Although complete removal of the deactivated catalyst is technically possible, the known processes for removing the salt fraction by extraction are for their part likewise problematic since they require the use of a large amount of auxiliaries, which leads to a large amount of problematic waste waters. Thus, although the repeated, sequential admixing of the crude product with a base, followed in each case by a phase separation (crossflow extraction; see e.g. K. Sattler, *Thermische Trennverfahren* [Thermal Separation Processes], 3rd Edition, **2001**, Wiley-VCH Weinheim, p. 542ff.), leads to a complete removal of salt, this is accomplished only using a large amount of base and its addition in solid form.

The alternative option of a countercurrent extraction (single addition of fresh extractant in the last extraction step and recycling of the extract to the preceding extraction step) is described in JP2003-206277. JP2003-206277 describes a two-stage countercurrent extraction, although it can also be operated with a larger number of stages. However, the process described in JP2003-206277 also has the disadvantages specified above. Thus, adequate removal of salt is achieved only by using a large amount of base, which leads to the formation of a large waste stream.

The object of the ~~present invention~~embodiments disclosed herein is therefore to provide an improved process for the industrial preparation of triacetone amine. This process should overcome the aforementioned problems. In particular, it should thus reduce the amount of waste materials produced. It should proceed using a cost-effective and nontoxic catalyst which can also be used efficiently in a continuous process procedure. Finally, the improved process should also permit a simplified and efficient work-up of the triacetone amine.

Short description ~~of the invention~~

Surprisingly, it has now been found that the aforementioned object is achieved by a process according to the present ~~invention~~disclosure. Accordingly, a first subject matter of the present ~~invention~~disclosure is

1. a process for the preparation of triacetone amine, comprising the reaction steps

   a) reaction of an acetone equivalent with a first base, selected from ammonia, in the presence of a catalyst, giving a product mixture which comprises acetone;
   b) deactivation of the product mixture from step a) by adding a further base, giving a triacetone amine crude product which comprises acetone;
   c) removal of acetone from the triacetone amine crude product from step b), giving an acetone-depleted triacetone amine crude product;
   d) extraction of the acetone-depleted triacetone amine crude product from step c) by adding an auxiliary, giving an extracted triacetone amine crude product.

In a first aspect, there is provided a process for the preparation of triacetone amine, comprising the reaction steps

a) reaction of an acetone equivalent with a first base, selected from ammonia, in the presence of a catalyst, giving a product mixture which comprises acetone;
b) stopping the catalytic activity of the catalyst used in step a) of the product mixture by adding a further base, wherein the further base is a base different from ammonia and has a lower pK_B value than ammonia, giving a triacetone amine crude product which comprises acetone;
c) removal of acetone from the triacetone amine crude product from step b), giving an acetone-depleted triacetone amine crude product;
d) extraction of the acetone-depleted triacetone amine crude product from step c) by adding an auxiliary, characterized in that a base is used as auxiliary in step d), thus desalting the solution obtained after step c) of the process, giving an extracted triacetone amine crude product.

2. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to subject matter 1~~ is characterized in that in a further step e), the extracted triacetone amine crude product from step d) is purified to give triacetone amine.

The process according to the ~~invention~~disclosure has the advantage that the catalyst used in step a) and deactivated in step b) is removed prior to the purification of the extracted triacetone amine crude product in step e), which can take place for example by distillation. This prevents an accumulation of the deactivated catalyst in the distillation bottom. In this connection, it permits the process as defined above~~according to the invention~~ to reduce the fraction of deactivated catalyst to below the limit of detection, and in so doing to use a considerably smaller amount of auxiliaries compared to the prior art, as a result of which the amount of waste streams produced is also kept low.

3. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 2~~ is characterized in that the acetone equivalent is one or more of the compounds selected from the group consisting of acetone, mesityl oxide, diacetone alcohol, phorone, diacetone amine, acetonin.

4. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 3~~ is characterized in that the acetone equivalent is acetone.

5. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 4~~ is characterized in that the removal of acetone from the triacetone amine crude product in step c) takes place by distillation.

6. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 5~~ is characterized in that the further base in step b) is selected form the group consisting of an alkali metal hydroxide, an alkaline earth metal hydroxide, an alkali metal carbonate, an alkaline earth metal carbonate.

7. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 6~~ is characterized in that sodium hydroxide or potassium hydroxide is used as further base in step b).

8. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 7~~ is characterized in that sodium hydroxide is used as further base in step b).

9. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 8~~ is characterized in that an aqueous solution of sodium hydroxide is used as further base in step b).

10. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 9~~ is characterized in that an aqueous solution of sodium hydroxide, comprising 40-60% (mass percent) NaOH, is used as further base in step b).

11. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 10~~ is characterized in that an aqueous solution of sodium hydroxide, comprising 45-55% (mass percent) NaOH, is used as further base in step b).

12. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 7~~ is characterized in that potassium hydroxide is used as further base in step b).

13. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 7 and 12~~ is characterized in that an aqueous solution of potassium hydroxide is used as further base in step b).

14. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 7 and 12 to 13~~ is characterized in that an aqueous solution of potassium hydroxide, comprising 40-60% (mass percent) KOH, is used as further base in step b).

15. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 7 and 10 to 14~~ is characterized in that an aqueous solution of potassium hydroxide, comprising 45-55% (mass percent) KOH, is used as further base in step b).

16. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 15~~ is characterized in that the amount of further base added in step b) is in the range from 1.0 to 2.0 molar equivalents, based on the amount of catalyst used in step a).

17. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 16~~ is characterized in that the amount of further base added in step b) is in the range from 1.0 to 1.4 molar equivalents, based on the amount of catalyst used in step a).

18. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 17~~ is characterized in that the amount of further base added in step b) is 1.2 molar equivalents, based on the amount of catalyst used in step a).

19. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 18~~ is characterized in that a base is used as auxiliary in step d).

20. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 19~~ is characterized in that a base selected from the group consisting of an alkali metal hydroxide, an alkaline earth metal hydroxide, an alkali metal carbonate, an alkaline earth metal carbonate is used as auxiliary in step d).

21. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 20~~ is characterized in that a base selected from the group consisting of sodium hydroxide or potassium hydroxide is used as auxiliary in step d).

22. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 21~~ is characterized in that a base, namely sodium hydroxide, is used as auxiliary in step d).

23. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 22~~ is characterized in that a base, namely an aqueous solution of sodium hydroxide, is used as auxiliary in step d).

24. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 23~~ is characterized in that a base, namely an aqueous solution of sodium hydroxide, comprising 40-60% (mass percent) NaOH, is used as auxiliary in step d).

25. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 24~~ is characterized in that a base, namely an aqueous solution of sodium hydroxide, comprising 45-55% (mass percent) NaOH, is used as auxiliary in step d).

26. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 21~~ is characterized in that a base, namely potassium hydroxide, is used as auxiliary in step d).

27. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 21 and 26~~ is characterized in that a base, namely an aqueous solution of potassium hydroxide, is used as auxiliary in step d).

28. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 21 and 26 to 27~~ is characterized in that a base, namely an aqueous solution of potassium hydroxide, comprising 40-60% (mass percent) of KOH, is used as auxiliary in step d).

29. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 21 and 26 to 28~~ is characterized in that an aqueous solution of potassium hydroxide, comprising 45-55% (mass percent) KOH, is used as base in step d).

30. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 29~~ is characterized in that the amount of base added in step d) as auxiliary is in the range from 1.0 to 6.0 molar equivalents, based on the amount of catalyst used in step a).

31. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 30~~ is characterized in that the amount of base added as auxiliary in step d) is in the range from 1.0 to 4.0 molar equivalents, based on the amount of catalyst used in step a).

32. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 31~~ is characterized in that the amount of base added as auxiliary in step d) is in the range from 1.0 to 2.0 molar equivalents, based on the amount of catalyst used in step a).

33. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 32~~ is characterized in that the amount of base added as auxiliary in

step d) is in the range from 1.0 to 1.4 molar equivalents, based on the amount of catalyst used in step a).

34. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 33~~ is characterized in that the amount of base added as auxiliary in step d) is 1.2 molar equivalents, based on the amount of catalyst used in step a).

35. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 34~~ is characterized in that a phase separation additionally takes place between step b) and step c).

36. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 35~~ is characterized in that a phase separation additionally takes place between step b) and step c), and the organic phase of the triacetone amine crude product is used in steps c) to d).

37. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 36~~ is characterized in that the acetone equivalent used in step a) is acetone and the molar ratio of acetone to ammonia used in the reaction in step a) is 3 : 1 to 20 : 1.

38. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 37~~ is characterized in that the acetone equivalent used in step a) is acetone and the molar ratio of acetone to ammonia used in the reaction in step a) is 6 : 1 to 9 : 1.

39. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 38~~ is characterized in that the reaction in step a) is carried out at temperatures in the range from 20-80°C.

40. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 39~~ is characterized in that the reaction in step a) is carried out at temperatures in the range from 40-80°C.

41. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 40~~ is characterized in that the reaction in step a) is carried out at temperatures in the range from 60-70°C.

42. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 41~~ is characterized in that the reaction in step a) is carried out at a pressure in the range from 1 to 16 bar.

43. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 42~~ is characterized in that the reaction in step a) is carried out at a pressure in the range from 1 to 10 bar.

44. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 43~~ is characterized in that the reaction in step a) is carried out at a pressure in the range from 1 to 6 bar.

45. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 44~~ is characterized in that ammonia is added in step a) in gaseous form or as a liquid.

46. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 2 to 45~~ is characterized in that, in a further step e), the extracted triacetone amine crude product from step d) is purified by distillation to give triacetone amine.

47. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 46~~ is characterized in that the catalyst in step a) is selected from the group consisting of Brönsted acids, ammonium salts of a Brönsted acid, phosphonium salts of a Brönsted acid, Lewis acids.

48. In a further embodiment of the ~~invention~~disclosure, the process as defined ~~above~~~~according to one or more of subject matters 1 to 47~~ is characterized in that the catalyst in step a) is a Brönsted acid.

49. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 48~~ is characterized in that the catalyst in step a) is selected from the group consisting of hydrochloric acid, sulphuric acid, nitric acid, organic acids of the formula RCOOH, sulphonic acids of the formula RSO_3H, where R is selected from the group consisting of saturated, unsaturated, branched, unbranched, ring-closed, open-chain aliphatic, aromatic, substituted, unsubstituted hydrocarbon radicals.

50. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47~~ is characterized in that the catalyst in step a) is a phosphonium salt of a Brönsted acid.

51. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 50~~ is characterized in that the catalyst in step a) is a phosphonium salt of a Brönsted acid, where the Brönsted acid is selected from the group consisting of hydrochloric acid, sulphuric acid, nitric acid, of an organic acid of the formula RCOOH, sulphonic acids of the formula RSO_3H, where R is selected from the group consisting of saturated, unsaturated, branched, unbranched, ring-closed, open-chain aliphatic, aromatic, substituted, unsubstituted hydrocarbon radicals.

52. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47~~ is characterized in that the catalyst in step a) is an ammonium salt of a Brönsted acid.

53. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 52~~ is characterized in that the catalyst in step a) is an ammonium salt of a Brönsted acid, where the Brönsted acid is selected from the group consisting of hydrochloric acid, sulphuric acid, nitric acid, an organic acid of the formula RCOOH, sulphonic acids of the formula RSO_3H, where R is selected from the group consisting of saturated, unsaturated, branched, unbranched, ring-closed, open-chain aliphatic, aromatic, substituted, unsubstituted hydrocarbon radicals.

54. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 52 to 53~~ is characterized in that the catalyst in step a) is ammonium chloride or ammonium nitrate.

55. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 52 to 54~~ is characterized in that the catalyst in step a) is ammonium nitrate.

56. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47~~ is characterized in that the catalyst in step a) is a Lewis acid.

57. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 56~~ is characterized in that the catalyst in step a) is a Lewis acid, where the Lewis acid is selected from compounds of the elements of the fourth group of the Periodic Table of the Elements, compounds of elements of the thirteenth group of the Periodic Table of the Elements, salts of alkali metals, salts of alkaline earth metals.

58. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 56 to 57~~ is characterized in that the catalyst in step a) is a Lewis acid, where the Lewis acid is selected from the group consisting of halides, alkoxides, alkyl compounds of the elements of the fourth and thirteenth groups of the Periodic Table of the Elements.

59. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 56 to 58~~ is characterized in that the catalyst in step a) is a Lewis acid, where the Lewis acid is selected from the group consisting of $AlCl_3$, BF_3, $TiCl_4$, $Al(OR)_3$, $B(OR)_3$, $Ti(OR)_4$, AlR_3, where R is selected from the group consisting of saturated, unsaturated, branched, unbranched, ring-closed, open-chain aliphatic, aromatic, substituted, unsubstituted hydrocarbon radicals.

60. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 56 to 57~~ is characterized in that the catalyst in step a) is a

Lewis acid, where the Lewis acid is selected from the group consisting of salts of alkali metals, salts of alkaline earth metals.

61. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 47 and 56 to 57 and 60~~ is characterized in that the catalyst in step a) is a Lewis acid, where the Lewis acid is selected from the group consisting of $CaCl_2$, $MgCl_2$, LiCl.

62. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 61~~ is characterized in that the molar ratio of ammonia to catalyst in step a) is in the range 1 : 0.8 to 1 : 0.02.

63. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 62~~ is characterized in that the molar ratio of ammonia to catalyst in step a) is in the range 1 : 0.2 to 1 : 0.05.

64. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 63~~ is characterized in that in step d) a base is used as auxiliary and the base used in step d) and the base used in step b) are chemically identical.

65. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 64~~ is characterized in that the solvent in step a) is selected from the group consisting of aliphatic solvents, preferably pentane, hexane, heptane, octane, decane, cyclohexane, tetramethylsilane; aromatic solvents, preferably benzene, toluene, xylene; ether compounds, preferably diethyl ether, dipropyl ether, dibutyl ether, methyl tert-butyl ether; halogenated solvents, preferably dichloromethane, chloroform, tetrachloromethane; alcohols, preferably methanol, ethanol, propanol, isopropanol, butanol, tert-butanol; esters, preferably methyl acetate, ethyl acetate, propyl acetate, butyl acetate; acetone.

66. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 65~~ is characterized in that the solvent in step a) is acetone.

67. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 66~~ is characterized in that the extraction in step d) takes place at a temperature of 20-80°C.

68. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 67~~ is characterized in that the extraction in step d) takes place at a temperature of at least 20°C, preferably at 20-99°C, particularly preferably at 45-90°C, very particularly preferably at 45-80°C, most preferably at a temperature of 45 to 65°C, and ideally at a temperature of 60°C.

69. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of the subjects matters 1 to 68~~ is characterized in that the acetone-depleted triacetone amine crude product obtained in step c) has an acetone content of less than 40% (mass percent), in particular of less than 20% (mass percent), preferably of less than 15% (mass percent), particularly preferably of less than 10% (mass percent), very particularly preferably of less than 2.5% (mass percent), most preferably of less than 1.5% (mass percent), ideally of less than 1% (mass percent).

70. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 69~~ is characterized in that the extraction of the acetone-depleted triacetone amine crude product in step d) is carried out as crossflow extraction.

71. In a further embodiment of the ~~invention~~disclosure, the process as defined above~~according to one or more of subject matters 1 to 70~~ is characterized in that the extraction of the acetone-depleted triacetone amine crude product in step d) is carried out as countercurrent extraction.

Detailed description of the invention

The process according to the invention is suitable for producing triacetone amine, but also for producing extracted triacetone amine crude product. In the first step [step a)] of the process according to the invention, the reaction of acetone with a first base, selected from ammonia, takes place in the presence of a catalyst, giving a product mixture which comprises acetone.