

Process for the production of 3-aminomethyl-3,5,5-trimethylcyclohexyl amine

Description

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The invention relates to an improved process for producing 3-aminomethyl-3,5,5-trimethylcyclohexyl amine, also referred to below as isophorone diamine or IPDA for short, from 3-cyano-3,5,5-trimethylcyclohexanone, also referred to below
10 as isophorone nitrile or IPN for short, comprising a first stage for the at least partial conversion of isophorone nitrile into isophorone nitrile imine and a second stage for the aminizing hydrogenation of the reaction mixture of the first stage with hydrogen and ammonia in the presence of a
15 lower alcohol and a fixed bed hydrogenating catalyst based on Raney cobalt. The process according to the invention permits the continuous production of isophorone diamine in high purity with higher yield and/or higher space-time yield than known two-stage processes according to the preamble.

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Isophorone diamine is used as a starting product for the production of isophorone diisocyanate, an isocyanate component for polyurethane systems, as an amine component for polyamides and as a hardener for epoxy resins.

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Isophorone diamine is produced conventionally from isophorone nitrile, in which the carbonyl group is converted into an amino group and the nitrile group into an aminomethyl group in the presence of ammonia, hydrogen and conventional hydrogenation catalysts. The starting product
30 isophorone nitrile can be obtained in conventional manner by the addition of hydrogen cyanide to isophorone - see DE-OS 39 42 371.

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According to the process for producing isophorone diamine from isophorone nitrile described in US 3 352 913, the hydrogenation takes place 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 a pressure of at least 50 bar. For example the hydrogenation takes place in the presence of methanol as solvent with the use of suspended and fixed bed catalysts. In addition to the
5 desired isophorone diamine, large amounts of by-products are also obtained, e.g. in particular 3-aminomethyl-3,5,5-trimethylcyclohexanol (isophorone amino alcohol, IPAA). The low yield and the considerable proportion of by-products prove to be disadvantages of this system.

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In an attempt to obtain a higher yield of IPDA and to minimize the unavoidable incidence of IPAA, DE-OS 30 11 656 reveals a two-stage process, in which in the first stage IPN is converted catalyst-free with surplus ammonia into 3-
15 cyano-3,5,5-trimethyl-iminocyclohexane and the latter is hydrogenated to IPDA in the second stage. A disadvantage of this process is that in addition to the actual hydrogenation reactor a special imine formation reactor is required.

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According to EP-B 0 042 119 it is regarded as a further improvement of the process to subject the isophorone nitrile, prior to the reaction of the latter with ammonia and hydrogen in the presence of hydrogenation catalysts at temperatures of 10 to 120 °C and pressures of 1 to 300 bar,
25 to a preliminary reaction with ammonia in the presence of inorganic and organic ion exchangers in the ammonium form as imine formation catalysts. Whereas the ratio by volume of isophorone nitrile to ammonia is to vary between 1 and 0.5 to 20 in the imine formation stage, this ratio is increased
30 to between 1 and 10 to 20 in the hydrogenation stage.

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Although the process, which can be carried out in trickle bed reactors, certainly leads to a high yield of IPDA and to a high purity, the profitability of the process is impaired by the high surplus of ammonia, which makes a very high
35 pressure and hence sophisticated hydrogenation equipment necessary.

According to DE-A 44 26 472 it is also possible for isophorone nitrile to be converted with the use of a supported heteropoly acid catalyst with ammonia into the isophorone nitrile amine and for the latter to be converted
5 into isophorone diamine with the use of conventional fixed bed hydrogenation catalysts, including also Raney® cobalt. The high hydrogenating pressures required in practice - for example 238 bar - are disadvantageous, which calls for highly sophisticated equipment.

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In EP-B 0 042 119 a comparative example is also disclosed in which isophorone nitrile and liquid ammonia are pumped from above into a fixed bed hydrogenation reactor charged with commercial cobalt catalyst. The reaction system is held at
15 270 bar with H₂. There is obtained with this single-stage form of execution, despite roughly quantitative conversion of the isophorone nitrile, after working up by distillation only 48% of isophorone diamine together with many by-products. According to DE-A 43 43 890 the pressure was able
20 to be reduced to 3 to 10 MPa by the use of a C₁- to C₃-alcohol as solvent. With a trickle bed method of operation a yield of just under 90% was achieved with an LHSV value of 1 h⁻¹ (liquid hourly space velocity) and without recycling of re-crackable high-boiling by-products.

