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(72) Inventeur/Inventor:
MEESSEN, JOZEF HUBERT, NL
(73) Propriétaire/Owner:
STAMICARBON B.V., NL
(74) Agent: SMART & BIGGAR

(54) Titre : PROCÉDE DE PRÉPARATION D'UN COURANT AQUEUX COMPRENANT DE L'UREE
(54) Title: PROCESS FOR THE PREPARATION OF A UREA-COMPRISING AQUEOUS STREAM

(57) **Abrégé/Abstract:**

The invention relates to a process for the preparation of a urea-comprising aqueous stream, that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases, wherein the urea-comprising aqueous stream is separated directly from or after a recovery section in a urea production process and is thereafter diluted with water until the urea-comprising stream comprises 30-35 wt % urea.



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(71) Applicant (for all designated States except US): **DSM IP ASSETS B.V.** [NL/NL]; Het Overloon 1, NL-6411 TE Heerlen (NL).

(72) Inventor; and

(75) Inventor/Applicant (for US only): **MEESEN, Jozef, Hubert** [NL/NL]; Marchierstraat 10, NL-6321 AL Wijlre (NL).

(74) Agent: **SCHELTUS, Irma**; DSM Intellectual Property, P.O. Box 9, NL-6160 MA Geleen (NL).

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(54) Title: PROCESS FOR THE PREPARATION OF A UREA-COMPRISING AQUEOUS STREAM

(57) Abstract: The invention relates to a process for the preparation of a urea-comprising aqueous stream, that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases, wherein the urea-comprising aqueous stream is separated directly from or after a recovery section in a urea production process and is thereafter diluted with water until the urea-comprising stream comprises 30-35 wt % urea.

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PROCESS FOR THE PREPARATION OF A UREA-COMPRISING
AQUEOUS STREAM

The invention relates to a process for the preparation of a urea-
5 comprising aqueous stream which is suitable for use in a unit for the reduction of NO_x
in combustion engine exhaust gases.

NO_x emissions from combustion engines (for instance diesel engines)
with an exhaust gas catalyst can be reduced by injection of reducing components into
the exhaust pipe of these diesel engines before the exhaust gas is contacted with a
10 SCR or an EGR catalyst. The use of a urea-comprising aqueous stream as a reducing
agent for this purpose offers special advantages, in particular in diesel engines used to
power cars and trucks. The quality requirements to be met by such a urea-comprising
aqueous stream are known, for example from DIN Vornorm V70070.

Such a urea-comprising aqueous stream is currently prepared by
15 dissolving commercially available solid urea in clean water while adding heat. The solid
urea is available in the form of, for example, urea granulate or urea prills.

A problem encountered is that commercially available solid urea is
contaminated with formaldehyde or other additives, because the formaldehyde or the
other additives are added to improve the properties of the solid urea and facilitate
20 granulation or prilling of the urea. According to the Vornorm cited above only very low
amounts of contaminants are allowed in a urea-comprising aqueous stream that will be
used in a unit for the reduction of NO_x. A urea-comprising aqueous stream obtained by
dissolving solid urea does not meet the quality requirements according to the Vornorm.

At this moment the solid urea or the urea-comprising aqueous stream
25 obtained from it is purified by removing the formaldehyde, the reaction products of
formaldehyde and urea and other additives using known separation techniques.
However, this process is expensive.

It is the aim of the invention to eliminate these drawbacks.

This is achieved by separating the urea-comprising aqueous stream directly from or after a recovery section in a urea production process and thereafter dilute the urea-comprising aqueous stream with water until the urea-comprising stream comprises 30-35 wt% urea.

5 In this way a urea-comprising aqueous stream that does not contain formaldehyde, reaction products of formaldehyde and urea or other additives is directly obtained.

A further advantage is that no solid urea needs to be dissolved in water in order to obtain a urea-comprising aqueous stream.

