

Process for the preparation of 3-aminomethyl-3,5,5-trimethylcyclohexylamine

5 **Description**

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The invention relates to an improved process for the preparation of 3-aminomethyl-3,5,5-trimethylcyclohexylamine, hereafter also called isophoronediamine or abbreviated to IPDA, from 3-cyano-3,5,5-trimethylcyclohexanone, hereafter also called isophoronenitrile or abbreviated to IPN, by aminating hydrogenation with hydrogen and ammonia in the presence of a fixed bed catalyst. The process according to the invention affords the continuous preparation of isophoronediamine in high yield and high purity.

Isophoronediamine is used as a starting material for the preparation of isophorone diisocyanate, which is an isocyanate component for polyurethane systems, as an amine component for polyamides and as a hardener for epoxy resins. Isophoronediamine is conventionally prepared from isophoronenitrile, the carbonyl group being converted to an amino group and the nitrile group to an aminomethyl group in the presence of ammonia, hydrogen and conventional hydrogenation catalysts. The starting material, isophoronenitrile, can be obtained in conventional manner by the addition of hydrogen cyanide onto isophorone - see DE-OS 39 42 371.

According to the process described in US 3,352,913 for the preparation of isophoronediamine from isophoronenitrile, the hydrogenation is carried out in the presence of ammonia and in the presence of cobalt-, nickel-, iron- or noble metal- containing catalysts known per se, at 50 to 150°C and at a pressure of at least 50 bar. By way of example, the hydrogenation takes place in the presence of methanol

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as a solvent and using suspension or fixed bed catalysts. Apart from the desired isophoronediamine, by-products, such as 3-aminomethyl-3,5,5-trimethylcyclohexanol (isophoroneamino alcohol, IPAA) in particular, are formed
5 in relatively large amounts. The disadvantages of this process are found to be the low yield and a considerable proportion of by-products.

In the quest to obtain a higher yield of IPDA and minimize
10 the unavoidable formation of IPAA, DE-OS 30 11 656 teaches a two-step process; in the first step, IPN is converted with excess ammonia, without a catalyst, to 3-cyano-3,5,5-trimethyliminocyclohexane, and the latter is hydrogenated
15 in the second step to IPDA. The disadvantage of this process is that, as well as the actual hydrogenation reactor, a special imine formation reactor is required.

EP-B 0 042 119 proposes a further improvement to the process, wherein, prior to reaction with ammonia and
20 hydrogen in the presence of hydrogenation catalysts at temperatures of 10 to 120°C and pressures of 1 to 300 bar, the isophoronenitrile is subjected to a preliminary reaction with ammonia in the presence of inorganic and organic ion exchangers in the ammonium form as imine
25 formation catalysts. Whereas the volume ratio of isophoronenitrile to ammonia in the imine formation step is said to be 1:0.5 to 20, this ratio is increased to 1:10 to 20 in the hydrogenation step. Although the process, which can be carried out in trickle bed reactors, leads to a high
30 yield of IPDA and to a high purity, the economy of the process is compromised by the use of two different catalysts and the large excess of ammonia, which necessitates a very high pressure and hence an expensive hydrogenation apparatus.

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EP-B 0 042 119 also discloses a Comparative Example in which isophoronenitrile and liquid ammonia are pumped into



the top of a hydrogenation reactor charged with commercially available cobalt catalyst. The reaction system is kept at 270 bar with H_2 ; a certain gas stream is set up and a certain amount of off-gas is withdrawn. The reaction mixture leaving the bottom of the reactor is then worked up by distillation. Despite approximately quantitative conversion of the isophoronenitrile, this embodiment produces only 48% of isophoronediamine, together with many by-products. Said document does not suggest passing a mixture containing isophorone, ammonia and organic solvent, instead of a mixture of isophoronenitrile and ammonia, over a trickle bed reactor at an essentially low pressure and without the need for a preliminary imine formation reaction.

EP-A 0 449 089 discloses another process for the preparation of isophoronediamine: In two spatially separated reaction chambers, a solution of isophorone-nitrile in tetrahydrofuran is initially reacted with excess ammonia on acidic metal catalysts and the reaction mixture is hydrogenated at high pressure in a second reaction chamber with hydrogen in the presence of excess ammonia on catalysts containing cobalt, nickel, ruthenium and/or other noble metals, and optionally basic components. By way of example, the mixture leaving the first reaction step is passed through the hydrogenation reactor from bottom to top; the reactor is operated as a bubble reactor. Said document does not give any indication of also using the reactor as a trickle bed reactor and directly feeding said reactor with a mixture of isophoronenitrile, ammonia and an organic solvent.

