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METHOD FOR PURIFYING LACTAMS

The present invention relates to a process for the purification of lactams, more particularly of lactams obtained by the cyclizing hydrolysis of
5 aminonitrile.

The invention relates more particularly to the purification of ϵ -caprolactam obtained by the cyclizing hydrolysis of aminocapronitrile.

Several processes for the synthesis of
10 ϵ -caprolactam, a major chemical intermediate used in particular in the production of polyamides and more particularly of polyamide-6, have been provided. One of the most widely used processes industrially consists of a Beckmann rearrangement of cyclohexanone oxime in the
15 presence of sulfuric acid or of oleum.

Another process which forms the subject-matter of numerous studies consists in obtaining ϵ -caprolactam by the cyclizing hydrolysis of aminocapronitrile. This reaction, disclosed in French
20 Patent 2 029 540, can be carried out in the liquid phase in the presence of solvents and of catalysts or, as disclosed in Patent US 2 357 484, in the gas phase with alumina as catalyst.

Patent EP 150 295 also relates to a process
25 for the cyclizing hydrolysis of aminocapronitrile in the gas phase in the presence of a catalyst based on copper and on vanadium.

The ϵ -caprolactam obtained by these synthetic processes has to be purified in order to obtain a chemical intermediate or a monomer which makes it possible in particular to manufacture polymers
5 exhibiting characteristics satisfactory for the manufacture of finished products, such as yarns, fibers, and the like.

Numerous purification processes have been provided for the treatment of the caprolactam obtained
10 by the Beckmann rearrangement of cyclohexanone oxime.

Processes for the purification of the caprolactam obtained by the cyclizing hydrolysis of aminocapronitrile have also been provided. Thus, Patent US 5 496 941 discloses a process which consists in
15 separating the products with low boiling points and the products with high boiling points from the hydrolysis reaction medium. This separation is obtained in particular by distillation of the caprolactam.

The caprolactam thus recovered is subjected
20 to a hydrogenation, then either to a treatment through ion-exchange resins or to a distillation in the presence of sulfuric acid, and, finally, to a distillation in the presence of a base.

Other purification processes have been
25 provided. Thus, Patent WO 98/05636 provides a process for the purification by liquid/liquid extraction using

a solvent with an acidic nature or by a treatment through resins.

In these processes, a final distillation of the caprolactam is generally provided in order to
5 obtain a product which satisfies the criteria of purity required, in particular for its use as monomer in the manufacture of polyamides, such as polyamide-6.

However, this final distillation results in a high loss of caprolactam, which is very harmful to the
10 general economics of the process.

Thus, in the case of the purification of a caprolactam obtained by the Beckmann rearrangement reaction, Patent US 4 301 073 provides a process for purifying the caprolactam which comprises a stage of
15 extraction by a solvent and then a distillation in a basic medium. The distillation residue is treated to recover as much as possible of the caprolactam. This treatment consists of further distillations and a treatment with a strong acid of the caprolactam thus
20 distilled before it is recycled in the extraction stage. This process makes it possible to improve the degree of recovery of the caprolactam but requires the use of an acid for the treatment before recycling. This recycling process is complex and requires a significant
25 investment in order to carry out the acid treatment. Furthermore, this process cannot be transposed to the treatment of a caprolactam synthesized by a novel

reaction, which can generate entirely different impurities requiring novel purification processes.

One of the aims of the present invention is to overcome these disadvantages by providing a process
5 and a plant for the purification of a lactam resulting from the cyclizing hydrolysis of an aminonitrile which make it possible to obtain a high degree of recovery of lactam of high purity.

To this end, an aspect of the invention provides
10 a process for the purification of lactams, obtained by the cyclizing hydrolysis of an aminonitrile consisting in removing, from the hydrolysis reaction medium, the ammonia and optionally the solvent, in then recovering the lactam from the reaction medium by employing at least one
15 distillation of the said lactam in the presence of a base and in recovering, by this distillation, optionally a top fraction comprising compounds which are more volatile than the lactam, a fraction comprising the lactam with high purity and a distillation bottom product comprising lactam
20 and compounds with a higher boiling point than the lactam, characterized in that the process comprises a stage of treatment of said distillation bottom product which consists in recovering most of the lactam and in recycling said recovered lactam in the process for the purification
25 of the lactam.

