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- *as to the identity of the inventor (Rule 4.17(i))*
- *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
- *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*
- *of inventorship (Rule 4.17(iv))*

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Interstage removal of ammonia

The capacity of ammonia plants is increasing, thereby taking advantage on the economy of scale. This constantly increases the demand on new built plants as well as the demand for optimization and revamps of existing plants is in constant focus.

- 5 Therefore there is a need for new process and plants which increase capacity and efficiency of new and existing plants.

10 In a first aspect of the present process and plant is provided a way to increase the conversion of the reaction mixture and in this way increase the efficiency and/or lower the synthesis loop recycle gas flow rate and thus improve the energy efficiency and reduce the size of the recycle gas compressor.

15 In a second aspect of the present process and plant is provided a way to increase the capacity of an existing ammonia plant.

These and other advantages are achieved by a process comprising the steps of

- Providing a synthesis gas stream (make up gas) to an ammonia loop
- In the ammonia loop passing the syngas through a first ammonia converter
- Obtaining a first effluent from the first ammonia converter
- 20 - cooling of effluent from the first ammonia converter thereby obtaining a first cooled effluent
- Separating the first cooled effluent into at least a first ammonia stream and a first unreacted stream
- Heating the first unreacted stream
- 25 - Passing the first heated unreacted stream through a second ammonia converter
- Obtaining a second effluent from the second ammonia converter
- cooling the second effluent thereby obtaining a second cooled effluent
- Separating the second cooled effluent into at least a second ammonia stream and a second unreacted stream
- 30 - Recycling the second unreacted stream to the syngas stream.

Makeup gas (synthesis gas) is added to the loop in order to maintain a desired amount of reactants H₂ and N₂. I.e. a mix of recycled unreacted gas and make up gas is provided to the first converter.

The loop may comprise more than two reactors/separators in which case it is the unreacted stream from the last separator in the loop which is returned to the first converter together with the admixed make up gas.

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The present invention facilitates ammonia synthesis in at least two ammonia converters in series with interstage removal of ammonia from the reaction mixture. As the ammonia synthesis reaction is exothermic and restricted by equilibrium, interstage removal of ammonia increases the ammonia conversion per pass and thereby increase ammonia product yield.

10

The present process is highly relevant for revamping existing ammonia plants in order to increase the capacity and/or efficiency of such plants. However, the setup may also be applied in new ammonia plants.

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The standard ammonia loop consists of one and/or two converters in series and/or in parallel without interstage removal of NH_3 , with 1 to 3 beds in each converter with interbed heat exchangers.

20

The present invention provides a process for ammonia production by reacting gaseous mixture in serial ammonia converters with interstage removal of ammonia from the reaction mixture. As the ammonia synthesis reaction is exothermic and restricted by equilibrium, removal of ammonia between converters will favor the equilibrium reaction to proceed in the direction that increases ammonia conversion and thereby increase ammonia product yield.

25

Thus by the present process it is possible to improve the conversion per pass in the ammonia synthesis loop by installing additional ammonia converter(s) downstream the first ammonia separator in the loop.

30

The first and second ammonia converter may be of the same or different type. E.g. the first converter and/or second converter may be single bed and/or multibed; radial flow ;

adiabatic and/or a quench converters; vertical or horizontal converters; with or without interbed heat exchangers, with or without feed/effluent exchanger.

5 The first and second converters can be operated at the same or different inlet temperatures and/or inlet pressure in order to optimize the efficiency of the conversion and/or catalyst in the individual converters. The pressure in the convertor may e.g. be 100 – 350 bar, such as 140- 220

10 The catalyst in one or more of the ammonia converters may for example be a known ammonia catalyst such as promoted iron catalyst. Example Topsoe KM-1, KM-111.

15 A purge can be taken preferably from the final recycle stream in order to avoid accumulation of inerts in the loop. The N₂:H₂ ratio in the mixed stream of recycle unreacted gas + make up gas entering the first converter is preferably approximately 3. Such as 3 or 3 +/- 1% or 3 +/- 2%. The ratio may change slightly down the loop as N₂ and H₂ may be absorbed at different ratios in the ammonia stream leaving the at least first and second separator.

