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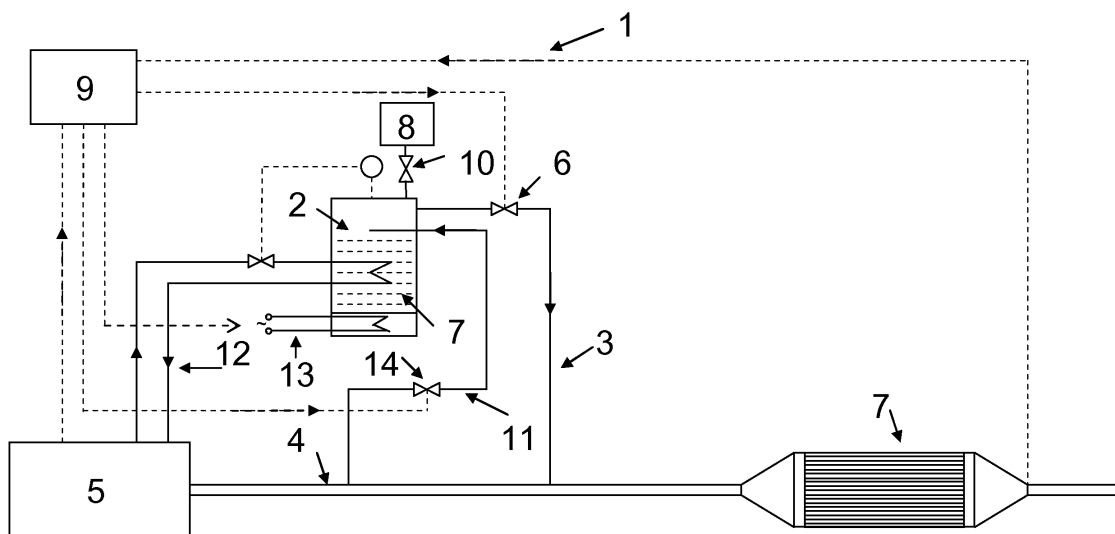
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Remarks:
Amended claims in accordance with Rule 137(2)
EPC.

(57) The invention relates to a method and a device for treating an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH_3) which is produced by decomposition of a precursor comprising ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$), and wherein

the method comprises the steps of placing the precursor in a pressure tight converter (2), flowing gas via said converter (2) at a temperature at which partial equilibrium decomposition of said precursor to ammonia (NH_3) occurs, and injecting the produced ammonia into the exhaust gas.

Fig. 1



Description

[0001] The invention relates to a method and a device for treating an exhaust gas of a combustion device.

[0002] More specifically, the invention relates to a device and method for producing ammonia and controlled delivering thereof to an exhaust gas flow, particularly but not exclusively to an exhaust gas flow of an internal combustion engine (ICE) for reducing NO_x contained in this exhaust gas flow, in particular on a SCR-catalyst. Although the invention is particularly applied to automotive applications, in particular diesel engines, the invention can also be applied to stationary diesel engines as well as in industry, power plants and fuel cell applications which are using ammonia.

[0003] Ammonia is known as a selective and effective reduction agent for the selective catalytic reduction (SCR) of nitrogen oxides in oxygen-containing exhaust gases from burners in power plants, from internal combustion engines etc. For the reasons of toxicity and safety problems arising from storing gaseous ammonia, in particular procedures have been developed wherein ammonia is generated on-board as a reduction agent by hydrolysis of urea.

[0004] EP 0 487 886 B1 discloses a method of quantitative hydrolysis of urea, wherein an aqueous urea solution is sprayed onto an evaporator and forwarded via a hydrolysis catalyst at the surface of the evaporator, and a hydrolysis catalyst being coated with active components catalyzing the quantitative hydrolysis of urea into ammonia and CO₂ and inhibiting the formation of solid urea reaction by-products. However, such a urea-using procedure causes large equipment costs due to the hydrolysis catalyst, the energy supply for urea decomposition and hydrolysis, as well as due to the transportation of urea and its introduction, which must be uniform over the cross section of the flow. A further disadvantage is the formation of undesirable by-products such as, for example isocyanic acid, and cyanuric acid, and in addition the formation of polymeric products blocking urea converter and catalyst.

[0005] DE 34 22 175 A1 and DE 42 00 514 A1 disclose processes related to the "just-in-time" production of NH₃ for the reduction of NO_x. The concept comprises using specific substances which hydro-thermally decompose to NH₃, the handling and toxicity of which being quite safe, in order to release the required quantity of NH₃ in accordance with the particular requirements and to inject the same into the exhaust gas flow. The generation of NH₃ takes place by heating ammonia generating compounds as e. g. ammonium carbamate. The adaptation to the particular operating state of the motor, as described in DE 34 22 175 A1, takes place by controlling the calorific power which e.g. acts on the carbamate. The NH₃ generation and thus the NH₃ quantity added to the exhaust gas flow can be regulated.

[0006] US 6 399 034 B1 discloses a process and device for NO_x-reduction of exhaust gases of an internal

combustion engine using a SCR-catalyst, wherein substances are used which are decomposing to NH₃ substantially free of residues under hydrothermal conditions, such as e.g. ammonium carbamate (NH₂CO₂NH₄), as well as substances which are able to reversibly NH₃-absorb/desorb and thus splitting off NH₃, such as e.g. iron (II) ammine sulphate.