10 Commercial production of urea usually takes place from NH_3 and CO_2 , according to known processes, such as for example a conventional urea process, a CO_2 stripping process, a thermal stripping processes and the ACES process. All these processes consist of a urea synthesis section and one or more recovery sections, in which a urea-comprising aqueous stream with a urea content of more than
15 50 wt% is produced. Subsequently, the urea-comprising aqueous stream is concentrated further, to a water content between 0.1 and 5 wt%, in an evaporation or crystallization section. This water content depends on the requirements set in the shaping section present in the urea production process. Examples of shaping techniques are prilling and granulation. For the benefit of the shaping technique, and
20 also for improvement of the product quality of the solid urea (transport properties), formaldehyde, reaction products of formaldehyde and urea or other additives are added to the urea-comprising aqueous stream during or after the evaporation/crystallization process.

The urea-comprising aqueous stream can be separated from an
25 existing urea process. The urea-comprising aqueous stream can be the total urea-comprising stream resulting from the urea process or it can be a part of it. It is also possible to design a new urea process that is particularly suitable for the production of a urea-comprising aqueous stream that will be used in a unit for the reduction of NO_x in combustion engine exhaust gases. An advantage of the design of a
30 new urea process is that this new process does not need to comprise an evaporation or crystallization section and a shaping section.

The urea-comprising aqueous stream is separated directly from or after a recovery section in the urea production process. The urea-comprising aqueous stream may come from one recovery section but also, if the urea production process
35 comprises more recovery sections, from several recovery sections.

The urea-comprising aqueous stream can be one stream that is separated from one location in the urea production process or it can be composed of several streams that are separated at various locations from the urea production process. After separation the urea-comprising aqueous stream is diluted with water.

5 In the recovery section(s) of a urea production process the ammonium carbamate, free carbon dioxide and free ammonia content of the urea synthesis solution is reduced. This is done by heating the solution and optionally by reducing the pressure. This temperature increase and optionally also pressure decrease causes the ammonium carbamate that is present to dissociate into free
10 ammonia and free carbon dioxide. A substantial part of this free ammonia and free carbon dioxide passes into the gas phase, which is separated from the rest of the urea-comprising aqueous stream in liquid/gas separators. In the various urea production processes different pressure and temperature levels are applied in the recovery section(s). In some processes a stripping medium is added to one or more dissociation
15 steps in order to promote the dissociation process. Examples of a suitable stripping medium are ammonia gas, carbon dioxide gas, air and steam. The various processes also differ in the way in which the expelled gaseous ammonia and carbon dioxide are processed. While the urea solution flows through the various dissociation steps, the alkalinity and the ammonium carbamate content are reduced.

20 It is preferred to separate the urea-comprising aqueous stream after a dissociation unit in a urea production process in which at least one of the recovery sections comprises a dissociation unit and in which dissociation is promoted by the addition of a stripping medium.

25 Preferably, the urea-comprising aqueous stream is separated after the recovery section(s).

In a urea process, wherein a urea storage tank is present after the recovery section(s) and, when a shaping section is present, before the shaping section, the urea-comprising aqueous stream is, more preferably, separated from the urea storage tank.

30 The urea-comprising aqueous stream that is separated directly from or after a recovery section in a urea production process preferably comprises 60-90 wt% urea.

For use of the urea-comprising aqueous stream in a unit for the reduction of NO_x in combustion engine exhaust gases, the urea-comprising stream that
35 is directly separated from a urea production process needs to be diluted until the urea-

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comprising stream comprises 30-35 wt% urea.

By separating the urea-comprising aqueous stream from a urea production process in the way according to the invention it is achieved that a urea-comprising aqueous stream is obtained that usually has a sufficiently low alkalinity and carbonate (as CO_2 content) to make these solutions suitable to be used in a unit for the reduction of NO_x in combustion engine exhaust gases.