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A further two-stage process for producing isophorone diamine is known from EP-A 0 449 089: in two reaction chambers physically separated from one another a solution of isophorone nitrile is first of all reacted in tetra-
30 hydrofuran with surplus ammonia on acid metal catalysts and the reaction mixture is hydrogenated in a second reaction chamber with hydrogen in the presence of surplus ammonia on cobalt-, nickel-, ruthenium- and/or other noble metal-containing catalysts and optionally basic components at very
35 high pressure. The high pressure makes very sophisticated hydrogenation equipment necessary.

EP-A 0 394 967 also covers the production of isophorone diamine from isophorone nitrile, wherein IPN is first of all converted into the aminonitrile under conditions of reductive amination in the presence of a first hydrogenation catalyst at moderate temperatures and the nitrile group is then converted into an aminomethyl group at higher temperature in the presence of a second hydrogenation catalyst having a hydrogenating effect towards nitrile groups. Although this process can be carried out at low pressures, it is regarded as a major disadvantage that a strict temperature regime has to be observed during the two reaction stages, as a result of which the space-time yield and hence the profitability of the process decline. Unless special promoters are additionally used, the IPDA product quality does not meet the requirements because of the mostly excessively high content in 3-cyano-3,5,5-trimethyl-aminocyclohexane, which is impossible to separate by distillation.

In the process according to JP-A 06-321870 an equilibrium mixture of isophorone nitrile and isophorone nitrile imine obtained from isophorone nitrile in the absence of an iminization catalyst, but in the presence of methanol and ammonia, is subjected to aminizing hydrogenation in the presence of a fixed bed catalyst containing a catalyst metal from the series Co, Ni, Ru and Pd, preferably Co on diatomite. The yield in IPDA is only 88%, and in addition the space-time yield is low. According to JP-A 07-188126 use is made in the aforementioned process for the aminizing hydrogenation of a fixed bed catalyst based on Raney cobalt, wherein the catalyst is obtained by the elimination by melting of aluminium out of a binary or ternary Raney alloy. In this case also the yield of IPDA given in the examples comes to only some 88%.

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The object of the present invention is to set forth a further two-stage process for producing IPDA and IPN, which

leads in a reliable manner to a higher yield of IPDA and/or higher space-time yield. IPDA should also be obtainable in high product purity. The hydrogenation stage should also be capable of being carried out at a pressure of not more than 10 MPa, preferably below 8 MPa, in order to keep the cost of equipment as low as possible.

The object is achieved by a process for producing 3-aminomethyl-3,5,5-trimethylcyclohexyl amine (isophorone diamine, IPDA) from 3-cyano-3,5,5-trimethylcyclohexanone (isophorone nitrile, IPN), comprising a first stage, in which isophorone nitrile is in the presence or absence of an iminization catalyst converted with ammonia at least partially into 3-cyano-3,5,5-trimethylcyclohexane imine, and a second stage, in which the reaction mixture of the first stage, not yet present on the whole, is, after the addition of an alcohol with 1 to 3 carbon atoms, subjected to aminizing hydrogenation with hydrogen with the use of a fixed bed hydrogenation catalyst based on Raney cobalt at a temperature in the range from 50 to 150 °C and a pressure in the range from 3 to 10 MPa, which is characterised in that the hydrogenation catalyst to be used has been produced by a process incorporating the steps: (i) intimate mixture of at least one powdery cobalt alloy and powdery cobalt as binder, wherein the cobalt alloy contains cobalt and optionally promoters as well as a leachable alloy component from the series aluminium, zinc and silicon and the powdery mixture contains cobalt and leachable alloy components in the ratio by weight of between 30 to 70 and 75 to 25, (ii) sintering of the powdery mixture to a mechanically stable moulding with a density of 1.3 to 5.5 g/cm³, a pore volume of 0.5 cm³/g (water adsorption) and a BET surface area (DIN 66 132) of less than 1 m²/g and (iii) activation of the sintered moulding by at least partial leaching out of the

leachable alloy component by means of an alkali hydroxide solution.