[0007] EP 1 263 520 B1 discloses the use of solid reducing agent as ammonium carbamate and for example ammonium salts as ammonium carbonate, ammonium formate for thermolysis into ammonia in SCR-applications. The reducing agent is brought into physical contact with a hot liquid heat transfer agent.

[0008] After gas formation, the gas is held in a gas collecting chamber and is fed in a controlled manner into the exhaust gas flow. Furthermore, the pumping power and/or the heating power and/or the amount of reduction agent fed into the exhaust gas are controlled by virtue of a controller.

[0009] EP 1 323 903 B1 also discloses the use of ammonium carbonate in order to produce ammonia in the same field of use, but as a solid mixture with heat carrier substance. Using such carrier substances can create problems like depositions or blockage. Vapours of these carrier substances could deposit on the SCR catalyst, which could have influence on the SCR catalyst activity and seriously decrease activity of SCR catalyst.

[0010] It is an object of the present invention to provide a method and device for treating an exhaust gas of a combustion device which enable a safe, reliable and clean treating and which are also suitable to be used at low temperature conditions.

[0011] A method of treating an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH₃) which is produced by decomposition of a precursor comprising ammonium carbonate (NH₄)₂CO₃, comprises the steps of:

- placing the precursor in a pressure tight converter;
- flowing gas via said converter at a temperature at which partial equilibrium decomposition of said precursor to ammonia (NH₃) occurs; and
- injecting produced ammonia into the exhaust gas.

[0012] According to the present invention, a flow through pressure-tight converter (which could be heated if needed) is provided as a NH₃ source, wherein a precursor comprising ammonium carbonate being located in said converter produces ammonia via maintaining equilibrium gas composition in the converter with gas flow.

[0013] Advantages of the use ammonium carbonate ((NH₄)₂CO₃), according to the present invention, as precursor for ammonia (NH₃) can be summarized as follows:

- a) Ammonium carbonate decomposes to ammonia

also at room temperature, which is much lower compared e.g. to urea; for comparison, urea decomposes at much higher temperatures and cannot completely decompose even at 400°C.

b) Ammonium carbonate is decomposing to simple compounds including ammonia, carbon dioxide and water without formation of additional by-products such as e.g. isocyanic acid or cyanuric acid, and polymers blocking the decomposition reactor; accordingly a poisoning of the SCR catalyst with polymerization side products is prevented.

c) Ammonium carbonate decomposes directly to gaseous ammonia, and does not require hydrolysis catalyst, or hydrolysis properties of SCR catalyst; for comparison, the necessity of hydrolysis in urea use needs more energy and can decrease NO_x efficiency of the catalyst due to competing reactions involved on the same surface of the catalyst and/or formation of side-products or deposits.

d) Ammonium carbonate (and its mixture with ammonium carbamate) is more stable at temperatures below 50°C compared to pure ammonium carbamate, which makes it safer to use as solid source of ammonia.

e) A special hydrolysis catalyst (as need e.g. to accelerate urea decomposition) is not required.

f) Ammonium carbonate in closed container is releasing ammonia even at room temperature and is in equilibrium with gaseous ammonia, which could be shifted by flow of gas towards production of ammonia. This requires much less energy to decompose and offers lower temperature range compared e.g. to ammonium carbamate or to solid urea, so it can act as reducing agent at room temperature, which is a significantly lower temperature than e.g. 160°C (which is minimal temperature for the urea use).

g) Freezing problems in winter operation conditions (as e.g. arising during use of aqueous urea solution) are avoided, making it beneficial for removing nitrogen oxides under cold start conditions.

h) The substances and its mixture with other substances presented in the invention have chemical properties presenting not any harm to the humans and environment with respect to toxicity. Their handling and toxicity are safe at ambient conditions.

i) The use ammonium carbonate is cheaper compared e.g. to pure ammonium carbamate.

[0014] The flow through pressure tight converter does

not need to be heated, but could be slightly heated, if needed, with heating coils being placed around the converter and/or inside the converter. Principally, the relatively low temperature range of the ammonium carbonate equilibrium decomposition makes it possible to use just the part of the exhausted gas flow, or if needed use also heat sources attributed to the engine. Although the electrical heating is not necessary, it is advantageous in situations where a faster ammonia release at start or cold start conditions is required.

[0015] According to a preferred embodiment, the ammonia being produced from said precursor is subsequently placed and stored in a reservoir such as a storage vessel. The ammonium storage vessel enables to have gaseous ammonia available even during conditions of intermittent engine operation and/or during start phases, regardless of a possible heating phase of the converter (when there is insufficient energy for heating the precursor). The intermediate amount of ammonia prepared and stored in a compensation vessel or more vessels may be small such, that it cannot create any harmful emissions in case of a hypothetical destruction of the device due to an accident.

[0016] According to a preferred embodiment, the production of ammonia and/or the injection of ammonia into the exhaust flow is controlled depending on the engine operating characteristics and/or depending on a NO_x-concentration in the exhaust gas, such that a specific quantity of ammonia required for an appropriate reduction of NO_x-gases in the exhaust gases may be injected and such that the production/injection may be timely terminated if necessary.

[0017] The storage vessel preferably acts as a dosing device. The dosing may be performed based upon control signals from a control unit processing in dependence on the engine characteristics, the NO_x-gases concentration in the exhaust gas and the NO_x-output exhaust concentration, its temperature and the temperature of converter.