In the rare cases that this is not the case, the alkalinity (as NH_3 content) and the carbonate (as CO_2 content) of the urea-comprising aqueous stream can simply be reduced further by subjecting the urea-comprising aqueous stream to dissociation before or after dilution with water. Preferably, the dissociation is performed before dilution with water, since then the amount of solution to be subjected to dissociation is limited. In some cases it might however be more favourable to perform the dissociation after the dilution with water, since this would allow for lower temperatures in the dissociation process without formation of solids during dissociation. A reduced temperature is of advantage to minimize the amount of urea lost through hydrolysis, as well as to minimize the amount of biuret formed during dissociation.

The dissociation can be performed, optionally, by the addition of heat and/or with the addition of a suitable stripping medium and/or by reduction of the pressure.

Preferably, the dissociation is performed by steam stripping at a pressure of 0.001-0.2 MPa. The use of steam as a stripping medium prevents the formation of solid material during stripping, which would result in transport problems of the solution. By the use of a reduced pressure during dissociation the temperature during dissociation can be kept low, such that hydrolysis of urea is prevented.

The invention will be elucidated hereinafter on the basis of Figures 1 and 2, without being restricted to these embodiments.

In figure 1 a urea production process is shown comprising a urea synthesis section (UP), a urea recovery section (UR), a urea storage tank (S), an evaporation section (EV) and a shaping section (US). The end product of this urea production process is solid urea (s).

From the urea storage tank (S) a urea-comprising aqueous stream is separated comprising 75 wt% urea. This urea-comprising aqueous stream (U1), having an alkalinity of 0.7% (as NH_3) and a carbonate content (as CO_2) of 0.7%, enters a dissociation unit (D), wherein the urea-comprising aqueous stream is stripped with steam under a pressure of 0.04 MPa.

After dissociation the urea-comprising aqueous stream is diluted with water to a urea content of 31.5 wt% urea.

A urea-comprising aqueous stream (U2) with a urea content of 31,5 wt%, an alkalinity (as NH_3) of 300 ppm and a carbonate content (as CO_2) of 300 ppm is stored as a urea-comprising aqueous stream (AU), that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases.

In figure 2 an other urea production process is shown that is especially developed for the production of a urea-comprising aqueous stream, that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases.

10 This urea production process comprises a urea synthesis section (UP), a urea recovery section (UR), comprising a dissociation unit wherein dissociation is promoted by the addition of CO_2 as a stripping agent. A urea-comprising aqueous stream (U1) with a urea content of 79wt% is separated after this dissociation unit in the urea recovery section and is diluted with water to a urea content of 31.5 wt%. This urea-comprising

15 aqueous stream (U1), having an alkalinity (as NH_3) of 0.1% and a carbonate content (as CO_2) of 0.05% is stored as a urea-comprising aqueous stream (AU), that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases.

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Claims:

1. Process for the preparation of a urea-comprising aqueous stream, that is suitable for use in a unit for the reduction of NO_x in combustion engine exhaust gases, wherein the urea-comprising aqueous stream is separated directly from or
5 after at least one recovery section in a urea production process and is thereafter diluted with water until the urea-comprising stream comprises 30-35 wt% urea.
2. Process according to claim 1, wherein at least one of the recovery sections comprises a dissociation unit, in which dissociation is prompted by the addition of a stripping medium, and wherein the urea-comprising aqueous stream is
10 separated after the dissociation unit.
3. Process according to claim 1 or 2, wherein the urea-comprising aqueous stream is separated after the recovery section(s).
4. Process according to any one of claims 1-3, wherein a urea storage tank is present after the recovery section(s) and, when a shaping section is present,
15 before the shaping section, and wherein the urea-comprising aqueous stream is separated from the urea storage tank.
5. Process according to any one of claims 1-4, wherein the urea-comprising aqueous stream separated directly from or after the at least one recovery section comprises 60-90 wt% urea.
- 20 6. Process according to any one of claims 1-5, wherein the urea-comprising aqueous stream is subjected to dissociation before or after dilution with water.
7. Process according to claim 2 or 6, wherein the dissociation is performed by steam stripping at a pressure of 0.001-0.2 MPa.