Thus, the distillation bottom product or residue recovered in the stage of purification by distillation predominantly may comprise lactam, the impurities being composed mainly of salts and of reaction by-products.

An embodiment may provide recycling the separated lactam from the distillation bottom product in any stage of the purification process or in the medium obtained after the cyclizing hydrolysis.

According to another embodiment, this treatment of the distillation bottom product may consist either of a recovery of the lactam by, for example, thin-layer evaporation of the lactam present in the distillation bottom product and in recycling said separated lactam in any stage of the purification process or in the medium obtained after the cyclizing hydrolysis.

According to a further embodiment, this treatment of the distillation bottom product may consist either of a recovery of the lactam by, for example, thin-layer evaporation of the lactam or crystallization of the latter or of a separation of the lactam from the other impurities, for example by extraction of most of the impurities with water.

The crystallization of the lactam can be carried out according to conventional crystallization processes with, as solvent, either water or an aqueous

solution saturated with lactam or the lactam itself. It is advantageously carried out in a single stage. However, if necessary, two or more crystallization stages can be employed.

5 A crystallization process and the conditions for its implementation are disclosed, for example, in Patents EP 943 608 and US 4 882 430.

 The lactam thus obtained may be recycled in the device for distillation in a basic medium, either by
10 direct feeding to the distillation column at a predetermined level or by addition to the stream of reaction medium entering said column, or at another stage of the process for the purification and recovery of the lactam or even in the medium resulting from the
15 cyclizing hydrolysis.

 According to an embodiment, the distillation bottom product may be treated in a thin-film or thin-layer evaporation process. The vaporized lactam may be recycled in the distillation device under conditions similar to
20 those of the embodiment employing the crystallization. The evaporation of the lactam may be obtained by heating at a temperature between 130°C and 150°C under a pressure of less than 10 mbar.

 Further, an embodiment may provide that the
25 distillation bottom product can be treated with water to dissolve the impurities, in particular the salts, the lactam recovered after separation by settling

advantageously being recycled in the process for the purification of the lactam by distillation in an acidic medium and then in a basic medium

These processes for the treatment of the distillation bottom product may make it possible to
5 recover at least 90% by weight, advantageously at least 95%, of the lactam present in the said bottom product. The losses of lactam in the purification process may thus be restricted to less than 3% by weight of the participating lactam.

10 The process according to one embodiment may thus make it possible to restrict the losses of lactam to a very low level during the purification by basic distillation. These losses may have only a very slight effect on the economics of the process and in particular
15 the quality of the lactam recovered is of a very high level.

According to another embodiment, the medium may comprise the lactam to be recovered which can be the reaction medium for the cyclizing hydrolysis of
20 aminonitrile after evaporation of the ammonia. Said reaction medium could be subjected to a hydrogenation before the evaporation of the ammonia.

This reaction medium, participating in the basic distillation, may have been subjected to several
25 preliminary treatments after evaporation of the ammonia.

In a further embodiment, the hydrolysis reaction medium may be treated to remove the products which are respectively less volatile and more volatile than the lactam. This treatment, disclosed in Patent US 5 496 941, consists in evaporating the ammonia, the water and the other volatile products and in then distilling the lactam. The hydrolysis reaction medium treated by the basic distillation may thus be the lactam recovered by distillation.

In another embodiment, in particular when the cyclizing hydrolysis is carried out in the vapor phase, only the ammonia may be extracted from the reaction medium, which may or may not have been subjected to a hydrogenation prior to the removal of the ammonia.

The lactam recovered by distillation or a ammonia-free reaction medium can be subjected to a hydrogenation or an oxidizing treatment, for example, before being fed to the basic distillation.