[0018] According to a preferred embodiment, the inventive method, in order to supply a sufficient amount of ammonia as reducing agent for oxides of nitrogen, comprises maintaining a certain sufficient flow of the part of exhaust gas, pressure in the reactor chamber for ammonium carbonate decomposition and/or additional buffer chamber by controlling gas flow and additional amount of ammonium carbonate on demand of required amount of ammonia, and controlling temperature of converter. Evolved gaseous NH₃ is produced at the rate essentially balanced to the amount of nitrogen oxides in the gas stream and in accordance with system strategy requirements.

[0019] The heat generated during the operation of the internal combustion engine can be additionally used for the process of decomposition to ammonia by making use of the heat of cooling water or oil. Additionally the electrical heating can be used especially for a cold start.

[0020] The intermediate storage of ammonia can be

realized in the decomposition reactor vessel itself or in a separate vessel with specific volume, pressure and temperature accommodating the sufficient amount of ammonia able to support efficient NO_x removal.

[0021] For a better control of dosing quantity of ammonia, the supply of the part of exhaust gas to converter, and the dosing device can be equipped with valves allowing reduction of the pressure and supplying a required dosing quantity of ammonia. The dosing is preferably controlled according to a given strategy by a programmed unit. Preferably such a strategy does not allow an injection of high quantities of ammonia which would lead to the slip of unconsumed ammonia out of the catalyst.

[0022] The converter allows easy addition of new substance or mixture of substances, when a part or almost all previous charge is consumed. The converter is preferably equipped and fitted with quick fasteners allowing its quick recharge with fresh substance or its removal or replacement. The amount of the heat supply (only when needed) is regulated according to amount of ammonia needed and in line with a strategy for removal of NO_x-gases from engine exhaust gas.

[0023] The method according to the present invention with the features of the main claim has the advantage over the related art that a gaseous reduction agent is provided which can be accurately metered, and does not cause the freezing problems in winter operation. It does not cool exhaust gas and therefore does not require minimal temperature limit to avoid formation of deposits and to prevent the poor low-temperature performance of SCR-catalyst.

[0024] The invention also relates to a device for treating an exhaust flow of a combustion device, wherein the exhaust gas is treated with ammonia (NH₃) which is produced by decomposition of a precursor comprising ammonium carbonate ((NH₄)₂CO₃), wherein said device is adapted to perform a method as described above. As to preferred embodiments or advantages of the device, reference is made to the embodiments and advantages of the method described above.

[0025] Further aspects of the present invention can be gathered from the following description and the appended claims.

[0026] The invention is described in more detail with reference to the following detailed description and based upon preferred embodiments shown in the drawings, in which:

Figure 1 shows a schematic diagram of a device for treating an exhaust gas of a internal combustion diesel engine according to a preferred embodiment of the present invention;

Figure 2 shows a temperature dependence illustrating of the thermal decomposition of ammonium carbonate in comparison with other ammonia precursors in the temperature range from 30 to 170°C;

Figure 3 shows a further temperature dependence illustrating the thermal decomposition of ammonium carbonate in comparison with ammonium carbamate in the temperature range from 30 to 170°C;

Figure 4 shows a formation of ammonia at constant flow of gas via converter and for different starting amounts of precursor ammonium carbonate at room temperature; and

Figure 5 shows an ammonia production at constant flow of gas via converter and for different starting amounts of precursor ammonium carbonate at room temperature.

[0027] Fig. 1 shows a schematic diagram of a preferred embodiment of a device according to the present invention, wherein the principle of producing ammonia and injecting said ammonia into the exhaust stream of a diesel engine is illustrated.

[0028] The device 1 comprises from a converter 2, a line 11 for a part of exhaust gas, equipped with a valve 14 for controlling gas flow, a supply line 3 for introducing NH₃ into and exhaust gas flow 4 of a diesel engine 5, a valve 6 provided as a dosing device as well as a SCR catalyst 7 being arranged in said exhaust gas flow 4 downstream of the diesel engine 5. The converter 2 is filled with sufficient amount of ammonium carbonate (NH₄)₂CO₃ or with a mixture of ammonium carbonate with ammonium hydrogen carbonate and/or ammonium carbamate from a reservoir 8. The reservoir 8 may be selectively isolated from or connected to the converter 2 via a valve 10.

[0029] The converter 2 usually operates at ambient temperature, but is equipped with heating coils 12, being placed around the converter 2 and/or inside the converter 2, as well as an additional electrical heater 13, to proper adjust temperature of the converter 2 if needed, in order to liberate a sufficient amount of ammonia from the ammonium precursor or the mixture of precursors until the gas equilibrium and pressure inside the converter 2 and/or the additional storage reservoir (or reservoirs) reaches a level which is needed to supply required amount of ammonia to the exhaust gas flow 4 in order to achieve an efficient catalytic removal of nitrogen oxides.

[0030] The process is controlled by a processing unit 9. The processing unit 9 particularly controls the introduction of part of exhaust gas to the converter and the gaseous ammonia into the exhaust gas flow 4 using the valve 6. The process control can be based on the performance of the engine 5 and/or the measured level of nitrogen oxides (NO_x) exiting the SCR-catalyst 7. In case that an additional amount of precursor is needed, such an additional amount of precursor can be introduced from the reservoir 8 via (opened) valve 10 to the converter 2.