EP-A 0 394 967 teaches a process for the amination of carbonylnitriles and iminonitriles and also includes the preparation of isophoronediamine from isophoronenitrile. The starting material is initially converted to the aminonitrile at moderate temperatures under conditions of



reductive amination, i.e. in the presence of hydrogen, ammonia and a hydrogenation catalyst; the nitrile group is then converted to an aminomethyl group at elevated temperature in the presence of a hydrogenation catalyst which is effective in the hydrogenation of nitrile groups. The process can be carried out either in reactors which use suspension catalysts or in reactors which use fixed bed catalysts. Although this process can be carried out at low pressures, the fact that a strictly observed temperature programme has to be operated during both the reaction steps is regarded as a considerable disadvantage since it results in an appreciable lowering of the space-time yield and hence the economy of the process. In addition, unless special promoters are also used, the product quality does not satisfy the requirements because the content of 3-cyano-3,5,5-trimethylaminocyclohexane, which cannot be separated off by distillation, is usually too high.

According to JP-A-4-300852, a pressure reduction in the preparation of isophoronediamine by the aminating catalytic hydrogenation of isophoronenitrile is also made possible by carrying out the hydrogenation in the presence of a supported ruthenium catalyst. However, said document refers only to conditions for suspension hydrogenation and not to conditions using a fixed bed reactor.

SRI International Report no. 1D "Isocyanates" by YU-REN CHIN (July 1983) gave a survey of an industrial process for the preparation of isophoronediamine from isophorone-nitrile: In the process described here, a mixture of isophoronenitrile, methanol and ammonia is passed with hydrogen over a fixed bed reactor containing a supported catalyst of cobalt-on-kieselguhr. In the embodiment described, the catalyst bed is always flooded (bubble column). The operating pressure is given as 150 bar. Also, hydrogen is used in excess and, after the reaction, this excess must be separated from the reaction mixture,

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- purified, compressed and then recycled. The economy of this process is reduced both by the high operating pressure and by the technical cost of recycling the hydrogen. As established by the Inventors of the present invention - see
- 5 Example 3b not according to the invention - this embodiment results in the increased formation of by-products which cannot easily be separated off, and to a lowering of the yield.
- 10 The object of the present invention thus consists in mitigating the disadvantages of the previously known processes for the preparation of isophoronediamine from isophoronenitrile using a reactor charged with a fixed bed catalyst, to the effect that the economy of the process is
- 15 increased. In particular, the cost of the technical equipment should be reduced compared with the previously known processes. It should moreover be possible to obtain isophoronediamine in high yield and to isolate it from the reaction mixture in very pure form by means of conventional
- 20 distillative measures.

A process for the preparation of 3-aminomethyl-3,5,5-trimethylcyclohexylamine (isophoronediamine, IPDA) from 3-cyano-3,5,5-trimethylcyclohexanone (isophoronenitrile, IPN)

25 has been found in which a mixture of isophoronenitrile, an organic solvent from the group comprising C₁- to C₃-alcohols, and ammonia is hydrogenated with hydrogen at a pressure in the range 3 to 10 MPa and at a temperature in the range 80 to 150°C, in the presence of at least one

30 hydrogenation catalyst from the group comprising cobalt and/or ruthenium fixed bed catalysts, and the reaction mixture is worked up by distillation, said process being characterized in that the aminating hydrogenation is carried out in a trickle bed reactor, the mixture of

35 isophoronediamine, organic solvent and ammonia being charged onto the catalyst bed at the top end of the reactor, and the reaction mixture obtained after trickling



through the catalyst bed being withdrawn at the bottom end of the reactor.