The fixed bed catalyst to be used according to the invention
5 for the aminizing hydrogenation of the reaction mixture
obtained from the first stage and containing isophorone
nitrile imine and isophorone nitrile is obtainable by the
process described in DE-A 43 35 360 and DE-A 43 45 265. As
regards the selection and physical characteristics of the
10 raw materials to be used in the production - catalyst metal,
alloy and, where required, promoters and auxiliary
substances such as moulding aids, lubricants, plasticizers
and/or pore formers - and physical characteristics of the
catalyst precursor as well as details of the process stages,
15 namely mixing of the components, sintering of the powder
mixture to mouldings of the catalyst precursor and
activation of the catalyst by at least partial leaching of
the leachable alloy component out of the catalyst precursor,
reference is made to the above-mentioned documents.

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There is given as the area of use for the catalysts
described in the aforementioned documents (DE-A 43 35 360
and DE-A 43 45 265) the hydrogenation of nitro groups, C-C
double bonds, sugars and aromatic rings. Indications or a
25 suggestion that imines and nitrile groups should also be
subjected to aminizing hydrogenation with these catalysts
cannot be derived from the above-mentioned documents. It was
surprising that the use of these special fixed bed catalysts
based on Raney cobalt in the two-stage process according to
30 the preamble for producing IPDA from IPN leads to a higher
yield with in most cases even simultaneously higher space-
time yield. It was also found that corresponding catalysts
based on Raney nickel are considerably less effective in the
two-stage production of IPDA from IPN than those based on
35 cobalt. Compared also with Raney cobalt fixed bed catalysts
used before in the process according to the preamble, the
catalysts to be used according to the invention showed a

significantly higher catalyst effectiveness in the second stage.

In the first stage of the process according to the invention
5 at least one part, preferably the greatest part, of the
isophorone nitrile introduced is converted into isophorone
nitrile imine. The reaction mixture leaving the iminization
stage should as far as possible contain isophorone nitrile
imine and isophorone nitrile in a molar ratio of more than
10 1. The iminization of the isophorone nitrile is conducted
with expediency up to a conversion rate of over 80%,
preferably over 90%.

If the iminization is carried out in the absence of an
15 iminization catalyst, several hours are required at a
reaction temperature in the range between 10 and some 60 °C
to achieve the desired iminization rate; although it is
certainly also possible for the iminization to be carried
out in the absence of a catalyst at a higher temperature
20 than 60 °C, for example at 100 °C, there is then the risk of
an increased formation of by-products, as a result of which
the product purity of the isophorone diamine obtained from
the reaction mixture of the second stage after working up by
distillation is adversely affected. It is advisable to use
25 an iminization catalyst in order to accelerate the
establishment of equilibrium in the first stage. The
iminization catalysts known from the prior art can be used
for this. Suitable iminization catalysts are acid inorganic
and organic ion exchangers (see EP-B 0 042 119), acid metal
30 oxides, such as in particular aluminium oxide and titanium
oxide (anatase) (see EP-A 0 449 089), supported heteropoly
acids (see DE-A 44 26 472) and acid zeolites. If an
iminization catalyst is used, the reaction temperature can
lie in the range between 10 and 150 °C, preferably between
35 60 and 130 °C and in particular between 80 and 120 °C.

Although the iminization of isophorone nitrile with liquid ammonia in the absence of a further solvent is possible, it has proved to be advantageous to use additionally a solvent from the series of an alcohol with 1 to 3 carbon atoms, preferably of a monovalent primary alcohol and in particular methanol. Preferably there is fed to the iminization reactor a mixture containing isophorone nitrile, liquid ammonia and methanol. The mixture contains with expediency 10 to 40 wt %, preferably 10 to 30 wt %, of isophorone nitrile and 10 to 40 wt %, preferably 20 to 40 wt %, of ammonia. It is advantageous to mix isophorone nitrile, ammonia and the alcohol together in a ratio such that an essentially homogeneous solution is obtained. In principle the aforementioned limit values for ammonia and isophorone nitrile can also be fallen short of or exceeded, if an essentially homogeneous solution is thereby obtained.

