop per theoretical

plate than trays. This has the advantage that the pressure drop in the rectification column **RD₂** remains as low as possible and thus the mechanical power of the compressor and the temperature of the alcohol/water mixture **G_{RD2}** to be evaporated remains low.

- 5 When the rectification column **RD₂** contains structured packings or unstructured packings, these may be divided or in the form of an uninterrupted packing. However, typically at least two packings are provided, one packing above the feed point of the stream **S_{RDS1}**/the portion of **S_{RDS1}**, in particular **S_{RDS12}**, and one packing below the relevant feed point. If an unstructured packing is used, for example a random packing, the random packing elements typically rest on a suitable sieve tray
10 or mesh tray.

S_{RDS1}/the portion of **S_{RDS1}** which is passed into **RD₂** and which is preferably **S_{RDS12}** is at least partially liquid.

- 15 It is thus further preferable to pass this stream into **RD₂** via a liquid compressor or a pump **P** since according to the invention **RD₂** is at the highest pressure ($p_2 > p_1$; $p_2 > p_{3A}$; $p_2 > p_{3B}$).

- At the end of step (d) of the process according to the invention, a mixture **G_{RD2}** comprising water and ROH is finally obtained in the rectification column **RD₂**. The composition of the mixture **G_{RD2}**
20 results especially from the composition of the stream **S_{RDS1}**/the portion of the stream **S_{RDS1}**, preferably **S_{RDS12}**, which is passed into **RD₂**.

4.6 Step (e) of the process according to the invention

- In step (e) of the process according to the invention the mixture **G_{RD2}** comprising water and ROH is
25 at a pressure **p₂** and a temperature **T₂** separated into an ROH-comprising vapour stream **S_{RDB2}** at the top of **RD₂** and a bottoms stream **S_{RDS2}** comprising water and optionally ROH at the bottom of **RD₂**.

- With the exception of the proviso that $p_2 > p_1$, the pressure **p₂** in **RD₂** may be chosen by those
30 skilled in the art according to their knowledge of the art. It is preferably in the range between 1 bar and 20 bar, preferably 1 bar and 15 bar, more preferably 1 to 10 bar, yet more preferably 3.00 bar to 9.00 bar, yet more preferably 3.20 to 8.90 bar, wherein, simultaneously, $p_2 > p_1$.

- The temperature **T₂** in **RD₂** may be chosen by those skilled in the art according to their knowledge
35 of the art. It is preferably in the range from 40°C to 220°C, preferably from 60°C to 190°C.

The separation according to step (e) of the process according to the invention is a distillative separation of the alcohol/water mixture **G_{RD2}** as is known to a person skilled in the art.

Obtained at the bottom of the rectification column **RD₂** is a stream **S_{RDS2}** which may comprise <1% by weight of alcohol, based on the total mass of **S_{RDS2}**.

Also obtained at the top of the rectification column **RD₂** is the vapour stream **S_{RDB2}** comprising ROH. The preferred mass fraction of ROH in this vapour stream **S_{RDB2}** is $\geq 99\%$ by weight, preferably $\geq 99.6\%$ by weight, more preferably $\geq 99.9\%$ by weight, in each case based on the total mass of **S_{RDB2}**, wherein the remainder is especially water.

In a preferred embodiment of the present invention **S_{RDB2}** is at least partially employed as reactant stream **S_{AE1}** in the reactive rectification column **RR_A** and, if step (a2) is performed, alternatively or in addition employed as reactant stream **S_{BE1}** in the reactive rectification column **RR_B**.

In step (e) the stream **S_{RDS1}**, preferably the portion **S_{RDS12}**, completely or partially passed into the second rectification column **RD₂** in step (d) is subjected to distillative separation.

4.7 Pressure management as a characterizing feature

The process according to the invention is characterized in that during operation of the rectification columns **RD₁** (step (c)) and **RD₂** (step (e)) a certain pressure ratio is established.

Accordingly, $p_2 > p_1$, $p_2 > p_{3A}$ and, in the cases where step (a2) is performed, $p_2 > p_{3B}$.

It has surprisingly been found that maintaining these pressures allows the demand for externally supplied electrical energy to be minimized and the majority of the energy required for the process to be covered by heating steam.

It is yet more advantageous when in addition the pressures are established such that $p_{3A} > p_1$ and, in the cases where step (a2) is performed, $p_{3B} > p_1$. Establishing the pressures p_{3A} and p_{3B} in such a way reduces the altogether required energy demand compared to the case where $p_{3A} < p_1/p_{3B} < p_1$.

4.8 Characterizing step (f): Energy transfer from **S_{RDB2}** to **G_{RD1}**

The step (f) of the process according to the invention which is characterizing in addition to the pressure regime is that energy is transferred from **S_{RDB2}** to the mixture **G_{RD1}** in the first rectification column **RD₁**.

According to the invention "energy transfer" is in particular to be understood as meaning "heat transfer".

This step (f) and the pressure regime according to the invention allow a particularly advantageous integration of the energy which would otherwise dissipate, which makes it possible to cover a particularly large portion of the energy demand of the process through the use of low-energy heating steam instead of through electrical current. This makes the process according to the invention particularly energy-efficient.

According to the invention the transfer of energy from S_{RDB2} to G_{RD1} in RD_1 may be effected in various ways familiar to those skilled in the art and preferably comprises heating G_{RD1} in RD_1 with S_{RDB2} , for example via a heat exchanger WT .

According to the invention in step (f) the energy is especially transferred from S_{RDB2} to G_{RD1} in RD_1 directly or indirectly, preferably directly.