The reaction according to the invention is carried out in a conventional trickle bed reactor. The designs of such reactors are known to those skilled in the art: The fixed bed catalyst is arranged in the form of one or more layers in a container; the reactor is also equipped with devices for thermostating the catalyst layers in order to ensure that the desired temperature is maintained in the respective catalyst layer. As an alternative to a single trickle bed reactor, several trickle bed reactors can be connected in series, the reaction mixture leaving the first reactor being recharged into the top of the second reactor. The trickle bed reactor or reactors are also equipped with suitable devices for charging the reactants, i.e. in this case the mixture containing isophoronenitrile, ammonia and solvent, and the hydrogen, as well as with devices for distributing the liquid over the surface of the first catalyst bed and, finally, with suitable devices for discharging the reaction mixture leaving the reactor.

An essential feature of the process according to the invention is that the reaction mixture to be hydrogenated trickles over the catalyst bed and also that a sufficiently large gas volume exists within the catalyst bed to enable the hydrogen to be transported through the liquid film onto the catalyst surface. It has been found that the trickle bed process is substantially responsible for obtaining a high product yield and, in particular, a high selectivity. If the reactor is operated not as a trickle bed reactor but as a bubble reactor, in which case the catalyst bed is always flooded, a considerably lower yield of isophoronediamine is obtained under otherwise identical reaction conditions in respect of temperature, pressure and concentration of the reactants; the bubble process also gives a different range of by-products, methylisophorone-



diamine (methyl-3-aminomethyl-3,5,5-trimethylcyclohexylamine) being formed in particular. Said methylisophoronediamine cannot be separated from isophoronediamine by distillation under conventional conditions, so the formation of said by-product must already be avoided in the preparation of the isophoronediamine high product. Surprisingly, said by-product is only formed in very small amounts under the conditions of the process according to the invention. A further disadvantage of the bubble process is that hydrogen has to be added in excess in order to achieve saturation of the solution with H_2 ; this entails expensive recycling of the excess H_2 .

When the process according to the invention is carried out, hydrogen can be fed into the reactor either in excess or in an amount such that no hydrogen has to be discharged from the reactor and recycled. The introduction of hydrogen in excess is less preferable because the technical cost of separating off the excess H_2 , condensing ammonia and organic solvent contained in the discharged hydrogen, and compressing the purified hydrogen and recycling it is considerable and reduces the economy of the process on account of the higher investment demanded; moreover, an even lower yield of IPDA has been found compared with the process not using excess H_2 . Thus hydrogen is preferably introduced not in excess but only in the amount which is required to maintain the desired operating pressure.

The aminating hydrogenation is carried out at a pressure in the range 3 to 10 MPa, preferably at 5 to 8 MPa. Said moderate operating pressures, which are possible when using the claimed mixtures of isophoronenitrile, ammonia and solvent and the trickle bed process under the claimed temperature conditions, increase the economy, compared with processes which demand a high operating pressure, by



lowering the level of investment required for the apparatus.

The requisite volume of fixed bed catalyst is governed by
5 the LHSV value (liquid hourly space velocity) (dependent on the operating pressure, the temperature and the catalyst activity) which must be observed in order to achieve a quantitative conversion of isophoronenitrile. Conventionally, the LHSV value is 0.5 h^{-1} or preferably
10 above. The LHSV value is preferably between 0.8 and 1.5 h^{-1} . It is particularly good to attain an LHSV value in said range when the reaction is carried out at a temperature in the range between 90 and 130°C and at a pressure of between 5 and 8 MPa .

15 In addition to the abovementioned parameters of pressure, temperature and catalyst dependence, the LHSV value is also influenced by the chosen concentration ratios of isophorone, ammonia and solvent and by the type of solvent
20 used. The mixture charged onto the catalyst bed conveniently contains 10 to 40% by weight and preferably 10 to 30% by weight of isophoronenitrile and 10 to 40% by weight and preferably 20 to 40% by weight of ammonia. Solvents which can be used are methanol, ethanol,
25 n-propanol and isopropanol. Methanol is the preferred solvent. The isophoronenitrile, ammonia and solvent are mixed in proportions such that an essentially homogeneous solution results. In principle, it is also possible to fall short of or exceed the abovementioned limiting values
30 for ammonia and isophoronenitrile provided a homogeneous solution results. The use of a mixture containing less than 10% by weight of isophoronenitrile has an adverse effect on the economy of the process.