In the case of iminization in the presence of an iminization catalyst the catalyst can be used in the form of a suspended catalyst or a fixed bed catalyst. The use of a fixed bed catalyst is advantageous, since elaborate steps for the separation of the reaction mixture from the catalyst then become superfluous. In the case of iminization of isophorone nitrile in the presence of a fixed bed catalyst the latter is employed in the form of conventional catalyst mouldings, such as extrusion mouldings, pellets and tablets, as a bed in a fixed bed reactor. The iminization catalyst can be arranged in its own reactor. It is also possible, however, to arrange the iminization catalyst in a reactor which contains both a bed of the iminization catalyst and a bed of the catalyst used for the aminizing hydrogenation. Depending on whether the reactor is operated as a trickle bed reactor or as a bubble reactor, the bed of the iminization catalyst is located above (trickle bed) or below (bubble reactor) the bed of the hydrogenation catalyst. It has proved to be advantageous to use a single reactor which contains a bed of the hydrogenation catalyst and a bed of the iminization

catalyst; preferably such a reactor is operated in the form of a trickle bed reactor. The mixture of isophorone nitrile, liquid ammonia and alcohol, in particular methanol, is then supplied at the reactor head. In these cases hydrogen
5 advantageously flows into the reactor simultaneously from above for the aminizing hydrogenation.

In addition to the aforementioned components of the mixture to be fed to the iminization stage, the latter can contain
10 additionally fractions with a higher or lower boiling point than isophorone diamine from the working up by distillation of the reaction mixture drawn off from the trickle bed reactor. Such fractions can also contain, in addition to residues of isophorone diamine, by-products from which
15 isophorone diamine again forms under the reaction conditions. The yield in isophorone diamine can be increased substantially by returning fractions of this kind into the mixture to be used. It is particularly advantageous to feed to the trickle bed reactor the fraction with a boiling point
20 above isophorone diamine, which in addition to residues of isophorone diamine contains 3,3,5-trimethyl-6-imino-7-aza-bicyclo-[3,2-1]-octane as main product, together with the mixture of isophorone nitrile, ammonia and solvent. The recycling of the fraction containing the above-mentioned by-
25 product - a bicyclic compound of amidine structure - makes it possible to increase the yield in isophorone diamine considerably and hence to enhance the profitability of the process. The fraction containing the bicyclic amidine can, if this is desired, also be added directly to the reaction
30 mixture to be fed to the second stage.

The catalyst used in the second stage of the process according to the invention is a fixed bed catalyst. Conventional fixed bed reactors are used to carry out this
35 process stage. As already mentioned in connection with the iminization stage, the reactor can be operated both as a trickle bed reactor and as a bubble column, although, as

follows also from DE-A 43 43 390, the trickle bed method of operation is preferred.

The layouts of the reactors are known to the skilled man:
5 the fixed bed catalyst is arranged in a vessel in the form of one or more beds; the reactor also possesses devices for controlling the temperature of the catalyst beds, in order to ensure that the desired temperature is maintained in the particular catalyst bed. Instead of a single trickle bed
10 reactor, several trickle bed reactors can also be connected one behind the other, wherein the reaction mixture leaving the first reactor is charged again at the head of the second reactor. The trickle bed reactor or reactors also possess suitable devices for charging the reaction mixture and the
15 hydrogen, also devices for distributing the liquid over the surface of the first catalyst bed and finally suitable removal devices for the reaction mixture leaving the reactor.