4.8.1 Direct energy transfer from S_{RDB2} to G_{RD1} in RD_1

According to the invention "direct energy transfer from S_{RDB2} to G_{RD1} in RD_1 " means that an energy transfer, preferably heating, of G_{RD1} in RD_1 with S_{RDB2} is effected such that G_{RD1} is contacted with S_{RDB2} without G_{RD1} undergoing mixing with S_{RDB2} , thus transferring energy from S_{RDB2} to G_{RD1} . However, according to the invention, cases of direct energy transfer are to be understood as also including cases where an energy transfer, preferably heating, of a stream S_X discharged from RD_1 with S_{RDB2} is effected without S_{RDB2} undergoing mixing with S_X , thus transferring energy from S_{RDB2} to S_X , and S_X is then passed back into RD_1 where it undergoes mixing with G_{RD1} in RD_1 and thus transfers the energy absorbed from S_{RDB2} to G_{RD1} in RD_1 .

In a particular embodiment of the present invention, S_X is selected from the group consisting of S_{RDS12} , S_{RDX1} .

Contacting without mixing is achieved by processes known to those skilled in the art, for example by contacting via a dividing wall made of metal, plastic etc., in particular in the heat exchanger WT , preferably a condenser K or evaporator V which is in particular selected from bottoms evaporators VS and intermediate evaporators VZ .

According to the invention it is preferable when direct energy transfer from S_{RDB2} to the mixture G_{RD1} in the first rectification column RD_1 is performed according to at least one of the steps (α -i), (α -ii), (α -iii), more preferably performed according to at least one of the steps (α -i), (α -ii).

(α -i) A first portion S_{RDS11} of the bottoms stream S_{RDS1} discharged from RD_1 is passed into the second rectification column RD_2 , and energy is transferred from S_{RDB2} to a second portion S_{RDS12} of the bottoms stream S_{RDS1} discharged from RD_1 , preferably via a heat exchanger WT , and S_{RDS12} is then recycled into RD_1 . This step (α -i) also comprises embodiments in which energy is initially

transferred from S_{RDB2} , preferably via a heat exchanger WT , to the overall bottoms stream S_{RDS1} , and the bottoms stream S_{RDS1} is only then divided into S_{RDS11} and S_{RDS12} , and S_{RDS11} is then passed into RD_2 and S_{RDS12} is recycled into RD_1 .

- 5 (α-ii) At least one stream S_{RDX1} distinct from S_{RDB1} and S_{RDS1} comprising ROH and water is discharged from RD_1 , energy is then transferred from S_{RDB2} to S_{RDX1} , preferably via a heat exchanger WT , and S_{RDX1} is recycled into RD_1 .

10 It is preferable when S_{RDX1} is withdrawn below the vapour stream S_{RDB1} on RD_1 . S_{RDX1} is then especially selected from bottoms stream S_{RDX1S} , intermediate stream S_{RDX1Z} .

A bottoms stream S_{RDX1S} is a stream whose withdrawal point on RD_1 is at the same height or below the withdrawal point of S_{RDS1} . S_{RDX1S} may then be passed through a heat exchanger WT , in particular a bottoms evaporator VS , and energy may be transferred from S_{RDB2} to S_{RDX1S} therein.

15 An intermediate stream S_{RDX1Z} is a stream whose withdrawal point on RD_1 is between the withdrawal points of S_{RDB1} and S_{RDS1} . S_{RDX1Z} may then be discharged from RD_1 and passed through a heat exchanger WT , in particular an intermediate evaporator VZ , and energy may be transferred from S_{RDB2} to S_{RDX1Z} therein.

20 (α-iii) S_{RDB2} is passed through RD_1 , thus transferring energy from S_{RDB2} to G_{RD1} , preferably via a heat exchanger WT . Such an embodiment may be realized for example when S_{RDB2} is passed through the rectification column RD_1 through a conduit whose surface S_{RDB2} transfers energy to G_{RD1} in RD_1 .

25 4.8.1 Indirect energy transfer from S_{RDB2} to G_{RD1} in RD_1

According to the invention "indirect energy transfer from S_{RDB2} to G_{RD1} in RD_1 " means that an energy transfer, preferably heating, of G_{RD1} with S_{RDB2} is effected in RD_1 such that G_{RD1} is not directly contacted with S_{RDB2} but rather at least one additional, preferably precisely one additional, heat transfer medium W_1 distinct from G_{RD1} and S_{RDB2} is employed which during energy transfer from S_{RDB2} to G_{RD1} in RD_1 undergoes mixing neither with S_{RDB2} nor with G_{RD1} in RD_1 . Energy is transferred from S_{RDB2} to the at least one heat transfer medium W_1 without S_{RDB2} and the at least one heat transfer medium W_1 undergoing mixing, and then transferred from the at least one heat transfer medium W_1 to G_{RD1} in RD_1 without the at least one heat transfer medium W_1 and G_{RD1} undergoing mixing.

According to the invention, cases of indirect energy transfer are to be understood as also including cases where energy is transferred from S_{RDB2} to the at least one, preferably precisely one, heat transfer medium W_1 without S_{RDB2} and the at least one heat transfer medium W_1 undergoing

mixing, and subsequently an energy transfer, preferably heating, of a stream S_X discharged from RD_1 with the at least one heat transfer medium W_1 is effected without the at least one heat transfer medium W_1 undergoing mixing with S_X , thus transferring energy from the at least one heat transfer medium W_1 to S_X , and S_X is then recycled into RD_1 where it undergoes mixing with G_{RD1} in RD_1 and thus transfers the energy absorbed by S_{RDB2} via the at least one heat transfer medium W_1 to G_{RD1} in RD_1 .

In a particular embodiment of the present invention, S_X is selected from the group consisting of S_{RDS12} , S_{RDX1} .