20 The second (or third, fourth etc in case of three, four etc reactors) unreacted stream is admixed with make up synthesis gas before being passed to the first ammonia converter.

25 At least one compressor can be arranged in one or more of the unreacted streams such as in the first and/or second unreacted stream (recycle). In case the ammonia loop comprises a third, fourth etc. set of ammonia converter/separator a compressor may alternatively/also be arranged in one or more of the unreacted stream from these.

The NH₃ plant is preferably run under standard NH₃ conversion conditions.

30 Thus by the present process and plant it is possible to increase the conversion per pass and in this way increasing the efficiency and/or the synthesis loop recycle gas flow rate and thus improve the energy efficiency and reduce the size of the recycle gas compressor.

In some setups the present process and plant may be particularly useful in relation to revamp projects. The present process is most suitable for revamping existing plants comprising at least one ammonia synthesis converter in order to increase the capacity and/or efficiency of such plants.

5

The present application also relates to a method for revamping a plant having one or more ammonia converters.

10

Existing NH_3 plants may be optimized by adding at least one interstage NH_3 removal step and/or adding one or more reactors thereby obtaining a NH_3 loop comprising at least two NH_3 converters with inter stage NH_3 removal in between.

15

Inter stage NH_3 removal may be added by adding a separation step or e.g. by making an existing separation step an inter stage separation step (inter stage removal) step by adding a converter.

20

If for example the existing plant has a single NH_3 converter, one or more extra NH_3 converters may be added together with means for at least one step of interstage NH_3 removal.

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A single interstage NH_3 removal step may advantageously be arranged between a first and second NH_3 converter. In a plant comprising more than two NH_3 converters (from revamp or from new) a single interstage NH_3 removal step may be preferred, preferably arranged between a first and second NH_3 converter.

30

The process of ammonia production subdivided into following main sections:

1) Synthesis gas production: The goal is preparing mixture of hydrogen and nitrogen. The raw materials are water, air, and hydrocarbons like natural gas, naphtha, coal, solid fuels, coke oven gas, bio gas etc.

This section is further subdivided into

- a) Feedstock pretreatment and gas generation
- b) Carbon monoxide conversion
- c) Gas purification
- 2) Compression: Compress the synthesis gas to a pressure needed for ammonia synthesis
- 3) Ammonia synthesis loop and ammonia condensation
- 4) Purge gas management

10 In fig. 1 a schematic of the process and ammonia loop is seen. Recycle gas 7 and make-up gas 8 (synthesis gas) is mixed to a mixed stream 9. The mixed stream 9 is heated in E1 and introduced to the 1st converter C1, where the gas is converted over promoted iron catalyst (e.g. reduced and/or unreduced Topsoe KM -1 or KM-111). The effluent 11 from converter C1 is cooled in E1/E2, and most of the produced ammonia is condensed and separated in a first gas/liquid separator (inter stage NH₃ removal), V1. The vapor phase (first unreacted stream) 15 from separator V1 is sent to recirculator compressor K1 . The discharge stream 1 from K1 is heated in E4 and introduced to the 2nd converter C2, where the gas is converted over promoted iron catalyst (e.g. reduced and/or unreduced Topsoe KM-1 or KM-111). The effluent from converter C2 is cooled in E4/E3 and condensed ammonia is separated in the second gas/liquid separator, V2. The vapor phase (second unreacted stream) 7 from separator V2 is recycled to the mix point 16. The condensed ammonia streams 6 and 14 from the first and second separator is led to storage and/or further treatment.

25 The loop with at least two reactors and inter stage removal of NH₃ may be achieved by revamping an existing loop as illustrated in fig 2 – 4.

In fig. 2 a NH₃ loop comprising a first converter C1 followed by a second converter C2 and a first separation V1 is revamped by adding an inter stage NH₃ removal step V2 in between the first and second convertor as illustrated by the dotted arrow.

In fig 3 a NH₃ loop comprising a first converter C1 followed two separation steps V1 V2 is revamped by adding second converter C2 in between the two separation steps as indicated by the dotted line whereby the first separation step becomes an inter stage NH₃ removal step.

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In fig. 4 a NH₃ loop comprising a first converter C1 and a first separation step V1 is revamped by adding both a converter C2 and separator V2 between C1 and V1 or after the second separator.