20 The aminizing hydrogenation, i.e. the second reaction stage, is carried out at a temperature in the range of 80 to 150 °C, preferably 90 to 130 °C. The aminizing hydrogenation takes place at a pressure in the range of 3 to 10 MPa, preferably at 5 to 8 MPa and in particular below 8 MPa. Due
25 to the above-mentioned moderate operating pressures, which are possible with the use of the claimed mixtures of isophorone nitrile, ammonia and solvent and the trickle bed method of operation under the claimed temperature conditions, the investment volume is reduced and hence the
30 profitability increased compared with processes which require a high operating pressure. By the specified pressure is meant the overall pressure, which is composed of the partial pressures of ammonia, hydrogen, C₁-C₃-alcohol and the other components of the reaction mixture.

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The volume of fixed bed catalyst required for the second stage is determined by the LHSV value (liquid hourly space

- velocity), which is dependent on the operating pressure, the temperature and the catalyst activity and must be adhered to in order to achieve as quantitative a conversion of the mixture containing isophorone nitrile imine and isophorone nitrile as possible. The LHSV comes conventionally to at least 0.5 h^{-1} and preferably lies in the range above 0.5 h^{-1} to 3 h^{-1} . It is an advantage of the process according to the invention that a virtually quantitative conversion is achieved, and IPDA can be obtained in high yield and high purity, with an LHSV value of about 2 h^{-1} , a reaction temperature in the range between 90 and 130°C and a pressure between 5 and less than 8 MPa . The high LHSV value leads in addition to a high space-time yield.
- 15 In the embodiment particularly preferred according to the invention, in which a trickle bed reactor contains a lower bed of hydrogenation catalyst and an upper bed of iminization catalyst, the respective bed height is adjusted to the corresponding catalyst activity - the skilled man will determine this activity and hence the bed height by means of simply performed preliminary tests. It follows from the examples that if titanium dioxide is used as iminization catalyst, a bed height which comes to about a quarter of that of the hydrogenation catalyst is sufficient.
- 25 The hydrogen required for the hydrogenation can be fed to the reactor either in excess or in an amount such that no hydrogen has to be discharged from the reactor and recycled. Preferably hydrogen is not fed in excess, in order to avoid the technical complexities involved in the separation of this excess, the condensation of the ammonia and solvent contained in the latter, and the compression of the purified hydrogen and recycling.
- 30 The process according to the invention is distinguished by its simplicity, low investment volume, high yield, high space-time yield and high IPDA product purity.
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Example 1

- A reaction tube operated as a trickle bed reactor was filled in the lower region with 160 ml of hydrogenation catalyst and in the region lying above the latter with 40 ml of iminization catalyst. Titanium dioxide (titanium dioxide P 25 of Degussa AG) in the form of 1 mm extrudates was used as iminization catalyst. Activated Raney cobalt catalyst, produced according to DE patent application 43 45 265.5, in the form of tablets 5 mm in height and 3 mm in diameter, was used as hydrogenation catalyst. The batch solution, which contained IPN and methanol, as well as liquid ammonia were pumped into the reaction tube from above. The hydrogen was also introduced into the reaction tube from above. The reaction temperature was held at 100 °C by an oil heater. The pressure was set at 6 MPa. The liquid was collected in a separation vessel. The gas current at the reactor inlet was adjusted so that the whole of the hydrogen was consumed.
- The batch solution contained 24 wt % of IPN and 76 wt % of methanol. 260 ml/h of this batch solution and 140 ml/h of ammonia were mixed directly prior to charging to the reactor and the mixture was pumped into the reactor. The LHSV value therefore came to 2h^{-1} .
- According to the analysis of the product mixture a yield of 92.2% of isophorone diamine (= IPDA), referred to IPN used, was obtained. In addition 3% of 2-aza-4,6,6-trimethyl-bicyclo-3,2,1-octane (= bicyclic compound) and 3.3% of 3,5,5-trimethyl-6-imino-7-aza-bicyclo-3,2,1-octane (= amidine) were contained in the product mixture. A product purity of 99.8% was obtained after the distillation of the liquid product mixture. Methyl-IPDA was detectable in a quantity of only 200 ppm.
- It follows from a comparison with the following comparative examples 1 and 2 that the use of the hydrogenation catalyst

according to the invention leads by virtue of its higher hydrogenation activity to a surprisingly high increase in the yield and space-time yield.