In another embodiment, the process for the purification of the lactam can comprise a treatment through an ion-exchange resin in an acidic medium or a distillation in the presence of a strong acid, for example sulfuric acid.

The process for the purification and recovery of the lactam can also comprise one or more successive crystallizations of the lactam, the lactam thus

crystallized forming the medium fed to the basic distillation in accordance with the invention.

Consequently, the distillation process in accordance to one embodiment may be a stage in a lactam purification process which can be combined with numerous other known treatments of lactams used in the numerous disclosed purification processes.

According to another embodiment, the purification process can be employed in a distillation process comprising several distillation columns mounted in series in order to carry out the successive separation of the various products.

These plants for the implementation of the purification process by distillation in a basic medium or an acidic medium and then a basic medium are also subject-matters of the present invention.

Accordingly, another aspect of the invention provides a plant for the implementation of a process for the purification of lactams by distillation of said lactam, characterized in that it comprises three distillation columns mounted in series, the first (2,22) being a dehydration column, the second column (6,26) and the third column (11,31) making it possible to separate either the compounds which are less volatile than the lactam or the compounds which are more volatile, the purified lactam being withdrawn from the third column (11, 31), and in that a basic compound is added to the lactam before feeding to at least one of the columns.

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In a first embodiment which may be suitable for carrying out the treatment in a basic medium of the lactam to be purified, the plant may comprise a first distillation column to which the lactam to be purified is fed with a predetermined amount of base. In this column, known as the dehydration column, the water may be removed in the form of a top fraction, the distillation bottom product, comprising the lactam and the base, being fed to a second distillation column.

In this second distillation column, the products which are more volatile than the lactam may be removed in the form of a top fraction and the separated lactam may be fed to a third distillation column.

5 In this third column, the lactam may be distilled and recovered as a top fraction, and the distillation bottom product can advantageously be treated according to the processes of the embodiments in order to recover most of the lactam. The lactam recovered from the
10 distillation bottom product may be advantageously recycled in the second distillation column.

According to a second embodiment, the plant may also comprise three distillation columns mounted in series. The first column is, as in the first embodiment, a
15 dehydration column which may make it possible to remove most, preferably all, of the water.

In the second column, the least volatile compounds, and in particular less volatile than the
20 lactam, may be separated and may constitute a distillation bottom product, and the lactam recovered at the column top may be fed to the third column.

In this final column, the purified lactam may be collected in the form, advantageously, of an intermediate
25 fraction by withdrawal, either in the vapor phase or in liquid phase. The top fraction, composed of the compounds which are more volatile than

the lactam, may be removed and the distillation bottom product may preferably be recycled in the second column.

In this embodiment, the distillation bottom
5 product from the second column may advantageously be treated in order to recover the lactam. The lactam thus recovered may advantageously be recycled in the second column.

Preferably, the basic compound may be added to
10 the medium to be treated before it is fed to the first dehydration stage.

However, another embodiment may relate to a process which consists in treating the medium comprising the lactam to be purified by distillation in an acidic
15 medium and more particularly in sulfuric acid medium, followed by distillation in a basic medium. Such a treatment may be employed in a plant in accordance with the second embodiment described above.

In this second embodiment, the acid may be added
20 to the medium to be treated before it is fed to the dehydration column, the base being added to the top fraction produced in the second distillation column before it is fed to the third distillation column.

The salts generated by the presence of the
25 acid, for example amine sulfates or sulfates originating from the neutralization of the base may be removed and bled off in the distillation bottom product

produced in the second distillation column. This distillation bottom product may advantageously be subjected to a lactam recovery treatment which, in the present case, can be advantageously be:

- 5 - either a washing of the distillation bottom product with water to dissolve the salts, such as ammonium and/or amine sulfates, after separation of the aqueous phase and the organic phase composed of the lactam, the latter organic phase preferably being
10 recycled in the medium fed to the dehydration column or recycled in the second column, or alternatively subjected to another treatment, such as thin-layer evaporation or crystallization,
- or an evaporation of the lactam carried out
15 in a thin-film or thin-layer evaporator,
- or a crystallization of the lactam from water, an aqueous lactam solution or lactam.