35 Apart from the constituents mentioned above, the mixture to be charged onto the catalyst can contain fractions, boiling above or below isophoronediamine, from the distillative



working-up of the reaction mixture withdrawn from the trickle bed reactor. In addition to isophoronediamine residues, such fractions can also contain by-products from which isophoronediamine is re-formed under the reaction conditions. The yield of isophoronediamine can be markedly increased by recycling such fractions into the mixture to be used. It is particularly advantageous to feed the fraction boiling after the isophoronediamine, which, in addition to isophoronediamine residues, contains 3,3,5-trimethyl-6-imino-7-azabicyclo[3,2-1]octane as the main product, into the trickle bed reactor together with the mixture of isophoronenitrile, ammonia and solvent. Recycling of the fraction containing the abovementioned byproduct - a bicyclic compound of amidine structure - makes it possible significantly to increase the yield of isophoronediamine and consequently to improve the economy of the process.

The catalysts to be used according to the invention are cobalt and/or ruthenium fixed bed catalysts. These catalysts are conventionally in the form of pellets, extrudates or other mouldings. It is particularly convenient to use supported catalysts, cobalt or ruthenium being present on a support of adequate specific surface area. Conventionally, the specific surface area of the supported catalysts to be used according to the invention is in the range between 5 and 500 m²/g (measured with nitrogen by the BET method).

The cobalt content of the supported catalysts is generally between 10 and 70% by weight and preferably between 30 and 50% by weight, based on the supported catalyst. The surface of the support, which is frequently made up of oxide or silicate materials, is partially or essentially completely covered with cobalt, it also being possible for cobalt oxide to be present underneath the surface due to the method of production. Examples of suitable support



materials are oxides from the group comprising silicon dioxide, aluminium oxide and titanium dioxide, silicates such as aluminium and calcium silicate, and naturally occurring and synthetically produced zeolites and glass
5 frits. Although commercially available supported catalysts with a high Co content, such as catalysts containing 50% by weight of Co on aluminium silicate, are described as being essentially completely covered with Co, it has been found that catalysts with a different support but the same
10 specific surface area thereof and the same Co content have an influence on the selectivity and the range of byproducts. Obviously the surface of the catalyst support is at least partially accessible and intervenes in the catalytic process.

15 For reasons of cost, supported catalysts with ruthenium as the catalytically active component generally contain only a small proportion by weight of ruthenium, being conventionally between 0.5 and 10% by weight and preferably
20 between 2 and 5% by weight. The production of the abovementioned Co and Ru catalysts, which conventionally comprises impregnation, drying and shaping steps, is known to those skilled in the art.

25 It has been established that isophoronediamine is obtainable by the process according to the invention in higher yield when using a supported cobalt catalyst than when using a supported ruthenium catalyst. This is surprising inasmuch as the abovementioned documents JP-A
30 4-300852 and EP-A 0 394 967 have indicated that ruthenium catalysts are particularly effective in the hydrogenation of the nitrile group, which is more difficult to hydrogenate than the carbonyl group.

35 It is known that isophoronediamine exists in the form of cis and trans isomers - see Die Angewandte Makromolekulare Chemie 153 (1987) 1-13, no. 2502. By studying the isomer



- ratio of the isophoronediamine prepared according to the invention, it has been found that the proportion of cis isomer formed is completely predominant when using a ruthenium catalyst: cis/trans isomer ratio according to
- 5 Example 9 / 84 to 16. When using a supported cobalt catalyst, the other conditions being identical, the cis and trans isomers are formed in similar proportions: cis/trans isomer ratio according to Example 8 / 60 to 40.
- 10 It has furthermore been found that it is possible to prepare isophoronediamine with a cis/trans isomer ratio above that which is obtainable using a supported cobalt catalyst, and in high yield, by carrying out the aminating
- 15 layer of a supported ruthenium catalyst and a lower layer of a cobalt fixed bed catalyst are arranged. Particularly suitable catalysts are ruthenium-on- γ -aluminium oxide and a supported cobalt catalyst with an oxide or silicate support and a cobalt content of 10 to 70% by weight, based on the
- 20 catalyst. Instead of having one reactor with two layers of different fixed bed catalysts arranged therein, it is also possible to connect two reactors in series, each containing only one fixed bed catalyst; in this case, the first reactor contains the ruthenium fixed bed catalyst and the
- 25 second reactor contains the cobalt fixed bed catalyst. To obtain a high yield of isophorone with the desired isomer ratio, it is advantageous if the ruthenium fixed bed catalyst, which is positioned first, makes up 3 to 30%, preferably 5 to 20%, of the total catalyst volume.
- 30 As explained earlier, the process according to the invention differs in essential respects from previously known processes of this kind, resulting in substantial advantages. By combining the use of a starting mixture of
- 35 isophoronitrile, ammonia and an alcoholic solvent and the trickle bed operation of the reactor provided with a fixed bed, it is possible to achieve both a high product yield of