5 Comparative example 1

A reaction tube operated as a trickle bed was filled with 160 ml of hydrogenation catalyst in the lower region and with 40 ml of iminization catalyst in the region lying above the latter. Whereas the iminization catalyst corresponded to that of Example 1 (TiO_2 , P 25 of Degussa AG), there was used as hydrogenation catalyst a commercial supported cobalt catalyst based on 50% cobalt on a silicate support (injection mouldings of 4 - 5 mm diameter and height). The operating conditions - pressure, temperature, LHSV value, flow rate and concentration of the methanolic IPN batch solution and flow rate of the liquid ammonia - corresponded to those of Example 1.

20 According to the analysis of the product mixture a yield of 82% of IPDA was obtained.

Comparative example 2

25 Comparative example 1 was repeated, the only difference being that the liquid flow rates were changed: 130 ml/h of methanolic IPN batch solution (24 wt % IPN) and 70 ml/h of liquid ammonia were mixed directly prior to charging to the reactor and the mixture was pumped into the reactor. The LHSV value therefore came to 1 h^{-1} .

According to the analysis of the product mixture a yield of 90.7% of IPDA was obtained, referred to IPN used. In addition 4% of bicyclic compound and 3.2% of amidine were contained in the product mixture. Distillation of the product mixture produced a product purity of 99.8 GC-Fl. %.

The increase in the yield, which still lay below that of Example 1 according to the invention, was unfortunately accompanied by a halving of the space-time yield.

5 **Comparative example 3**

The conversion was carried out in a similar way to Example 1, but using inert steel balls 2 mm in diameter instead of the TiO_2 iminization catalyst. All the other test conditions
10 conformed to Example 1.

According to the analysis of the product mixture a yield of 73.7% of IPDA was obtained.

15 This comparative example shows that in the absence of an effective iminization catalyst, but using an activated Raney cobalt catalyst according to the invention as hydrogenation catalyst, only a moderate IPDA yield is obtained.

20 **Example 2**

A solution containing 15 wt % of IPN, 30 wt % of ammonia and 55 wt % of methanol was produced and stirred in a pressure vessel under the pressure obtained for 24 h at 25 °C; in
25 this case isophorone nitrile (IPN) was converted largely into isophorone imino nitrile (IPIN). This solution was then pumped at 400 ml/h, corresponding to an LHSV value of 2 h⁻¹, through the reactor, which was filled with 160 ml of hydrogenation catalyst according to the invention (as in
30 Example 1) and 40 ml of inert steel balls. According to the analysis of the product mixture a yield of 92.1% of IPDA was obtained. After distillation of the product mixture a product purity of 99.78% IPDA was obtained.

Example 3

In a reaction tube filled according to Example 1 the reaction - iminization with subsequent hydrogenation - was carried out as per Example 1, the only difference being that an LHSV value of 1 h^{-1} was set. According to the analysis of the product mixture a yield of 94.2% of IPDA was obtained, referred to IPN used. In addition bicyclic compound and 1% of amidine were contained in the product mixture.

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The example shows that the yield in IPDA was able to be increased still further by a lowering of the LHSV value (compared with Example 1).

Claims

1. Process for producing 3-aminomethyl-3,5,5-trimethylcyclohexyl amine (isophorone diamine, IPDA)
5 from 3-cyano-3,5,5-trimethyl-cyclohexanone (isophorone nitrile, IPN),
comprising a first stage,
in which isophorone nitrile is in the presence or
absence of an iminization catalyst converted with
10 ammonia at least partially into 3-cyano-3,5,5-trimethylcyclohexane imine,
and a second stage,
in which the reaction mixture of the first stage, not
yet present on the whole, is, after the addition of an
15 alcohol with 1 to 3 carbon atoms, subjected to
aminizing hydrogenation with hydrogen with the use of a
fixed bed hydrogenation catalyst based on Raney cobalt
at a temperature in the range from 50 to 150 °C and a
pressure in the range from 3 to 10 MPa,
20 characterised in that
the hydrogenation catalyst to be used has been produced
by a process incorporating the steps: (i) intimate
mixture of at least one powdery cobalt alloy and
powdery cobalt as binder, wherein the cobalt alloy
25 contains cobalt and optionally promoters as well as a
leachable alloy component from the series aluminium,
zinc and silicon and the powdery mixture contains
cobalt and leachable alloy components in the ratio by
weight of between 30 to 70 and 75 to 25, (ii) sintering
30 of the powdery mixture to a mechanically stable
moulding with a density of 1.3 to 5.5 g/cm³, a pore
volume of 0.5 cm³/g (water adsorption) and a BET
surface area (DIN 66 132) of less than 1 m²/g and (iii)
activation of the sintered moulding by at least partial
35 leaching out of the leachable alloy components by means
of an alkali hydroxide solution.