"At least one heat transfer medium W_1 " comprises the cases where the energy of W_1 is first transferred to one or more further heat transfer media W_2 , W_3 , W_4 , W_5 etc. distinct from G_{RD1} and S_{RDB2} and the last of these heat transfer media, referred to as " W_Y " is contacted with G_{RD1} in RD_1 , thus transferring energy, preferably heat, from W_Y to G_{RD1} but without W_Y and G_{RD1} undergoing mixing. Energy, preferably heat, may likewise be transferred from W_Y to a stream S_X discharged from RD_1 without W_Y and S_X undergoing mixing, and S_X subsequently recycled into RD_1 where it undergoes mixing with G_{RD1} in RD_1 , thus transferring the energy absorbed by W_Y to G_{RD1} in RD_1 .

The described contacting is in each case preferably performed in a heat exchanger WT , preferably a condenser K or evaporator V , which is especially selected from bottoms evaporators VS and intermediate evaporators VZ .

According to the invention it is preferable when indirect energy transfer from S_{RDB2} to the mixture G_{RD1} in the first rectification column RD_1 is performed according to at least one of the steps (β -i), (β -ii), (β -iii), more preferably performed according to at least one of the steps (β -i), (β -ii).

(β -i) A first portion S_{RDS11} of the bottoms stream S_{RDS1} discharged from RD_1 is passed into the second rectification column RD_2 , and a second portion S_{RDS12} of the bottoms stream S_{RDS1} discharged from RD_1 is recycled into RD_1 . Energy is transferred from S_{RDB2} to at least one, preferably precisely one, heat transfer medium W_{i1} distinct from S_{RDS12} and then transferred from the at least one heat transfer medium W_{i1} to S_{RDS12} , and then S_{RDS12} is recycled into RD_1 . This step (β -i) also comprises embodiments in which energy is initially transferred from the at least one, preferably precisely one, heat transfer medium W_{i1} distinct from S_{RDS12} , preferably via a heat exchanger WT , to the overall bottoms stream S_{RDS1} , and the bottoms stream S_{RDS1} is only then divided into S_{RDS11} and S_{RDS12} , and S_{RDS11} is then passed into RD_2 and S_{RDS12} is recycled into RD_1 .

(β -ii) At least one stream S_{RDX1} distinct from S_{RDB1} and S_{RDS1} comprising ROH and water is discharged from RD_1 . Energy is transferred from S_{RDB2} to at least one, preferably precisely one, heat transfer medium W_{i1} distinct from S_{RDX1} , preferably via a heat exchanger WT , and then

transferred from the at least one heat transfer medium W_{ii1} to S_{RDX1} , and S_{RDX1} is then recycled into RD_1 .

It is preferable when S_{RDX1} is withdrawn below the vapour stream S_{RDB1} on RD_1 . S_{RDX1} is then especially selected from bottoms stream S_{RDX1S} , intermediate stream S_{RDX1Z} .

A bottoms stream S_{RDX1S} is a stream whose withdrawal point on RD_1 is at the same height or below the withdrawal point of S_{RDS1} . S_{RDX1S} may then be passed through a heat exchanger WT , in particular a bottoms evaporator VS , and energy may be transferred from S_{RDB2} to S_{RDX1S} therein.

An intermediate stream S_{RDX1Z} is a stream whose withdrawal point on RD_1 is between the withdrawal points of S_{RDB1} and S_{RDS1} . S_{RDX1Z} may then be discharged from RD_1 and passed through a heat exchanger WT , in particular an intermediate evaporator VZ , and energy may be transferred from S_{RDB2} to S_{RDX1Z} therein.

(β -iii) Energy is transferred from S_{RDB2} to at least one heat transfer medium W_{iii1} distinct from G_{RD1} and the at least one heat transfer medium W_{iii1} is then passed through RD_1 , thus transferring energy from the at least one heat transfer medium W_{iii1} to G_{RD1} .

Such an embodiment may be realized for example when the at least one heat transfer medium W_{iii1} is passed through the rectification column RD_1 through a conduit whose surface transfers energy from the at least one heat transfer medium W_{iii1} to G_{RD1} in RD_1 .

Employable heat transfer media W_1, W_2, W_3, W_4, W_5 /at least one heat transfer medium W_{i1} /at least one heat transfer medium W_{ii1} /at least one heat transfer medium W_{iii1} include any heat transfer media known to those skilled in the art. Such heat transfer media are preferably selected from the group consisting of water; alcohol-water solutions; salt-water solutions, also including ionic liquids, such as for example LiBr solutions, dialkylimidazolium salts, such as especially dialkylimidazolium dialkylphosphates; mineral oils, such as for example diesel oils; thermal oils such as for example silicone oils; biological oils such as for example limonene; aromatic hydrocarbons such as for example dibenzyltoluene. The most preferred heat transfer medium is water.

Salt-water solutions that may be used are also described for example in DE 10 2005 028 451 A1 and WO 2006/134015 A1.

4.9 Addition of fresh alcohol

The alcohol ROH is consumed in the process according to the invention, and especially in a continuous process mode therefore requires replacement with fresh alcohol ROH.

Fresh alcohol is in particular added to at least one of the columns selected from rectification column **RD₁**, rectification column **RD₂**, reactive rectification column **RR_A** and, if step (a2) is performed, alternatively or in addition added to the reactive rectification column **RR_B**.

- 5 In a preferred embodiment of the present invention a stream **S_{XE1}** distinct from **S_{AE1}** and **S_{BE1}** comprising ROH is accordingly added to at least one of the columns selected from rectification column **RD₁**, rectification column **RD₂**, reactive rectification column **RR_A** and, if step (a2) is performed, alternatively or in addition added to reactive rectification column **RR_B**.
- 10 The supply of the fresh alcohol ROH is especially effected directly as reactant stream **S_{AE1}** comprising ROH into the reaction column **RR_A** or in the embodiments in which step (a2) is performed into the reaction columns **RR_A** and **RR_B**.