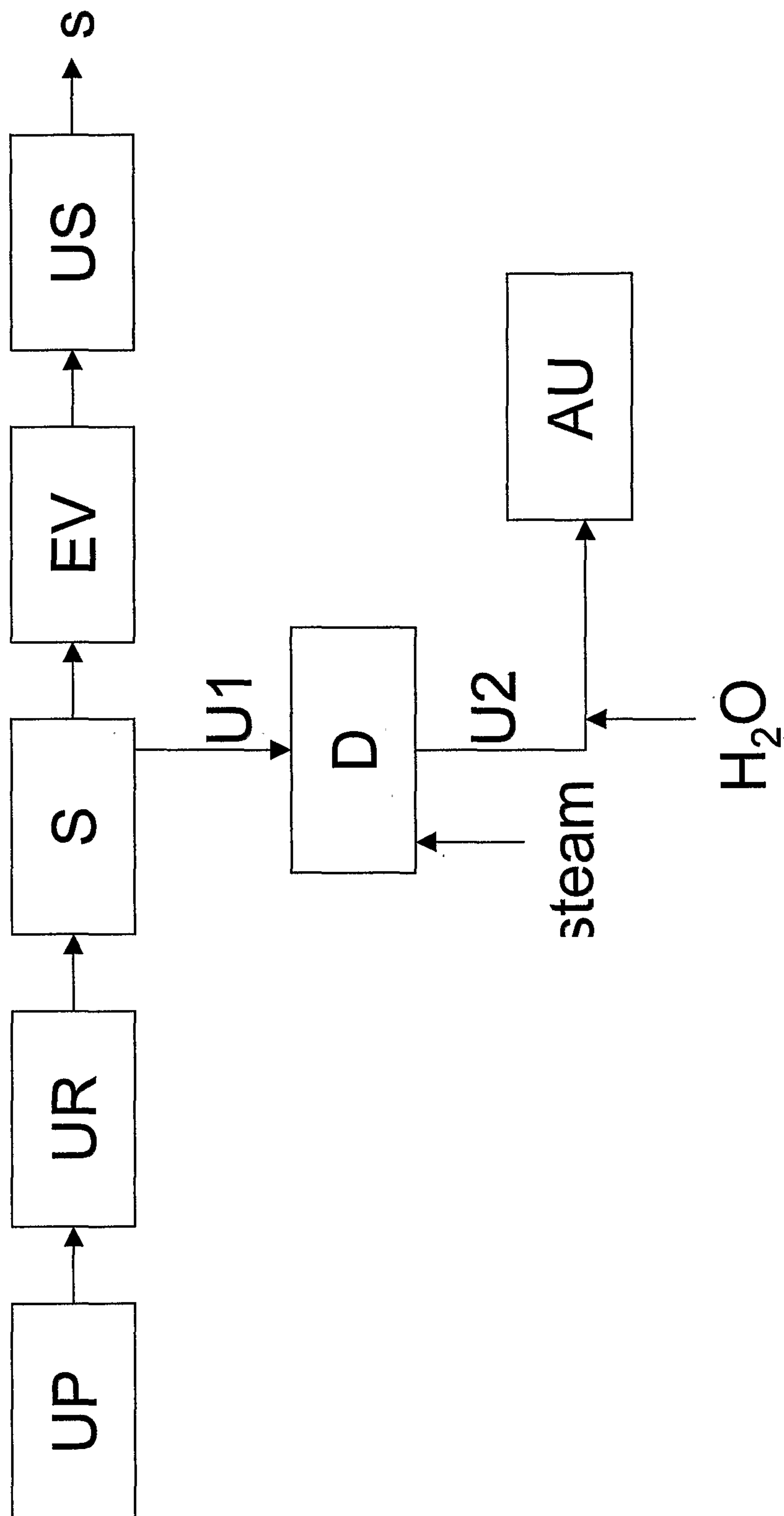


Fig. 1