isophoronediamine and a high selectivity. The formation of undesired by-products such as methylisophoronediamine, which adversely affects the product quality because it is difficult to separate off, is extensively avoided by the reaction process. The abovementioned combination of essential features of the invention is also such that the aminating hydrogenation can be carried out at moderate pressures, thereby greatly reducing the consequent cost of the pressure apparatus compared with the case of a high-pressure apparatus. As it is not necessary according to the invention to use hydrogen in excess and hence to have to purify and recycle the excess, in contrast to previously known processes using a reactor with a fixed bed catalyst, expensive devices for separating off, purifying and recycling the excess H_2 are dispensed with. Another essential advantage of the process according to the invention is the unexpectedly long service life of the catalysts, which is attributed to the trickle bed process: Even after several months of operation, no reduction in yield, no change in the range of by-products and no change in the isomer ratio were found. A further advantage is the ability to obtain isophoronediamine in high yield with a particular isomer ratio, within certain limits. In the process according to the invention, there is also no need for a preliminary reaction between isophoronenitrile and ammonia in order to form isophoroneimine, which was considered in several documents to be essential for obtaining a high yield and high selectivity in the aminating hydrogenation; according to the invention, by contrast, a solution of isophoronenitrile in the solvent used is mixed with ammonia directly upstream of the trickle bed reactor and the mixture is fed into the reactor.

The process according to the invention is illustrated in greater detail with the aid of the following Examples, without implying a limitation.



Example 1

A reaction tube is filled with 200 ml of hydrogenation catalyst. The feed solution containing IPN and methanol, and liquid NH_3 , are pumped into the top of the reactor. Hydrogen also flows into the tube from the top. The reaction temperature is kept at 120°C by means of oil heating. The pressure is regulated at 60 bar. The liquid is collected in a separating vessel. The gas stream at the reactor inlet is adjusted so that a gas stream of 100 Nl/h is established downstream of the separating vessel.

The catalyst used was a commercially available solid Co catalyst (ca. 50% Co on silicate support) in the form of extrudates of 4-5 mm in diameter and length. The feed solution contained 24% by weight of IPN and 76% by weight of methanol. 130 ml/h thereof and 70 ml/h of NH_3 were mixed directly before being charged into the reactor, and the mixture was pumped into the reactor; the LHSV value was thus 1 h^{-1} . According to analysis of the product mixture, the yield of IPDA was 88.7%, based on IPN used. The product mixture also contained 5% of 2-aza-4,6,6-trimethylbicyclo[3,2,1]octane (= bicyclic compound) and 4.3% of 3,5,5-trimethyl-6-imino-7-azabicyclo[3,2,1]octane (= amidine).

Example 2

The experiment was carried out in the same experimental set-up as already described in Example 1, except that no gas stream was withdrawn from the separating vessel, so only the consumed hydrogen was replaced in order to keep the reaction pressure at 60 bar. The catalyst, liquid streams and liquid composition were the same as described in Example 1.

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Analysis of the product mixture of this experiment showed an IPDA yield of 89.2%, based on IPN used. The product mixture also contained 4.5% of bicyclic compound and 4.4% of amidine. Surprisingly, an even higher yield of IPDA and
5 a reduction in the "bicyclic" by-product are made possible by the process without excess H_2 . This experiment was run for 2000 h with no observable loss of yield or change in the range of products.

10 Examples 3a and 3b

The experimental set-up used was the same as in Experiment 1. The hydrogenation pressure was 60 bar and the reaction temperature was 120°C. The H_2 stream was adjusted so that
15 100 NL/h of H_2 were withdrawn from the separating vessel. The feed solution contained 30% by weight of IPN and 70% by weight of methanol. 130 ml/h thereof were pumped into the reactor together with 50 ml/h of liquid NH_3 . 200 ml of cobalt catalyst were introduced as the catalyst. Two
20 processes were studied. In the trickle bed process (3a), gas and liquid flowed through the reactor from top to bottom; in the bubble column process (3b), the reactor was in the flooded state and they flowed through it from bottom to top.