- In the process according to the invention it is further preferable to employ the ROH-comprising vapour stream **S_{RDB1}** at least partially as reactant stream **S_{AE1}** in step (a1) and optionally as reactant stream **S_{BE1}** in step (a2). The vapour stream **S_{RDB2}** may alternatively or in addition be employed at least partially as reactant stream **S_{AE1}** in step (a1) and optionally as reactant stream **S_{BE1}** in step (a2).
- 15

- 20 In the particularly preferred embodiment in which **S_{RDB1}** and **S_{RDB2}** are employed at least partially as reactant stream **S_{AE1}** in step (a1) and optionally as reactant stream **S_{BE1}** in step (a2), **S_{RDB1}** and **S_{RDB2}** may be supplied to the respective reactive rectification column **RR_A/RR_B** separately from one another or first mixed with one another and then supplied to the respective reactive rectification column **RR_A/RR_B**. **S_{RDB1}** and **S_{RDB2}** are preferably firstly mixed with one another and then supplied
- 25 to the respective reactive rectification column **RR_A/RR_B**.

In this preferred embodiment it is yet more preferable when the fresh alcohol ROH is added to one of the rectification columns **RD₁**, **RD₂**, preferably **RD₁**.

- 30 When the fresh alcohol ROH is added to the rectification column **RD₁** or **RD₂** it is preferably supplied either in the rectifying section of the respective rectification column or directly at the top of the respective rectification column. The optimal feed point depends on the water content of the employed fresh alcohol and also on the desired residual water content in the vapour stream **S_{RDB1}/S_{RDB2}**. The higher the proportion of water in the employed alcohol and the higher the purity requirement in the vapour stream **S_{RDB1}/S_{RDB2}**, the more advantageous is a feed a number of
- 35 theoretical trays below the top of the rectification column **RD₁/RD₂**. Up to 20 theoretical trays below the top of the rectification column **RD₁/RD₂** and in particular 1 to 5 theoretical trays are preferred.

- When the fresh alcohol ROH is added to the rectification column **RD₁/RD₂** it is added at the top of the rectification column **RD₁/RD₂** at temperatures up to boiling point, preferably at room
- 40

- temperature. The fresh alcohol may have a dedicated feed provided for it or else when a portion of the alcohol withdrawn at the top of the rectification column $\mathbf{RD}_1/\mathbf{RD}_2$ is recycled may be mixed therewith after condensation and supplied to the rectification column $\mathbf{RD}_1/\mathbf{RD}_2$ together. In this case it is particularly preferable when the fresh alcohol is added to a condensate container in which the
- 5 alcohol condensed from the vapour stream $\mathbf{S}_{RDB1}/\mathbf{S}_{RDB2}$ is collected.

5. Examples

5.1 Example 1 (noninventive)

- 5 The setup according to example 1 corresponds to the two-column interconnection according to figure 1, wherein $p_1 > p_{3A} > p_2$.

A stream S_{AE2} <3A02> of aqueous NaOH (50% by weight) of 5 t/h is supplied to the top of a reaction column RR_A <3A> at 25°C. A vaporous methanol stream S_{AE1} <3A01> of 70.2 t/h is supplied in countercurrent above the bottom of the reaction column RR_A <3A>. The reaction column RR_A <3A> is operated at a pressure p_{3A} of 1.6 bar. At the bottom of the column RR_A <3A> a virtually water-free product stream S_{AP} <3A08> of 10.8 t/h is withdrawn (30% by weight sodium methoxide in methanol). At the evaporator VS_{3A} <3A06> of the reaction column RR_A <3A> about 0.7 MW of heating power are introduced using heating steam. A vaporous methanol-water stream S_{AB} <3A03> is withdrawn at the top of the reaction column RR_A <3A>. A portion of this stream is recycled via a condenser K_{RRA} <3A05> to the reaction column RR_A <3A>, and the remaining portion (64.4 t/h) compressed in a compressor VD_{31} <10> to 7.1 bar, wherein about 4 MW of compressor power are necessary, and supplied to a first rectification column RD_1 <1>. The rectification column RD_1 <1> is operated at $p_1 = \sim 7$ bar. At the top of the rectification column RD_1 <1> a liquid fresh methanol stream of 9.5 t/h is supplied (not shown in figure 1) and vaporous methanol stream S_{RDB1} <101> is withdrawn. A portion of S_{RDB1} <101> is recycled via the condenser K_{RD1} <102> into column RD_1 <1>. The remaining portion of S_{RDB1} <101> (42.9 t/h) is supplied to the reaction column RR_A <3A>. The condenser K_{RD1} <102> of column RD_1 <1> which is simultaneously the evaporator VS_{RD2} of the second rectification column RD_2 <2> provides the heating power for the column RD_2 <2>. The embodiment according to example 1 utilized direct contacting where the condenser K_{RD1} <102> is simultaneously employed as bottoms evaporator VS_{RD2} <204>.

Discharged at the bottom of the rectification column RD_1 <1> is a liquid stream of a water-methanol mixture S_{RDS1} <103> of which a portion S_{RDS12} <104> of 30.9 t/h is passed into the rectification column RD_2 <2> and the remaining portion of the stream S_{RDS1} <103> is recycled as S_{RDS11} <105> into RD_1 <1>. At the evaporator VS_{RD1} <106> of the rectification column RD_1 <1> about 5.4 MW of heating power are introduced via heating steam.