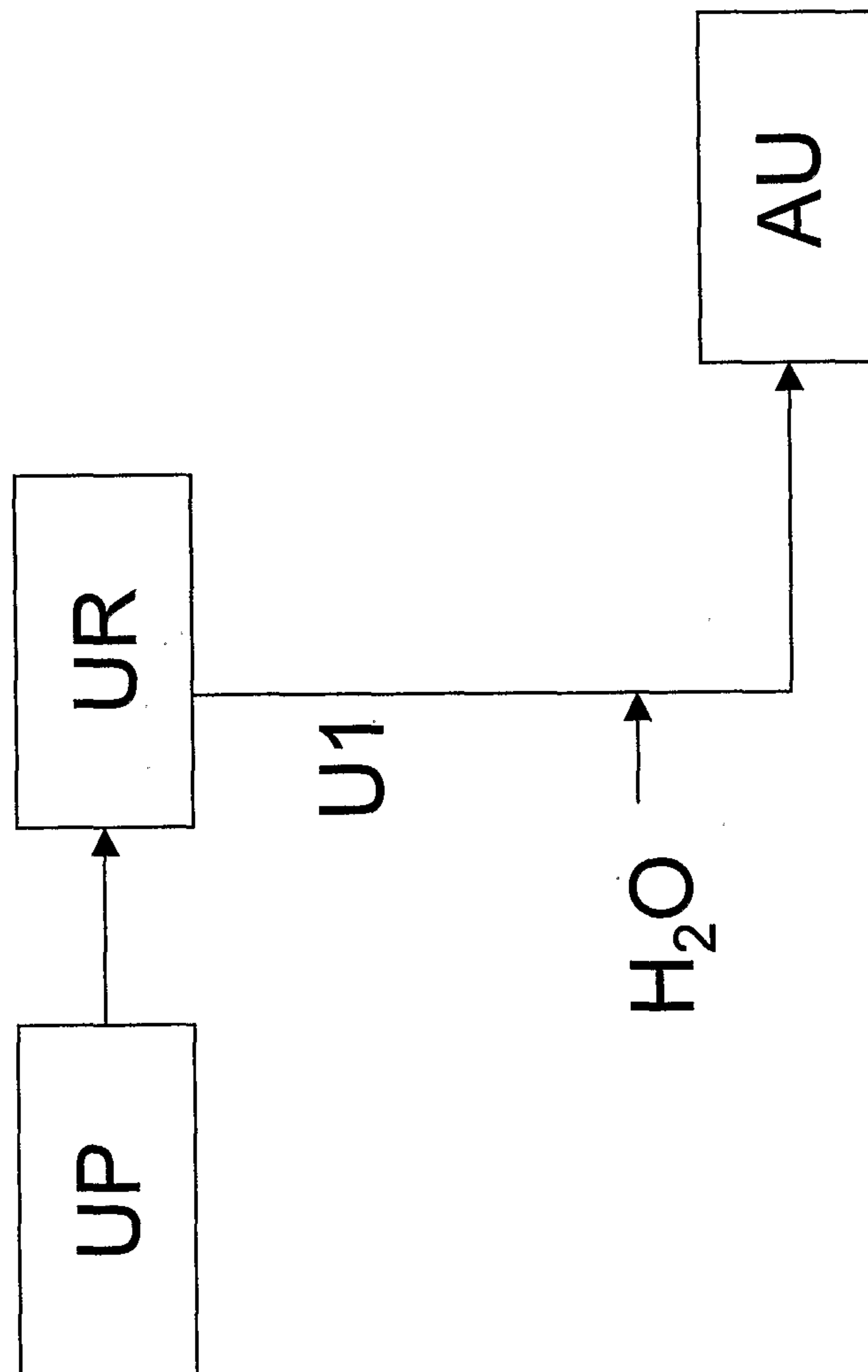


Fig. 2