[0031] The control of the part of the exhaust gas flow and pressure control is allowing also safety control of the

system, providing the control to stop an overproduction of ammonia. Under idle engine conditions, i. e. when an ammonia supply is not required, the part of the exhaust gas flow will be shut down and valve 6 can be in its closed position.

[0032] A further possibility to obtain ammonia from the ammonium carbonate precursor is to spray or dose small particles or dust of ammonium carbonate from the reservoir 8 into the preheated converter 2 (or more than one converter 2 used alone or subsequently), such that all precursor material will decompose and will release ammonia, which can subsequently be dosed into the exhaust stream by the dosing valve 6 as already described above.

[0033] Although the electrical heating is not necessary, it is advantageous in situations where a more quick ammonia release during cold start conditions is required. Principally, the relatively low temperature range of the ammonium carbonate equilibrium decomposition makes it also possible to use just the part of the exhausted gas flow, or if needed use heat sources attributed to the engine such as cooling water and/or oil.

[0034] As can be gathered from Fig. 2 (showing the normalized weight depending on the temperature) and Fig. 3 (showing the temperature dependence of the temperature derivative of weight, i. e. a graph "dw/dT vs. T"), an advantage of using ammonium carbonate ((NH₄)₂CO₃) and/or its mixtures compared to ammonium carbamate (NH₂CO₂NH₄) is that the temperature range of decomposition of ammonium carbonate ((NH₄)₂CO₃) to ammonia starts approximately at 50°C, which is higher by (10-15)°C compared to ammonium carbamate decomposition, and finishes approximately at a temperature higher by 30°C (almost at 170°C) if compared to the ammonium carbamate (see Fig. 3). On the other hand, ammonium carbamate starts to decompose at 30°C with peak at 80°C (see dashed line in Fig. 3) This makes it potentially risky to use a source of ammonia because of stability at rather lower temperatures. Furthermore, the decomposition of ammonium carbamate produces small amounts of by-products, as can be seen in Fig. 2 from the tailing of ammonium carbamate decomposition curve (though much less pronounced compared e. g. with urea). By using ammonium carbonate ((NH₄)₂CO₃), the weight loss is complete, which evidences a complete decomposition without the formation of additional side products.

[0035] Furthermore, as can be seen from Fig. 4 and 5, sufficient amount of ammonia can be produced from ammonium carbonate at room temperature by flow of gas via the converter filled with adequate amount of ammonium carbonate. In this case just change of equilibrium gas composition is enough to produce ammonia, which is proportional to certain extend with amount of ammonium carbonate (as shown in Fig. 5). This could be applied also to other ammonium salts, such as ammonium hydrogen carbonate, ammonium carbamate, ammonium formate, and their mixtures.

Claims

1. A method of treating an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH₃) which is produced by decomposition of a precursor comprising ammonium carbonate ((NH₄)₂CO₃),
characterized in that
said method comprises the steps of:
 - a) placing the precursor in a pressure tight converter (2);
 - b) flowing gas via said converter (2) at a temperature at which partial equilibrium decomposition of said precursor to ammonia (NH₃) occurs; and
 - c) injecting ammonia produced in step b) into the exhaust gas.
2. The method according to claim 1,
characterized in that
said gas being flowed in step b) comprises a part of the exhaust gas.
3. The method according to claim 1 or 2,
characterized in that
said gas being flowed in step b) comprises a gas being different from the exhaust gas, in particular air.
4. The method according to anyone of the claims 1 to 3,
characterized in that
said flowing of gas is adjusted such as to produce a desired amount of ammonia (NH₃).
5. The method according to anyone of the claims 1 to 4,
characterized in that
the temperature used in step b) is ambient temperature.
6. The method according to anyone of the claims 1 to 4,
characterized in that
the temperature used in step b) is a predetermined temperature achieved by cooling or heating.
7. The method according to anyone of the preceding claims,
characterized in that
the temperature used in step b) is maintained in said converter (2) until a predetermined pressure has been build up in said converter (2).
8. The method according to anyone of the preceding claims,
characterized in that
said precursor is introduced into said converter (2) in step a) from a separate reservoir (8), which may be selectively connected or isolated from said converter (2).

9. The method according to anyone of the preceding claims,
characterized in that
said ammonia (NH_3) is repeatedly injected into the exhaust gas flow (4). 5
10. The method according to anyone of the preceding claims,
characterized in that
the placing of precursor in step a) and/or the production of ammonia (NH_3) in step b) and/or the injection of ammonia (NH_3) in step c) is controlled depending on the engine operating characteristics and/or depending on a NO_x -concentration in the exhaust gas (4). 10 15
11. The method according to anyone of the claims 1 to 10,
characterized in that
in step a) a precursor is used that consists only of ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$). 20
12. The method according to anyone of the claims 1 to 10,
characterized in that
in step a) a precursor is used that consists of a mixture of ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$) with ammonium hydrogen carbonate and/or ammonium carbonate. 25 30
13. A device for treating nitrogen oxides in an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH_3) which is produced by equilibrium decomposition of a precursor comprising ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$),
characterized in that
said device is adapted to perform a method according to anyone of the preceding claims. 35 40

Amended claims in accordance with Rule 137(2) EPC.

1. A method of treating an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH_3) which is produced by decomposition of a precursor comprising ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$),
characterized in that
said method comprises the steps of: 45 50
- a) placing the precursor in a pressure tight converter (2);
- b) flowing gas via said converter (2), at least temporarily at ambient temperature, such that partial equilibrium decomposition of said precursor to ammonia (NH_3) occurs; and 55

c) injecting ammonia produced in step b) into the exhaust gas.