The rectification column RD_2 <2> is operated at a pressure p_2 of 1.1 bar. Withdrawn at the top of the rectification column RD_2 <2> is a vaporous methanol stream S_{RDB2} <201>. A portion of S_{RDB2} <201> is recycled via the condenser K_{RD2} <203> into column RD_2 <2>. The remaining portion of S_{RDB2} <201> (27.3 t/h) is supplied to the reaction column RR_A <3A>. This portion of the vaporous stream S_{RDB2} <201> is compressed to 2 bar in a compressor VD_{23} <13>, wherein about 0.6 MW of compressor power are necessary. Discharged at the bottom of the rectification column RD_2 <2> is a liquid stream of water S_{RDS2} <202> (contaminated with 500 ppmw of methanol) of 3.7 t/h. For

evaporation at the rectification column $RD_2 <2>$ (since direct heat integration is effected, the function of the bottoms evaporator $VS_{RD2} <204>$ shown in figure 1 is co-assumed by the condenser $K_{RD1} <102>$) about 14.8 MW of heating power are introduced via heat integration with the column $RD_1 <1>$.

- 5 The respective, non-recycled portions of the vaporous methanol streams $S_{RDB1} <101>$ and $S_{RDB2} <201>$ withdrawn at the tops of $RD_1 <1>$ and $RD_2 <2>$ are mixed and recycled to the bottom of the reaction column $RR_A <3A>$.

- 10 Altogether in this example about 6.1 MW of heating power via heating steam and about 4.6 MW of electrical power (compressor power) are required and must be externally provided.

5.2 Example 2 (noninventive)

The setup according to example 2 corresponds to the two-column interconnection according to figure 2, wherein $p_1 > p_2 > p_{3A}$.

15

A stream $S_{AE2} <3A02>$ of aqueous NaOH (50% by weight) of 5 t/h is supplied to the top of a reaction column $RR_A <3A>$ at 25°C. A vaporous methanol stream $S_{AE1} <3A01>$ of 70.2 t/h is supplied in countercurrent above the bottom of the reaction column $RR_A <3A>$.

- 20 The reaction column $RR_A <3A>$ is operated at a pressure p_{3A} of 1.1 bar. At the bottom of the column $RR_A <3A>$ a virtually water-free product stream $S_{AP} <3A08>$ of 10.8 t/h is withdrawn (30% by weight sodium methoxide in methanol). At the evaporator $VS_{3A} <3A06>$ of the reaction column $RR_A <3A>$ about 0.8 MW of heating power are introduced using heating steam. A vaporous methanol-water stream $S_{AB} <3A03>$ is withdrawn at the top of the reaction column $RR_A <3A>$. A portion of this stream is recycled via a condenser $K_{RRA} <3A05>$ to the reaction column $RR_A <3A>$ and the remaining portion (64.4 t/h) compressed in a compressor $VD_{31} <10>$ to 9 bar, wherein about 5.8 MW of compressor power are necessary, and supplied to a first rectification column $RD_1 <1>$.
- 25 The rectification column $RD_1 <1>$ is operated at $p_1 = \sim 8.9$ bar. At the top of the rectification column $RD_1 <1>$ a liquid fresh methanol stream of 9.5 t/h is supplied (not shown in figure 2) and vaporous methanol stream $S_{RDB1} <101>$ is withdrawn. A portion of $S_{RDB1} <101>$ is recycled via the condenser $K_{RD1} <102>$ into column $RD_1 <1>$. The remaining portion of $S_{RDB1} <101>$ (42.9 t/h) is supplied to the reaction column $RR_A <3A>$. The condenser $K_{RD1} <102>$ of column $RD_1 <1>$ which is simultaneously the evaporator $VS_{RD2} <204>$ of the second rectification column $RD_2 <2>$ provides the heating power for the column $RD_2 <2>$. The embodiment according to example 2 utilized direct contacting where the condenser $K_{RD1} <102>$ is simultaneously employed as bottoms evaporator
- 30 $VS_{RD2} <204>$.
- 35

Discharged at the bottom of the rectification column $RD_1 <1>$ is a liquid stream of a water-methanol mixture $S_{RDS1} <103>$ of which a portion $S_{RDS12} <104>$ of 31.9 t/h is passed into the rectification column $RD_2 <2>$ and the remaining portion of the stream $S_{RDS1} <103>$ is recycled as $S_{RDS11} <105>$

into $RD_1 <1>$. At the evaporator $VS_{RD1} <106>$ of the rectification column $RD_1 <1>$ about 5.2 MW of heating power are introduced via heating steam.

The rectification column $RD_2 <2>$ is operated at a pressure p_2 of 1.5 bar. Withdrawn at the top of the rectification column $RD_2 <2>$ is a vaporous methanol stream $S_{RDB2} <201>$. A portion of $S_{RDB2} <201>$ is recycled via the condenser $K_{RD2} <203>$ into column $RD_2 <2>$. The remaining portion of $S_{RDB2} <201>$ (28.2 t/h) is supplied to the reaction column $RR_A <3A>$. Discharged at the bottom of the rectification column $RD_2 <2>$ is a liquid stream of water $S_{RDS2} <202>$ (contaminated with 500 ppmw of methanol) of 3.7 t/h. For evaporation at the rectification column $RD_2 <2>$ (since direct heat integration is effected, the function of the bottoms evaporator $VS_{RD2} <204>$ shown in figure 1 is co-assumed by the condenser $K_{RD1} <102>$) about 15.9 MW of heating power are introduced via heat integration with the column $RD_1 <1>$.

The respective, non-recycled portions of the vaporous methanol streams $S_{RDB1} <101>$ and $S_{RDB2} <201>$ withdrawn at the tops of $RD_1 <1>$ and $RD_2 <2>$ are mixed and recycled to the bottom of the reaction column $RR_A <3A>$.

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Altogether in this example about 6.0 MW of heating power via heating steam and about 5.8 MW of electrical power (compressor power) are required and must be externally provided.

