Claims

1. A process for the continuous preparation of toluenediamine by liquid-phase hydrogenation of dinitrotoluene by means of hydrogen in the presence of a suspended, nickel-comprising catalyst in a reactor with a product isolation unit downstream of the reactor to give a product output from the reactor comprising a liquid phase comprising toluenediamine and dinitrotoluene, in which the nickel-comprising catalyst is suspended, wherein the concentration of dinitrotoluene in the liquid phase of the product output from the reactor in the region between the reactor and the downstream product isolation unit is set to a value in the range from 1 to 200 ppm by weight, based on the total weight of the liquid phase of the product output from the reactor, with product formation being reduced and catalyst deactivation being largely avoided.

2. The process according to claim 1, wherein the concentration of dinitrotoluene in the liquid product output from the reactor is set to a value in the range from 1 to 100 ppm by weight, based on the total weight of the liquid product output from the reactor, preferably to a value of from 2 to 50 ppm by weight, based on the total weight of the liquid product output from the reactor, more preferably to a value in the range from 3 to 30 ppm by weight, based on the total weight of the liquid product output from the reactor.

3. The process according to claim 1 or 2, wherein the hydrogenation of dinitrotoluene to toluenediamine is carried out at a temperature in the range from 50 to 250°C.

4. The process according to any of claims 1 to 3, wherein the hydrogenation of dinitrotoluene to toluenediamine is carried out at a pressure in the range from 5 to 50 bar.

5. The process according to any of claims 1 to 4, wherein the liquid phase of the product output comprises aminonitrotoluene and the concentration of aminonitrotoluene in the liquid phase of the product output from the reactor in the region between the reactor and the product isolation unit is set to a value in the range from 0 to 2000 ppm by weight, based on the total weight of the liquid phase of the product output.

6. The process according to any of claims 1 to 5, wherein the reactor is selected from the group consisting of a stirred tank reactor, a loop reactor and a bubble column.

7. The process according to any of claims 1 to 6, wherein the reactor is a jet loop reactor having external and internal circulation.

8. The process according to any of claims 1 to 7, wherein the product isolation unit is selected from the group consisting of a filter, a static decanter and a dynamic decanter.

9. The process according to any of claims 1 to 8, wherein the suspended catalyst comprises at least one metal of transition groups I., II., V., VI. and/or VIII. of the Periodic Table in addition to nickel as catalytically active metal..

10. The process according to any of claims 1 to 9, wherein the catalyst has a nickel content in the range from 0.1 to 99% by weight, based on the total weight of the catalyst.

11. The process according to any of claims 1 to 10, wherein the catalyst comprises at least one oxidic support component.

12. The process according to any of claims 1 to 11, wherein the catalyst comprises platinum and nickel.

13. The process according to any of claims 1 to 11, wherein the catalyst comprises palladium, nickel and iron.

14. The process according to any of claims 1 to 13, wherein the catalyst is used in an amount of from 0.1 to 15% by weight, based on the total weight of the reaction mixture in the reactor.

15. The process according to any of claims 1 to 14, wherein the reaction is carried out in a three-phase mixture composed of hydrogen-comprising gas phase, suspended catalyst and liquid phase comprising from 0 to 40% by volume of an alcohol, from 10 to 60% by volume of water and from 20 to 70% by volume of toluenediamine.

16. The process according to any of claims 1 to 15, wherein the setting of the dinitrotoluene concentration in the liquid product output from the reactor in the

region between the reactor and the downstream product isolation unit is carried out on the basis of a repeated measurement of the dinitrotoluene concentration in the liquid product output from the reactor at time intervals of ≤24 hours.

Claims

1. A process for the continuous preparation of toluenediamine by liquid-phase hydrogenation of dinitrotoluene by means of hydrogen in the presence of a suspended, nickel-comprising catalyst in a reactor with a product isolation unit downstream of the reactor to give a product output from the reactor comprising a liquid phase comprising toluenediamine and dinitrotoluene, in which the nickel-comprising catalyst is suspended, wherein the concentration of dinitrotoluene in the liquid phase of the product output from the reactor in the region between the reactor and the downstream product isolation unit is set to a value in the range from 1 to 200 ppm by weight, based on the total weight of the liquid phase of the product output from the reactor by lowering the reaction temperature, the hydrogen partial pressure, the residence time, the catalyst concentration or catalyst activity or by increasing amount of dinitrotoluene fed in to obtain the lowermost value within the range (while the corresponding opposite measures are used to obtain the uppermost value within the range), with by-product formation being reduced and catalyst deactivation being largely avoided.

2. The process according to claim 1, wherein the concentration of dinitrotoluene in the liquid product output from the reactor is set to a value in the range from 1 to 100 ppm by weight, based on the total weight of the liquid product output from the reactor, preferably to a value of from 2 to 50 ppm by weight, based on the total weight of the liquid product output from the reactor, more preferably to a value in the range from 3 to 30 ppm by weight, based on the total weight of the liquid product output from the reactor.

3. The process according to claim 1 or 2, wherein the hydrogenation of dinitrotoluene to toluenediamine is carried out at a temperature in the range from 50 to 250°C.

4. The process according to any of claims 1 to 3, wherein the hydrogenation of dinitrotoluene to toluenediamine is carried out at a pressure in the range from 5 to 50 bar.

5. The process according to any of claims 1 to 4, wherein the liquid phase of the product output comprises aminonitrotoluene and the concentration of aminonitrotoluene in the liquid phase of the product output from the reactor in the region between the reactor and the product isolation unit is set to a value in the range from 0 to 2000 ppm by weight, based on the total weight of the liquid phase of the product output.

6. The process according to any of claims 1 to 5, wherein the reactor is selected from the group consisting of a stirred tank reactor, a loop reactor and a bubble column.

7. The process according to any of claims 1 to 6, wherein the reactor is a jet loop reactor having external and internal circulation.

8. The process according to any of claims 1 to 7, wherein the product isolation unit is selected from the group consisting of a filter, a static decanter and a dynamic decanter.

9. The process according to any of claims 1 to 8, wherein the suspended catalyst comprises at least one metal of transition groups I., II., V., VI. and/or VIII. of the Periodic Table in addition to nickel as catalytically active metal..

10. The process according to any of claims 1 to 9, wherein the catalyst has a nickel content in the range from 0.1 to 99% by weight, based on the total weight of the catalyst.

11. The process according to any of claims 1 to 10, wherein the catalyst comprises at least one oxidic support component.

12. The process according to any of claims 1 to 11, wherein the catalyst comprises platinum and nickel.

13. The process according to any of claims 1 to 11, wherein the catalyst comprises palladium, nickel and iron.

14. The process according to any of claims 1 to 13, wherein the catalyst is used in an amount of from 0.1 to 15% by weight, based on the total weight of the reaction mixture in the reactor.

15. The process according to any of claims 1 to 14, wherein the reaction is carried out in a three-phase mixture composed of hydrogen-comprising gas phase, suspended catalyst and liquid phase comprising from 0 to 40% by volume of an alcohol, from 10 to 60% by volume of water and from 20 to 70% by volume of toluenediamine.

16. The process according to any of claims 1 to 15, wherein the setting of the dinitrotoluene concentration in the liquid product output from the reactor in the region between the reactor and the downstream product isolation unit is carried

out on the basis of a repeated measurement of the dinitrotoluene concentration in the liquid product output from the reactor at time intervals of ≤24 hours.