2. Process according to claim 1,
characterised in that
in the first stage an iminization catalyst from the
series of acid oxides, zeolites, acid ion exchangers
and supported heteropoly acids is used and the
iminization is carried out in the presence of a C₁- to
C₃-alcohol.
3. Process according to claim 2,
characterised in that
the iminization catalyst is used in the form of
mechanically stable mouldings in a fixed bed reactor.
4. Process according to one of claims 1 to 3,
characterised in that
the fixed bed hydrogenation catalyst is arranged in a
hydrogenation reactor and the reactor is operated as a
trickle bed reactor.
5. Process according to one of claims 1 to 4,
characterised in that
in a fixed bed reactor a bed of a fixed bed iminization
catalyst is arranged above a bed of a fixed bed
hydrogenation catalyst and the reactor is operated as a
trickle bed reactor.
6. Process according to one of claims 1 to 5,
characterised in that
the aminizing hydrogenation is carried out at a
temperature in the range from 90 to 130 °C and a
pressure in the range from 5 to 8 MPa.
7. Process according to one of claims 1 to 6,
characterised in that
there is fed to the iminization stage a mixture
consisting mainly of 10 to 40 wt % of isophorone
nitrile, 10 to 40 wt % of ammonia and methanol.



8. Process according to one of claims 1 to 7,
characterised in that
the reaction mixture from the second stage is worked up
by distillation and a high-boiling fraction with a
higher boiling point than isophorone diamine and
containing 3,5,5-trimethyl-6-imino-7-aza-bicyclo-
[3,2,1]-octane (= amidine) is fed to the first stage
together with a solution containing isophorone nitrile,
ammonia and methanol.

THIS SHEET HAS BEEN
REPLACED

Process for the production of 3-aminomethyl-3,5,5-trimethylcyclohexyl amine

5 A b s t r a c t

The invention relates to an improved process for producing 3-aminomethyl-3,5,5-trimethylcyclohexyl amine (= isophorone diamine) from isophorone nitrile.

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It is known, for the production of isophorone diamine, to iminize isophorone nitrile in a first stage and to subject the reaction mixture to aminizing hydrogenation in a second stage in the presence of a fixed bed hydrogenation catalyst
15 based on Raney cobalt.

By the use according to the invention of a fixed bed hydrogenation catalyst produced in a special manner - mixing of a powdery Co-containing Raney alloy with powdery cobalt,
20 sintering of the powdery mixture to mouldings and activation of the latter by leaching out by means of alkali hydroxide solution -, the yield and/or space-time yield in isophorone diamine were able to be increased.

This Sheet Has Been Replaced



PROCESS FOR THE PRODUCTION OF 3-AMINOETHYL-3, 5,
5-TRIMETHYLCYCLOHEXYL AMINE

A process for producing 3-aminomethyl-3, 5, 5-trimethylcyclohexyl amine (isophorone diamine) from isophorone nitrile. Isophorone nitrile is iminated in a first stage and then the reaction mixture is subjected to aminating hydrogenation in a second stage in the presence of a fixed bed hydrogenation catalyst based on Raney cobalt. The fixed bed hydrogenation catalyst is produced in a special manner by mixing a powdery Co-containing Raney alloy with powdery cobalt, sintering the powdery mixture to shaped moldings and then activating by leaching with alkali hydroxide solution. The yield and/or space-time yield in isophorone diamine production can be increased.