5.3 Example 3 (inventive)

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The setup according to example 3 corresponds to the two-column interconnection according to figure 3, wherein $p_2 > p_1 > p_{3A}$. The intermediate evaporator $VZ_{RD1} <107>$ with the stream $S_{RDX1} <112>$ withdrawn at the withdrawal point <111> shown in figure 3 is likewise omitted in the setup according to example 3. The condenser $K_{RD2} <203>$ is also simultaneously the bottoms evaporator $VS_{RD1} <106>$. The fresh methanol stream $S_{XE1} <205>$ is in figure 3 supplied to the rectification column $RD_2 <2>$ but in the setup according to example 3 supplied to $RD_1 <1>$.

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A stream $S_{AE2} <3A02>$ of aqueous NaOH (50% by weight) of 5 t/h is supplied to the top of a reaction column $RR_A <3A>$ at 25°C. A vaporous methanol stream $S_{AE1} <3A01>$ of 70.2 t/h is supplied in countercurrent above the bottom of the reaction column $RR_A <3A>$. The reaction column $RR_A <3A>$ is operated at a pressure p_{3A} of 1.1 bar. At the bottom of the column $RR_A <3A>$ a virtually water-free product stream $S_{AP*} <3A08>$ of 10.8 t/h is withdrawn (30% by weight sodium methoxide in methanol). At the evaporator $VS_{3A} <3A06>$ of the reaction column $RR_A <3A>$ about 1.4 MW of heating power are introduced using heating steam. A vaporous methanol-water stream $S_{AB} <3A03>$ is withdrawn at the top of the reaction column $RR_A <3A>$. A portion of this stream is recycled via a condenser $K_{RRA} <3A05>$ to the reaction column $RR_A <3A>$ and the remaining portion (64.4 t/h) compressed in a compressor $VD_{31} <10>$ to 1.7 bar, wherein about 1.1 MW of compressor power are necessary, and supplied to a first rectification column $RD_1 <1>$. The rectification column $RD_1 <1>$ is operated at $p_1 = \sim 1.5$ bar. At the top of the rectification column $RD_1 <1>$ a liquid fresh

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methanol stream S_{XE1} <205> of 9.5 t/h is supplied (shown at the top of the rectification column RD_2 <2> in figure 3) and vaporous methanol stream S_{RDB1} <101> is withdrawn. A portion of S_{RDB1} <101> is recycled via the condenser K_{RD1} <102> into column RD_1 <1>. The remaining portion of S_{RDB1} <101> (56.8 t/h) is supplied to the reaction column RR_A <3A>. The embodiment according to example 3 utilized direct contacting where the condenser K_{RD2} <203> simultaneously serves as bottoms evaporator VS_{RD1} <106>.

Discharged at the bottom of the rectification column RD_1 <1> is a liquid stream of a water-methanol mixture S_{RDS1} <103> of which a portion S_{RDS12} <104> of 17 t/h is passed into the rectification column RD_2 <2> and the remaining portion of the stream S_{RDS1} <103> is recycled as S_{RDS11} <105> into RD_1 <1>.

The pressure of the discharged stream S_{RDS12} <104> is increased in a pump P <15> to 9 bar and the stream S_{RDS12} <104> supplied to the second rectification column RD_2 <2>. The rectification column RD_2 <2> is operated at a pressure p_2 of 8.9 bar. In the condenser K_{RD2} <203> of column RD_2 <2> which is simultaneously the evaporator of the column RD_1 <1> about 8.2 MW of heating power are provided for the column RD_1 <1>. Withdrawn at the top of the rectification column RD_2 <2> is a vaporous methanol stream S_{RDB2} <201>. A portion of S_{RDB2} <201> is recycled via the condenser K_{RD2} <203> into column RD_2 <2>. The remaining portion of S_{RDB2} <201> (13.4 t/h) is supplied to the reaction column RR_A <3A>. Discharged at the bottom of the rectification column RD_2 <2> is a liquid stream of water (contaminated with 500 ppmw of methanol) of 3.7 t/h. At the evaporator VS_{RD2} <204> of the rectification column RD_2 <2> about 12.9 MW of heating power are introduced using heating steam.

The respective, non-recycled portions of the vaporous methanol streams S_{RDB1} <101> and S_{RDB2} <201> withdrawn at the tops of RD_1 <1> and RD_2 <2> are mixed, decompressed and recycled to the bottom of the reaction column RR_A <3A>.

Altogether in this example about 14.3 MW of heating power via heating steam and about 1.1 MW of electrical power (compressor power) are required and must be externally provided.

Compared to the embodiment according to the noninventive example 1 this variant thus saves about 75% of the required compressor power to be externally provided (electrical energy).

5.4 Example 4 (inventive)

The setup according to example 4 corresponds to the two-column interconnection according to figure 4, wherein $p_2 > p_{3A} > p_1$. The intermediate evaporator VZ_{RD1} <107> with the stream S_{RDX1} <112> withdrawn at the withdrawal point <111> shown in figure 4 is likewise omitted in the setup according to example 4. The condenser K_{RD2} <203> is also simultaneously the bottoms evaporator

$\mathbf{VS}_{RD1}$ <106>. The fresh methanol stream $\mathbf{S}_{XE1}$ <205> is in figure 4 supplied to the rectification column $\mathbf{RD}_2$ <2> but in the setup according to example 4 supplied to $\mathbf{RD}_1$ <1>.