10

Example 1: configuration as per the present process and plant, i.e. with condensation and separation of ammonia between converters

Example 2: configuration as per prior art i.e. no ammonia separation between converters.

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In the following table conversion of syngas into ammonia (NH₃ conversion). It is seen that the conversion is higher in example 1.

20

In example 2, in order to obtain the same ammonia production, higher catalyst volume is needed. However ammonia conversion as high as of example 1 is not achieved.

	Example 1: Invention		Example 2: Prior art	
	Converter C1	Converter C2	Converter C1	Converter C2
Inlet pressure barg	251	261	261.6	254
Inlet composition mol %				
H ₂ :N ₂ ratio	2.97		2.97	
H ₂	61.79	55.18	62.03	49.64
N ₂	20.87	18.65	20.88	16.78
NH ₃	3.05	8.47	3.00	17.5

Inerts (CH ₄ +Ar+He)	14.11	17.7	14.09	16.08
Outlet NH ₃ mol%	17.30	14.15	17.5	21.66
NH ₃ conver- sion	14.25	5.68	14.5	4.16
Relative cata- lyst volume % (relative to catalyst vol- ume in inven- tion)	100	100	100	345
Total Ammo- nia production % relative to invention	100		100	

Claims

1. A process for producing ammonia, said process comprising the steps of
- Providing a synthesis gas stream to an ammonia loop
 - 5 In the ammonia loop passing the syngas through a first ammonia converter
 - Obtaining a first effluent from the first ammonia converter
 - cooling of effluent from the first ammonia converter thereby obtaining a first cooled effluent
 - Separating the first cooled effluent into at least a first ammonia stream and a
 - 10 first unreacted stream
 - Heating the first unreacted stream
 - Passing the first heated unreacted stream through a second ammonia converter
 - Obtaining a second effluent from the second ammonia converter
 - cooling the second effluent thereby obtaining a second cooled effluent
 - 15 - separating the second cooled effluent into at least a second ammonia stream and a second unreacted stream.
2. Process according to claim 1, wherein the second unreacted stream is mixed with synthesis gas provided to the loop (make up gas) to form a mixed stream and the
- 20 mixed stream is passed through the first convertor.
3. Process according to claim 1, comprising heating the second unreacted stream
- Passing the second heated unreacted stream through a third ammonia converter
 - 25 - Obtaining a third effluent from the third ammonia converter
 - cooling the third effluent thereby obtaining a third cooled effluent
 - separating the third cooled effluent into at least a third ammonia stream and a third unreacted stream.
- 30 4. Process according to any of the claims 1 or 3, wherein the third unreacted stream is mixed with synthesis gas provided to the loop (make up gas) to form a mixed stream and the mixed stream is passed through the first convertor.

5. Process according to any of the claims 1 -4, comprising compressing the first unreacted stream in a recycle gas compressor.

- 5 6. Method for revamping a plant with one or more ammonia converters, said method comprising the steps of
- providing means for interstage removal of NH_3 , and/or adding one or more NH_3 converters.

- 10 7.. Plant revamped according to claim 6.

8. Ammonia loop comprising
- a mixing point for mixing synthesis gas (make up gas) with unreacted recycled gas to form a mixed stream
 - 15 - a first converter for reacting the mixed stream into a first effluent comprising ammonia
 - a first separator for separating the first effluent into a first ammonia stream and a first unreacted stream
 - a second convertor for reacting the first unreacted stream into a second effluent comprising ammonia
 - 20 - a second separator for separating the second effluent into a second ammonia stream and a second unreacted stream
 - at least one recycle compressor, and
 - means for cooling the first and second effluent comprising ammonia prior to separation.