These treatments can be combined, in particular the washing with water can be carried out
20 prior to the evaporation or the crystallization.

According to a characteristic of the embodiments, the lactam may be advantageously ϵ -caprolactam obtained by the cyclizing hydrolysis of aminocapronitrile either in the vapor phase or in the
25 liquid phase, with or without solvent.

The aminonitrile may, for example, be produced by partial hydrogenation of adiponitrile. This reaction is

disclosed in particular in Patents US 4 601 859; US 2 762 835, US 2 208 598, DE 4 235 466 and US 5 981 790.

The ammonia present in the cyclizing hydrolysis reaction medium may be removed by distillation or
5 flashing.

The reaction medium fed in the process of the invention can comprise at least 99.9% by weight of lactam. This concentration range is given only by way of indication. Thus, media comprising less lactam can
10 also be purified by the process of the invention.

By way of nonlimiting indication, the operating conditions of the various columns of the second embodiment of the plant are given below.

The first dehydration stage may be carried out
15 at a temperature of between 100°C and 130°C approximately, at a pressure of between 50 mbar and 200 mbar approximately.

The dehydrated stage may be fed to a second distillation column in which the second stage of
20 separation of the products with high boiling point may be carried out by production of a distillation bottom product.

The operating conditions of this column may be a column bottom temperature of less than 150°C and a
25 pressure of between 5 and 10 mbar.

The top fraction thus produced can comprise less than 100 ppm of impurities with a higher boiling point than the lactam.

This top fraction can be fed to a further distillation column, the operating conditions of which may be a column bottom temperature of less than 150°C and a pressure of between 5 and 10 mbar.

The lactam, in particular ϵ -caprolactam, produced as intermediate fraction may exhibit a degree of purity which satisfies the criteria required for polyamide manufacture, in particular for textile applications. Preferably, this intermediate fraction may be withdrawn in the vapor phase from the column in order to obtain a very low concentration of products with a high boiling point. However, it may also be possible to withdraw this intermediate fraction in the liquid phase, without departing from the scope of the invention.

The distillation bottom product obtained in the column bottom may be recycled in the second distillation column.

In a preferred embodiment in accordance with the process for the purification of the lactam, the distillation bottom product collected in the second column bottom may be fed to a stage for separation of the lactam, for example a thin-film or thin-layer evaporation process at a

temperature of less than 150°C and under a pressure of between 5 and 10 mbar.

In another preferred embodiment, this distillation bottom product may be subjected to a crystallization process in order to recover the lactam, more particularly caprolactam.

It may also be possible to treat this distillation bottom product with water in order to extract the salts, more particular the sulfates, originating from the neutralization of the free bases present in the hydrolysis medium. The organic phase recovered, comprising the lactum, may either be recycled in the dehydration column or the second column, or subjected to a further stage of separation of the lactam, such as a thin-film or thin layer of evaporation or a crystallization.

According to an embodiment, a base chosen from the group consisting of alkali metal hydroxides, alkaline earth metal hydroxides and metal carbonates, for example sodium hydroxide, lithium hydroxide, potassium hydroxide, magnesium hydroxide, calcium hydroxide, sodium carbonate, magnesium carbonate, lithium carbonate or mixed carbonate, can be used as basic compound. Preferably, an aqueous sodium hydroxide solution may be used. The amount of base added can vary within wide limits. Advantageously, it is between 0.05 and 2 g of base per 1 kg of caprolactam or lactam.

As indicated above, this base can be added either to the medium to be treated fed to the dehydration stage or to the top fraction resulting from the second column of the second embodiment of the plant, before it is fed to the third column.