A stream $\mathbf{S}_{AE2}$ <3A02> of aqueous NaOH (50% by weight) of 5 t/h is supplied to the top of a
 5 reaction column $\mathbf{RR}_A$ <3A> at 25°C. A vaporous methanol stream $\mathbf{S}_{AE1}$ <3A01> of 70.2 t/h is supplied in countercurrent above the bottom of the reaction column $\mathbf{RR}_A$ <3A>. The reaction column $\mathbf{RR}_A$ <3A> is operated at a pressure p_{3A} of 1.6 bar. At the bottom of the column $\mathbf{RR}_A$ <3A> a virtually water-free product stream $\mathbf{S}_{AP}$ <3A08> of 10.8 t/h is withdrawn (30% by weight sodium methoxide in methanol). At the evaporator $\mathbf{VS}_{3A}$ <3A06> of the reaction column $\mathbf{RR}_A$ <3A> about
 10 0.8 MW of heating power are introduced using heating steam. A vaporous methanol-water stream $\mathbf{S}_{AB}$ <3A03> is withdrawn at the top of the reaction column $\mathbf{RR}_A$ <3A>. A portion of this stream is recycled via a condenser $\mathbf{K}_{RRA}$ <3A05> to the reaction column $\mathbf{RR}_A$ <3A> and the remaining portion (64.4 t/h) is supplied to a first rectification column $\mathbf{RD}_1$ <1>. The rectification column $\mathbf{RD}_1$ <1> is operated at $p_1 = \sim 1.1$ bar. At the top of the rectification column $\mathbf{RD}_1$ <1> a liquid fresh methanol
 15 stream $\mathbf{S}_{XE1}$ <205> of 9.5 t/h is supplied (shown at the top of the rectification column $\mathbf{RD}_2$ <2> in figure 4) and vaporous methanol stream $\mathbf{S}_{RDB1}$ <101> is withdrawn.

A portion of $\mathbf{S}_{RDB1}$ <101> is recycled via the condenser $\mathbf{K}_{RD1}$ <102> into column $\mathbf{RD}_1$ <1>. The remaining portion of $\mathbf{S}_{RDB1}$ <101> (55 t/h) is compressed to 2 bar in a compressor $\mathbf{VD}_{13}$ <16>,
 20 wherein about 1.2 MW of compressor power are required, and supplied to the reaction column $\mathbf{RR}_A$ <3A>. Discharged at the bottom of the rectification column $\mathbf{RD}_1$ <1> is a liquid stream of a water-methanol mixture $\mathbf{S}_{RDS1}$ <103> of which a portion $\mathbf{S}_{RDS12}$ <104> of 18.9 t/h is passed into the rectification column $\mathbf{RD}_2$ <2> and the remaining portion of the stream $\mathbf{S}_{RDS1}$ <103> is recycled as $\mathbf{S}_{RDS11}$ <105> into $\mathbf{RD}_1$ <1>.

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The pressure of the discharged stream $\mathbf{S}_{RDS12}$ <104> is increased in a pump $\mathbf{P}$ <15> to 3.4 bar and the stream supplied to the second rectification column $\mathbf{RD}_2$ <2>. The rectification column $\mathbf{RD}_2$ <2> is operated at a pressure p_2 of 3.2 bar. In the condenser $\mathbf{K}_{RD2}$ <203> of column $\mathbf{RD}_2$ <2> which is simultaneously the evaporator of the column $\mathbf{RD}_1$ <1> about 6.3 MW of heating power are provided
 30 for the column $\mathbf{RD}_1$ <1>. Withdrawn at the top of the rectification column $\mathbf{RD}_2$ <2> is a vaporous methanol stream $\mathbf{S}_{RDB2}$ <201>. A portion of $\mathbf{S}_{RDB2}$ <201> is recycled via the condenser $\mathbf{K}_{RD2}$ <203> into column $\mathbf{RD}_2$ <2>. The remaining portion of $\mathbf{S}_{RDB2}$ <201> (15.2 t/h) is supplied to the reaction column $\mathbf{RR}_A$ <3A>. Discharged at the bottom of the rectification column $\mathbf{RD}_2$ <2> is a liquid stream of water (contaminated with 500 ppmw of methanol) of 3.7 t/h. At the evaporator $\mathbf{VS}_{RD2}$ <204> of
 35 the rectification column $\mathbf{RD}_2$ <2> about 11.4 MW of heating power are introduced using heating steam.

The respective, non-recycled portions of the vaporous methanol streams $\mathbf{S}_{RDB1}$ <101> and $\mathbf{S}_{RDB2}$ <201> withdrawn at the tops of $\mathbf{RD}_1$ <1> and $\mathbf{RD}_2$ <2> are mixed and recycled to the bottom of the reaction column $\mathbf{RR}_A$ <3A>.

Altogether in this example about 12.2 MW of heating power via heating steam and about 1.2 MW of electrical power (compressor power) are required and must be externally provided.

Compared to the embodiment according to the noninventive example 2 this variant thus saves about 79% of the required compressor power to be externally provided (electrical energy).

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5.5 Result

10 Comparison of the proportion of heating steam and electrical current required to cover the energy demand in the inventive and noninventive examples reveals that the inventive process surprisingly makes it possible to cover a large proportion of the energy demand through heating steam and to minimize the proportion of the power to be provided through electrical energy.