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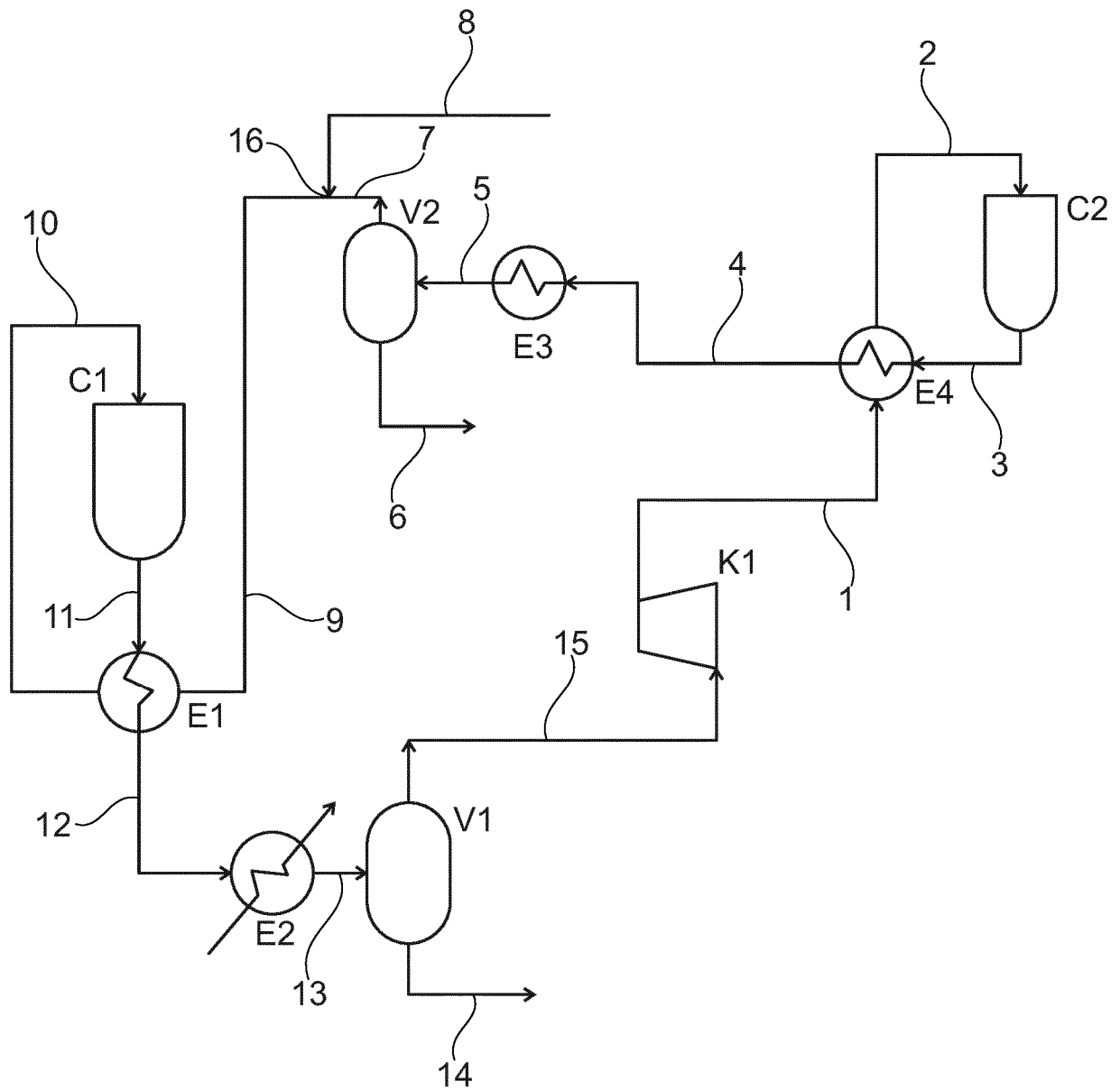
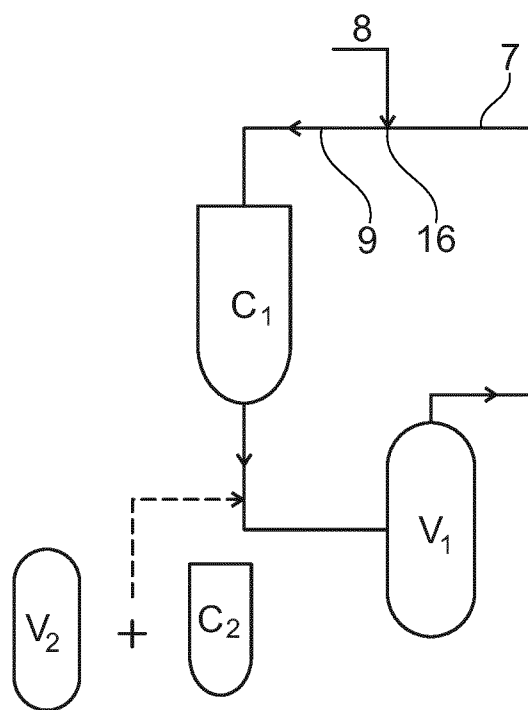
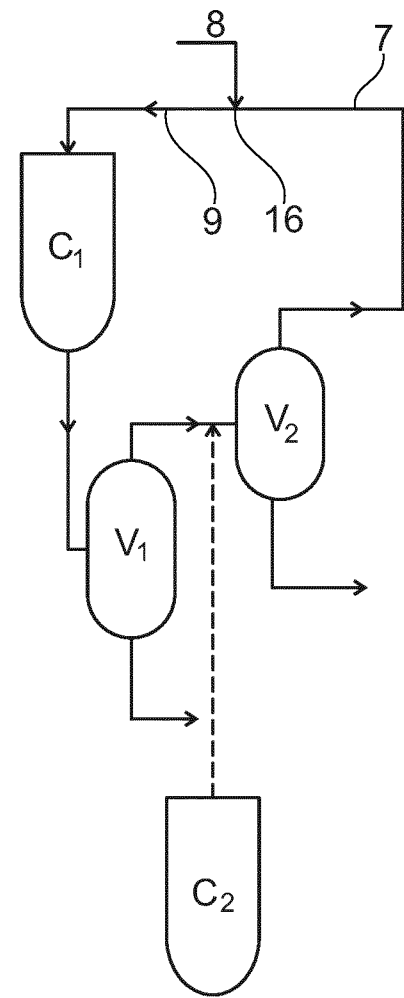
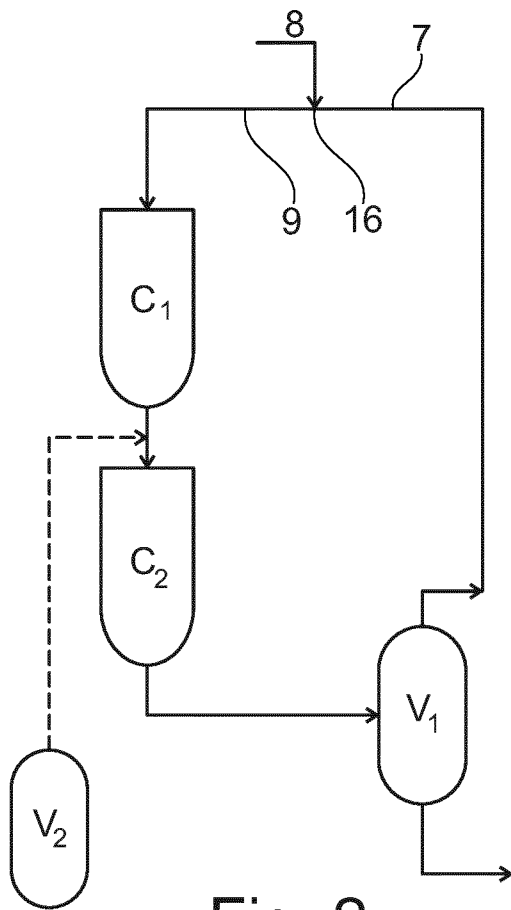


Fig. 1

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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2017/065074

A. CLASSIFICATION OF SUBJECT MATTER
INV. C01C1/04 B01J8/04
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C01C B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, CHEM ABS Data, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2004/042951 A1 (LIPPMANN DENNIS [DE] ET AL) 4 March 2004 (2004-03-04) paragraphs [0028], [0029], [0035], [0037], [0038]; figures 1,2, 4 -----	1-8
X	US 2007/293590 A1 (HIPP ANTON J [DE]) 20 December 2007 (2007-12-20) paragraphs [0001], [0015] - [0018], [0032] - [0036], [0066] - [0067]; figure 4 -----	1-8
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Further documents are listed in the continuation of Box C.



See patent family annex.

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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