25 A yield of 87.1% was achieved in the trickle bed process (3a). After purification by distillation, the product purity was 99.8%; methyl-IPDA was detectable in an amount of only about 200 ppm.

30 A yield of 85.5% was achieved in the experiment by the bubble column process (3b). Purification by distillation gave a product purity of only 99.5%. Analysis showed that the pure product still contained 3000 ppm of methyl-IPDA.
35 The methyl-IPDA by-product, which has a very adverse effect on the quality of the IPDA product, is practically inseparable by distillation.



Examples 4 to 6

The experiments were carried out in the same experimental set-up and under the same reaction conditions as described in Example 1. Only the catalyst was varied. The results are given in the Table.

Example	Catalyst	Yield of IPDA (%)
4	5% Ru/ γ -Al ₂ O ₃	82.1
5	Ni	55.3
6	2% Pd/ γ -Al ₂ O ₃	4

Both the Ru and the Pd were applied to γ -Al₂O₃ extrudates as the support. Examples 5 and 6 clearly show that the yields obtained with Ni and Pd catalysts are not satisfactory compared with the process according to the invention.

Example 7

The low-boiling components, namely methanol, NH₃, H₂O and the bicyclic compound, were removed from 8770 g of the reaction solution of Example 2 by fractional distillation.

1292 g of a crude solution remained. 1190 g of IPDA were distilled off from this solution at a bottom temperature of 147°C and at a pressure of 20 mbar. This corresponds to an isolation yield of 87.3%. 98 g remained as the bottom product of the distillation. A further high-boiling fraction of 74 g was distilled off from this bottom product under reduced pressure (10 mbar) and at a temperature of 162°C. It contained 20% of IPDA and 74% of amidine. This resulting fraction of 74 g corresponds to 5.6% of the amount of IPN used.

In another experiment, which was carried out analogously to Example 2, the high-boiling fraction, i.e. 5.6% based on



the amount of IPN used, was added. After hydrogenation and separation of the low-boiling components and purification of the IPDA by distillation, the isolation yield found was 92.3%. Accordingly, it was possible to achieve a 5% increase in the isolation yield of IPDA by recycling the high-boiling fraction containing the amidine.

The yield of IPDA can be increased by a further 4% by catalytically hydrogenating the fraction containing the bicyclic compound (boiling point below that of IPDA) in a separate hydrogenation reactor under elevated temperature conditions.

Examples 8 to 11

The experimental set-up used was the same as in Experiment 1. The hydrogenation pressure was 60 bar and the reaction temperature was 120°C. No H₂ was withdrawn from the separating vessel. The feed solution contained 30% by weight of IPN and 70% by weight of methanol. 130 ml/h thereof were mixed with 50 ml/h of liquid NH₃ and pumped into the reactor. This experimental set-up was operated with a ruthenium catalyst, a cobalt catalyst and various combinations of the two. The results of this experiment are shown in the Table.

	Example	Catalyst*)	Yield (%)	Isomer ratio cis:trans
30	8	200 ml Co	87.2	60:40
	9	200 ml Ru	81.5	84:16
	10	100 ml Ru + 100 ml Co	83.7	81:19
	11	10 ml Ru + 190 ml Co	86.9	78:22

*) Ru catalyst: 5% Ru on γ -Al₂O₃ extrudates

Co catalyst: 50% Co on Al silicate extrudate

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By combining an Ru catalyst and a Co catalyst, the Ru catalyst being arranged as the upper layer in the reactor, it is possible to obtain IPDA with a high cis/trans isomer ratio in higher yield than by using an Ru catalyst on its own.