Claims

1. Process for producing at least one alkali metal alkoxide of formula M_AOR , wherein R is a C_1 to C_6 hydrocarbon radical, and wherein M_A is selected from sodium, potassium, wherein:
 - 5 (a1) a reactant stream S_{AE1} comprising ROH is reacted with a reactant stream S_{AE2} comprising M_AOH in countercurrent at a pressure p_{3A} and a temperature T_{3A} in a reactive rectification column RR_A to afford a crude product RP_A comprising M_AOR , water, ROH, M_AOH ,
 - 10 wherein a bottoms product stream S_{AP} comprising ROH and M_AOR is withdrawn at the lower end of RR_A and a vapour stream S_{AB} comprising water and ROH is withdrawn at the upper end of RR_A ,
 - (a2) and optionally, simultaneously with and spatially separate from step (a1), a reactant stream S_{BE1} comprising ROH is reacted with a reactant stream S_{BE2} comprising M_BOH in countercurrent at
 15 a pressure p_{3B} and a temperature T_{3B} in a reactive rectification column RR_B to afford a crude product RP_B comprising M_BOR , water, ROH, M_BOH , wherein M_B is selected from sodium, potassium,
 - wherein a bottoms product stream S_{BP} comprising ROH and M_BOR is withdrawn at the lower end of
 20 RR_B and a vapour stream S_{BB} comprising water and ROH is withdrawn at the upper end of RR_B ,
 - (b) the vapour stream S_{AB} and, if step (a2) is performed, the vapour stream S_{BB} , in admixture with S_{AB} or separately from S_{AB} , is passed into a first rectification column RD_1 ,
 25 to obtain a mixture G_{RD1} comprising water and ROH in the first rectification column RD_1 ,
 - (c) the mixture G_{RD1} is in the first rectification column RD_1 at a pressure p_1 and a temperature T_1 separated into an ROH-comprising vapour stream S_{RDB1} at the upper end of RD_1 and a bottoms stream S_{RDS1} comprising water and ROH at the lower end of RD_1 ,
 - 30 (d) the bottoms stream S_{RDS1} is completely or partially passed into a second rectification column RD_2 ,
 to obtain a mixture G_{RD2} comprising water and ROH in the second rectification column RD_2 ,
 - (e) the mixture G_{RD2} is at a pressure p_2 and a temperature T_2 separated into an ROH-comprising
 35 vapour stream S_{RDB2} at the top of RD_2 and a bottoms stream S_{RDS2} comprising water at the lower end of RD_2 ,

characterized

in that $p_2 > p_1$, $p_2 > p_{3A}$, and in the cases where step (a2) is performed, $p_2 > p_{3B}$,

5 and in that (f) energy from S_{RDB2} is transferred to the mixture G_{RD1} in the first rectification column RD_1 .

2. Process according to Claim 1, wherein in step (f) energy is directly transferred from S_{RDB2} to G_{RD1} .

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3. Process according to Claim 2, wherein at least one of the steps (α -i), (α -ii), (α -iii) is performed:

(α -i) a first portion S_{RDS11} of the bottoms stream S_{RDS1} discharged from RD_1 is passed into the second rectification column RD_2 and energy is transferred from S_{RDB2} to a second portion S_{RDS12} of the bottoms stream S_{RDS1} discharged from RD_1 and S_{RDS12} is then recycled into RD_1 ;

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(α -ii) at least one stream S_{RDX1} distinct from S_{RDB1} and S_{RDS1} comprising ROH and water is discharged from RD_1 , energy is then transferred from S_{RDB2} to S_{RDX1} and S_{RDX1} is recycled into RD_1 ;

20 (α -iii) S_{RDB2} is passed through RD_1 , thus transferring energy from S_{RDB2} to G_{RD1} .

4. Process according to Claim 1, wherein in step (f) energy is indirectly transferred from S_{RDB2} to G_{RD1} .

25 5. Process according to Claim 4, wherein at least one of the steps (β -i), (β -ii), (β -iii) is performed:

(β -i) a first portion S_{RDS11} of the bottoms stream S_{RDS1} discharged from RD_1 is passed into the second rectification column RD_2 and a second portion S_{RDS12} of the bottoms stream S_{RDS1} discharged from RD_1 is recycled into RD_1 , wherein energy is transferred from S_{RDB2} to at least one heat transfer medium W_{i1} distinct from S_{RDS12} and is then transferred from the at least one heat transfer medium W_{i1} to S_{RDS12} , and S_{RDS12} is then recycled into RD_1 ;

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(β -ii) at least one stream S_{RDX1} distinct from S_{RDB1} and S_{RDS1} comprising ROH and water is discharged from RD_1 and energy is transferred from S_{RDB2} to at least one heat transfer medium W_{ii1} distinct from S_{RDX1} and then transferred from the at least one heat transfer medium W_{ii1} to S_{RDX1} , and S_{RDX1} is then recycled into RD_1 ;

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(β -iii) energy is transferred from S_{RDB2} to at least one heat transfer medium W_{iii1} distinct from G_{RD1} and the at least one heat transfer medium W_{iii1} is then passed through RD_1 , thus transferring energy from the at least one heat transfer medium W_{iii1} to G_{RD1} .

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6. Process according to Claim 5, wherein each of W_{i1} , W_{ii1} , W_{iii1} is water.
7. Process according to any of Claims 3, 5 and 6, wherein S_{RDX1} is withdrawn below the vapour
5 stream S_{RDB1} on RD_1 .
8. Process according to any of Claims 1 to 7, wherein S_{RDB2} is at least partially employed as
reactant stream S_{AE1} in the reactive rectification column RR_A and, if step (a2) is performed,
alternatively or in addition employed as reactant stream S_{BE1} in the reactive rectification column
10 RR_B .
9. Process according to any of Claims 1 to 8, wherein S_{RDB1} is at least partially employed as
reactant stream S_{AE1} in the reactive rectification column RR_A and, if step (a2) is performed,
alternatively or in addition employed as reactant stream S_{BE1} in the reactive rectification column
15 RR_B .
10. Process according to any of Claims 1 to 9, wherein a stream S_{XE1} distinct from S_{AE1} and S_{BE1}
comprising ROH is added to at least one of the columns selected from rectification column RD_1 ,
rectification column RD_2 , reactive rectification column RR_A and, if step (a2) is performed, is
20 alternatively or in addition added to the reactive rectification column RR_B .
11. Process according to any of Claims 1 to 10, wherein R is methyl or ethyl.
12. Process according to any of Claims 1 to 11, wherein step (a2) is performed.
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13. Process according to any of Claims 1 to 12, wherein $p_{3A} > p_1$ and in addition, in the cases
where step (a2) is performed, $p_{3B} > p_1$.
14. Process according to any of Claims 1 to 13, wherein the bottoms stream S_{RDS2} comprises water
30 and ROH.
15. Process according to any of Claims 1 to 14 which is carried out continuously.