2. The method according to claim 1,
characterized in that
said gas being flowed in step b) comprises a part of the exhaust gas.
3. The method according to claim 1 or 2,
characterized in that
said gas being flowed in step b) comprises a gas being different from the exhaust gas, in particular air.
4. The method according to anyone of the claims 1 to 3,
characterized in that
said flowing of gas is adjusted such as to produce a desired amount of ammonia (NH_3).
5. The method according to anyone of the claims 1 to 4,
characterized in that
in step b) the temperature is temporarily adjusted by cooling or heating.
6. The method according to anyone of the preceding claims,
characterized in that
in step b) the temperature is maintained in said converter (2) until a predetermined pressure has been build up in said converter (2).
7. The method according to anyone of the preceding claims,
characterized in that
said precursor is introduced into said converter (2) in step a) from a separate reservoir (8), which may be selectively connected or isolated from said converter (2).
8. The method according to anyone of the preceding claims,
characterized in that
said ammonia (NH_3) is repeatedly injected into the exhaust gas flow (4).
9. The method according to anyone of the preceding claims,
characterized in that
the placing of precursor in step a) and/or the production of ammonia (NH_3) in step b) and/or the injection of ammonia (NH_3) in step c) is controlled depending on the engine operating characteristics and/or depending on a NO_x -concentration in the exhaust gas (4).
10. The method according to anyone of the claims 1 to 9,

characterized in that

in step a) a precursor is used that consists only of ammonium carbonate $((\text{NH}_4)_2\text{CO}_3)$.

11. The method according to anyone of the claims 1 to 9, 5

characterized in that

in step a) a precursor is used that consists of a mixture of ammonium carbonate $((\text{NH}_4)_2\text{CO}_3)$ with ammonium hydrogen carbonate and/or ammonium carbonate. 10

12. A device for treating nitrogen oxides in an exhaust gas of a combustion device, wherein the exhaust gas is treated with ammonia (NH_3) which is produced by equilibrium decomposition of a precursor comprising ammonium carbonate $((\text{NH}_4)_2\text{CO}_3)$, 15

characterized in that

said device is adapted to perform a method according to anyone of the preceding claims. 20

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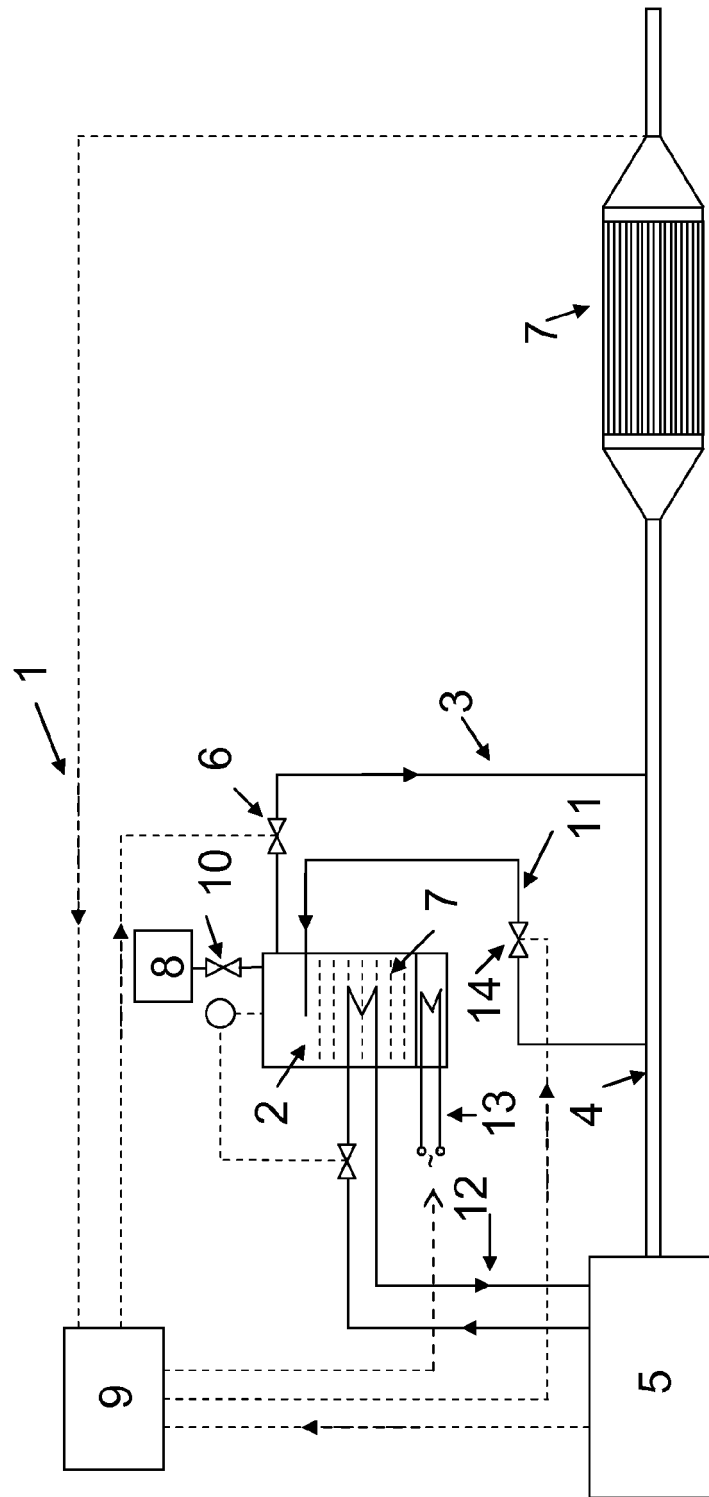