Example 12

A reaction tube (diameter 16 mm) of a trickle bed hydrogenation apparatus was filled with 120 ml of commercially available supported Co catalyst (Co on aluminium silicate, 50% by weight of Co; BET surface area (N_2) ca. 200 m²/g). The experiment was carried out analogously to Example 1. The reaction temperature was adjusted to 110°C and the total pressure (sum of the partial pressures of H₂, NH₃ and methanol) was adjusted to 60 bar. The starting solution contained 15% by weight of isophoronenitrile, 65% by weight of methanol and 20% by weight of ammonia. 100 ml of this starting solution were introduced onto the trickle bed per hour. After the solution had passed through the trickle bed, the reaction mixture contained the following (mol% based on isophorone-nitrile in the starting solution):

Isophoronediamine (IPDA)	89.2%
Isophoroneamino alcohol (IPAA)	2.1%
Amidine (see Example 1)	4.0%
Bicyclic compound (see Example 1)	3.8%

Example 13

Example 12 was repeated, the only difference being that another commercially available supported Co catalyst was used - Co on silica, 25% by weight of Co, BET surface area (N_2) ca. 200 m²/g. The reaction mixture contained the following (data in mol% based on starting isophorone-nitrile):

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IPDA	87.4%
IPAA	1.2%
Amidine	4.3%
5 Bicyclic compound	5.1%

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Claims

1. A process for the preparation of 3-aminomethyl-3,5,5-trimethylcyclohexylamine (isophoronediamine, IPDA) from 3-cyano-3,5,5-trimethylcyclohexanone (isophorone-nitrile, IPN) in which a mixture of isophoronenitrile, an organic solvent from the group comprising C₁- to C₃-alcohols, and ammonia is hydrogenated with hydrogen at a pressure in the range 3 to 10 MPa and at a temperature in the range 80 to 150°C, in the presence of at least one hydrogenation catalyst from the group comprising cobalt and/or ruthenium fixed bed catalysts, and the reaction mixture is worked up by distillation, said process being characterized in that the aminating hydrogenation is carried out in a trickle bed reactor, the mixture of isophoronediamine, organic solvent and ammonia being charged onto the catalyst bed at the top end of the reactor, and the reaction mixture obtained after trickling through the catalyst bed being withdrawn at the bottom end of the reactor.
2. A process for the preparation of isophoronediamine according to Claim 1, characterized in that the hydrogenation is carried out at a temperature in the range 90 to 130°C and at a pressure of 5 to 8 MPa.
3. A process according to Claim 1 or 2, characterized in that methanol is used as the solvent, the mixture charged onto the catalyst bed contains 10 to 40% by weight, preferably 10 to 30% by weight, of isophorone-nitrile and 10 to 40% by weight, preferably 20 to 40% by weight, of ammonia and the mixture forms a homogeneous solution.





4. A process according to one or more of Claims 1 to 3, characterized in that the hydrogenation catalyst used is a supported catalyst consisting essentially of an inorganic support material, preferably an oxide or silicate support material, and cobalt, the catalyst containing a total of 10 to 70% by weight of cobalt.
5. A process according to one or more of Claims 1 to 4, characterized in that a high-boiling fraction distilling after the isophoronediamine main fraction in the distillative working-up of the reaction mixture, and containing, in addition to isophoronediamine residues, 3,3,5-trimethyl-6-imino-7-azabicyclo[3,2,1]octane as the main component, is fed into the trickle bed reactor together with the mixture of isophoronitrile, solvent and ammonia.
6. A process according to one or more of Claims 1 to 5, characterized in that the aminating hydrogenation is carried out in a trickle bed reactor with an upper layer of a supported ruthenium catalyst, preferably ruthenium-on- γ -aluminium oxide, and a lower layer of a cobalt fixed bed catalyst arranged therein, it being possible for said upper layer and said lower layer also to be arranged in two separate reactors connected in series in the stated order, instead of in one reactor.
7. A process according to Claim 6, characterized in that the volume of the ruthenium-containing fixed bed catalyst makes up 3 to 30%, preferably 5 to 20%, of the total volume of fixed bed catalysts.
8. A process according to one or more of Claims 1 to 7, characterized in that hydrogen is fed into the top or bottom end of the trickle bed reactor in an amount



such that no hydrogen has to be discharged from the reactor and recycled.

9. A process according to one or more of Claims 1 to 8,
5 characterized in that the mixture of isophorone-
nitrile, solvent and ammonia, which preferably
contains 10 to 30% by weight of isophoronenitrile,
20 to 40% by weight of ammonia and methanol as the
10 solvent, is allowed to trickle over the catalyst bed
of the reactor with an LHSV value (liquid hourly space
velocity) of 0.8 to 1.5 h⁻¹.

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