5

It may also be possible to add an acid, preferably sulfuric acid to the medium fed to the dehydration stage. The amount of acid added may be determined as a function of the amount of free bases present in the medium to be treated. This amount of free bases may be determined by potentiometric titration of the medium. The amount of acid added can be between 0.5 mol of acid per mole of free bases and one mole of acid per mole of free bases, preferably between 0.7 and 0.9 mol of acid per mole of free bases. This concentration range may make it possible to obtain meltable salts and a weak corrosive action of the medium.

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Other advantages and details of the invention will become more clearly apparent in the light of the detailed description of examples, made with reference to the appended figures, in which:

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- Figure 1 represents a block diagram of a first embodiment of the plant of the invention, and

- Figure 2 represents a block diagram of a second embodiment of the plant of the invention.

Example 1:

A medium resulting from the cyclizing hydrolysis of an aminocapronitrile obtained by hemihydrogenation of adiponitrile was treated by hydrogenation before distillation (stripping) of the ammonia. The resulting medium was treated by passing through an ion-exchange resin. The conditions for carrying out these treatments are disclosed in the abovementioned patents or patent applications and in particular in Patent Applications WO 98/05636 and FR 2 786 180.

This medium comprises approximately 65% by weight of caprolactam and exhibits the following purity characteristics

Permanganate number (PN_{MnO_4}): 35

Content of volatile bases (N_{VB}): 1.8 meq/kg

UV number (N_{UV}): 0.5

These characteristics are determined according to standardized procedures disclosed in Standard ISO 8660 for the permanganate number (PN_{MnO_4}), Standard ISO 8661 for the content of volatile bases (N_{VB}) and Standard ISO 7059 for the UV number (N_{UV}). With reference to Figure 1, this medium is, in accordance with the invention, fed via the pipe 1 to the first distillation column 2, after addition, via the pipe 3, of 0.6 g of pure sodium hydroxide, in the form of a concentrated solution, per kg of caprolactam.

The column 2 has a bottom temperature of less than 130°C and a pressure of 70 mbar.

The top fraction 4 is composed of water.

The distillation bottom product 5 comprises
5 an amount of water of less than 0.5% by weight.

This distillation bottom product 5 is fed to a second column 6 operating under reflux 7, 8.

The top fraction 9, composed essentially of caprolactam and of compounds with a lower boiling point
10 than that of caprolactam, is removed. The amount of caprolactam removed represents 0.5% by weight of the caprolactam fed in. The distillation bottom product 10, comprising the caprolactam, is fed to a third column 11 also operating under reflux 12, 13.

15 The top fraction 14 is composed of caprolactam of conformable purity.

The purity characteristics are:

Permanganate number (PN_{MnO_4}): 1.5

Content of volatile bases (N_{VB}): 0.28 meq/kg

20 UV number (N_{UV}): 0.025

The distillation bottom product 15, composed predominantly of caprolactam and comprising, as impurities, compounds with a higher boiling point than that of caprolactam, is fed to a scraped-film
25 evaporator 16 operating under a pressure of 10 mbar and a temperature of 150°C.

The evaporated caprolactam is recycled via the pipe 17 to the third column 11.

The non-evaporated products are removed via the pipe 18.

5 The balance by weight of the caprolactam fed to the first column 2 and that produced at the top 14 of the third column 11 shows that 99.1% of the participating caprolactam is recovered with a high and suitable degree of purity.

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Example 2

An aminocapronitrile hydrolysis medium after distillation (stripping) of the ammonia is treated in a plant in accordance with the invention, represented diagrammatically by Figure 2, according to a second
15 embodiment of the process for the purification of caprolactam.

The characteristics of this medium are as follows:

Permanganate number (PN_{MnO_4}): 381

20

Content of volatile bases (N_{VB}): 232 meq/kg

UV number (N_{UV}): 12

Furthermore, the concentration of free bases, determined by potentiometric titration, is equal to 189 meq per kg of medium or solution.

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With reference to Figure 2, this medium is, in accordance with the invention, fed via the pipe 21 to the first distillation column 22, after addition,

via the pipe 23, of 0.8 mol of sulfuric acid, in the form of a concentrated solution, per mole of free bases present in the medium to be treated.