Fig. 1

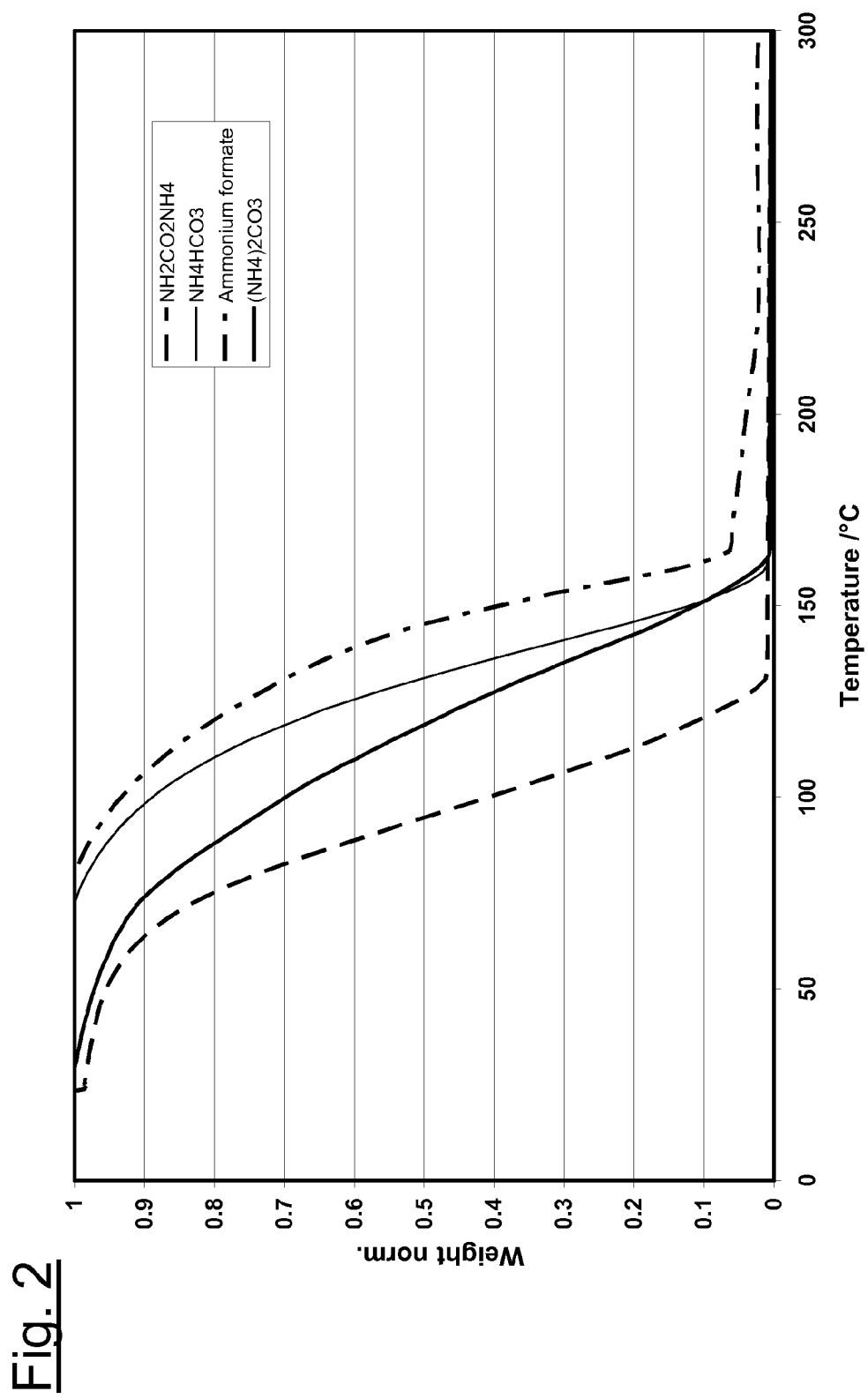


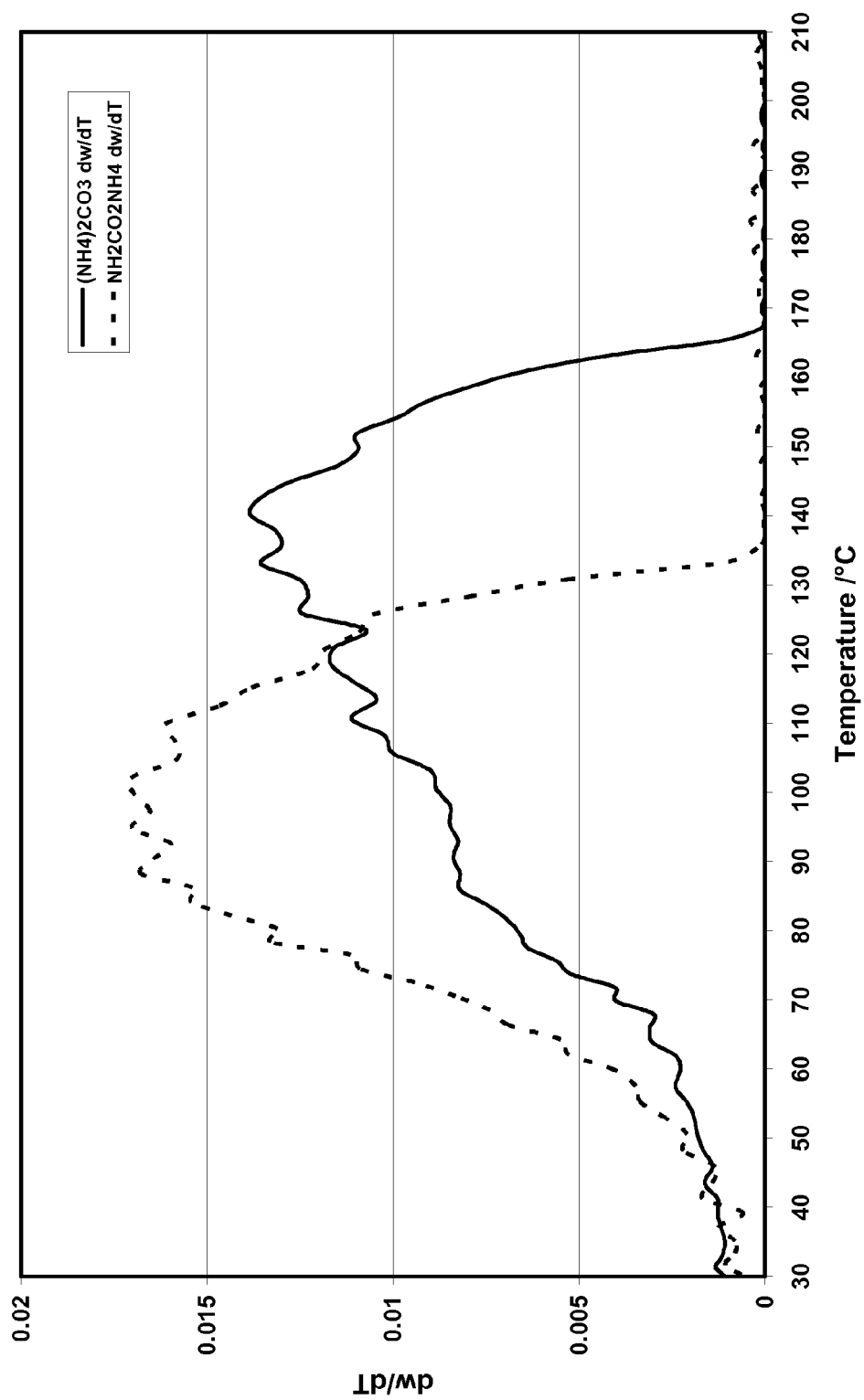
Fig. 3

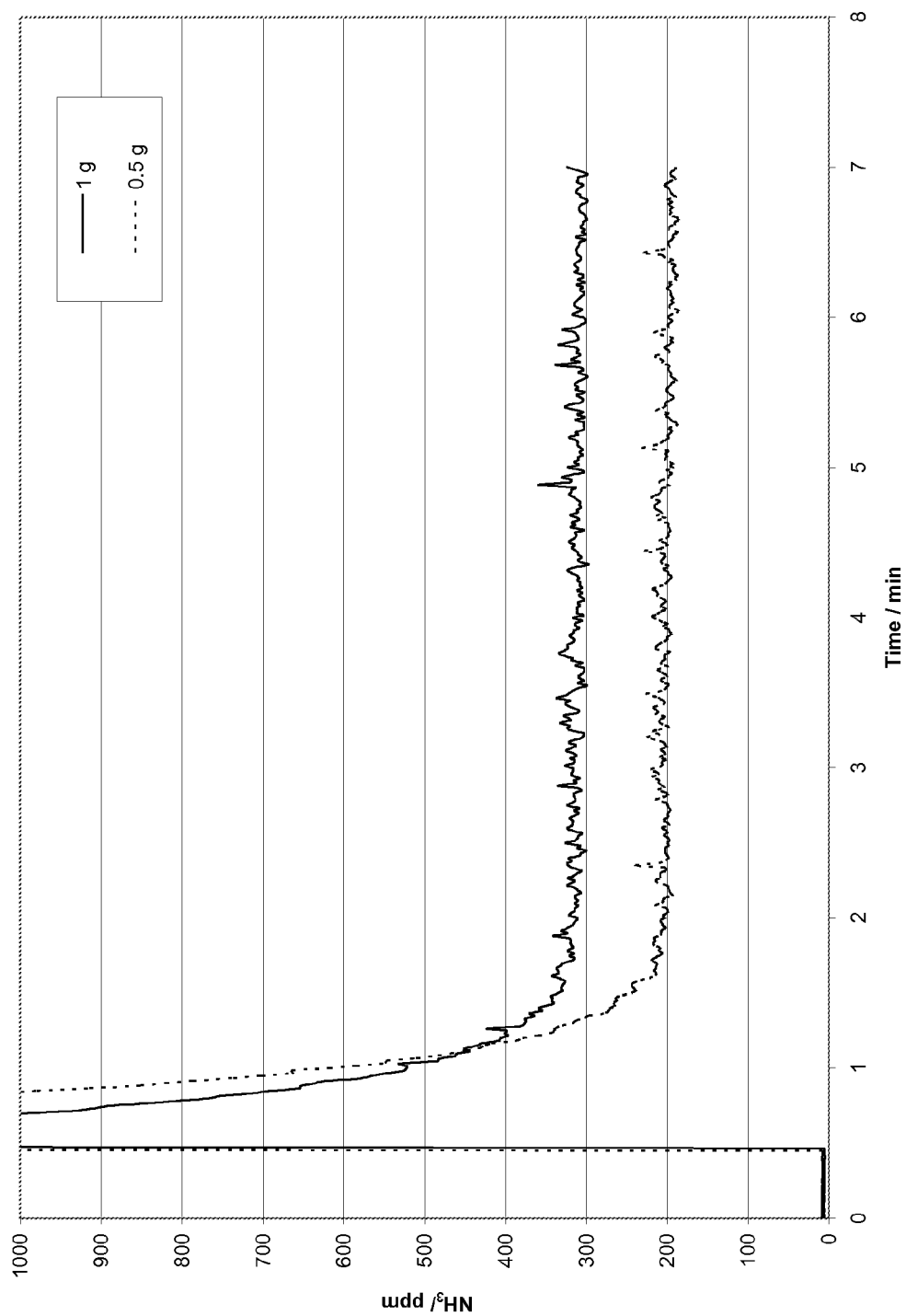
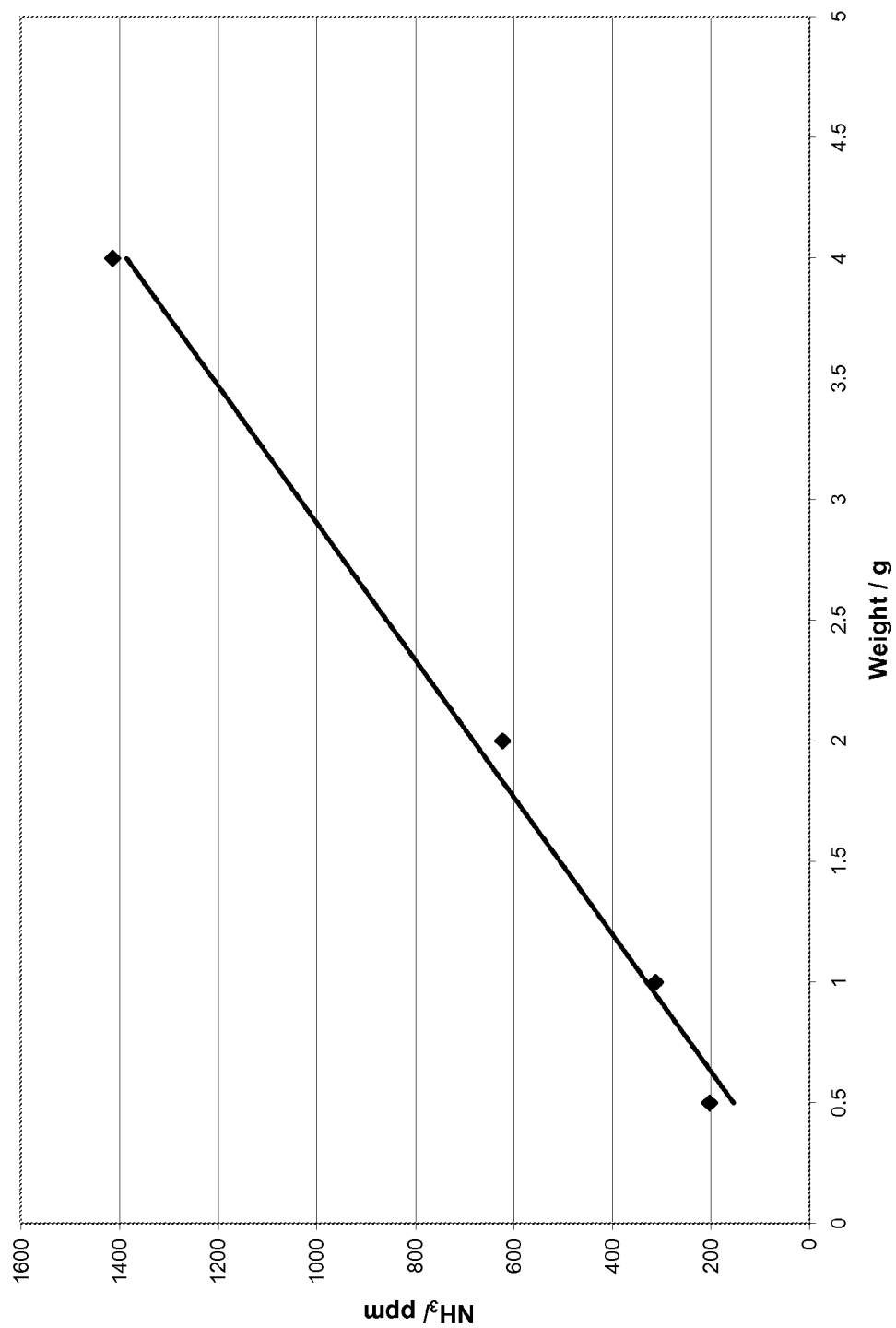
Fig. 4

Fig. 5



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 08 10 0134

DOCUMENTS CONSIDERED TO BE RELEVANT			
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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 26 May 2008	Examiner Tatus, Walter
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 08 10 0134

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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26-05-2008

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