The column 22 has a bottom temperature of
5 less than 130°C and a pressure of 70 mbar.

The top fraction 24 is composed of water.

The distillation bottom product 25 comprises an amount of water of less than 0.5% by weight.

This distillation bottom product 25 is fed to
10 a second column 26 operating under reflux 27, 28.

The top fraction 29 resulting from this second column 26, composed essentially of caprolactam and compounds with a lower boiling point than that of caprolactam, is fed to a third column 31 also operating
15 under reflux 32, 33, after addition, via the pipe 39, to this top fraction of 0.6 g of pure sodium hydroxide, in the form of a concentrated solution, per kg of caprolactam.

The top fraction 34 from the third column 31,
20 composed of compounds with a lower boiling point than that of caprolactam, is removed.

The distillation bottom product 35 is recycled, in the embodiment illustrated, in the second column 26.

25 The purified caprolactam is drawn off from this third column 31 in the form of an intermediate fraction 40. This withdrawal operation is carried out

either in the gaseous form at the lower part of the column or in the liquid form in the upper part of the column.

The characteristics of the caprolactam
5 produced are:

Permanganate number (PN_{MnO_4}): 2.

Content of volatile bases (N_{VB}): 0.25 meq/kg

UV number (N_{UV}): 0.03

According to the process of the invention,
10 the distillation bottom product 30 from the second column 26, composed predominantly of caprolactam and comprising, as impurities, compounds with a higher boiling point than that of caprolactam and salts formed by the neutralization of the free bases by the sulfuric
15 acid, is fed to a scraped-film evaporator 36 operating under a pressure of 10 mbar and a temperature of 150°C. The evaporated caprolactam is recycled, in the embodiment illustrated, via the pipe 37 in the second column 26.

20 The non-evaporated products are removed via the pipe 38.

The balance by weight of the caprolactam fed to the first column 22 and that produced in the form of an intermediate fraction 40 from the third column 31
25 shows that 98.9% of the participating caprolactam is recovered with a high and suitable degree of purity.

Example 3

Example 2 is repeated, with the difference that the distillation bottom product 30 from the second column is treated by addition of water in order to obtain a solution comprising approximately 80% by weight of caprolactam. The mixture is subjected to stirring. After mixing, two liquid phases are formed. The lower phase is an aqueous phase which comprises 60% by weight of amine sulfates, corresponding to approximately 70% of the amount of amines present in the caprolactam charged to the dehydration column in the purification process. The upper phase, composed essentially of caprolactam, is recycled in the first distillation column.

By this process, 99% of the caprolactam charged in the distillation column is recovered with a high degree of purity, suitable for use of this product in the manufacture of polyamide, in particular for textile applications.

Example 4

Example 2 is repeated, with the difference that the distillation bottom product 30 from the second column is treated by a process of crystallization in one stage of the caprolactam.

The distillation bottom product is concentrated under reduced pressure in order to obtain a concentration by weight of water of approximately 8%.

The concentrated solution is at a temperature of 40°C and is then cooled to a temperature in the region of 20°C with a rate of cooling of approximately 10°C per hour.

5 The crystals formed are recovered by filtration through a sintered glass filter. The aqueous mother liquors recovered are stored for possible recycling.

10 The solid cake of caprolactam crystals is washed with a saturated aqueous solution of caprolactam. The degree of washing as wet base is advantageously between 1 and 5.

The recovered caprolactam has a sufficient purity to be recycled in the distillation process.

15 By this process, 99% of the caprolactam charged in the distillation column is recovered with a high degree of purity, suitable for use of this product in the manufacture of polyamide, in particular for textile applications.

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CLAIMS

1. A process for the purification of lactams obtained by the cyclizing hydrolysis of an aminonitrile, consisting in removing, from the hydrolysis reaction medium, the ammonia and optionally the solvent, in then recovering the lactam from the reaction medium by employing at least one distillation of said lactam in the presence of a base and in recovering, by this distillation, optionally a top fraction comprising compounds which are more volatile than the lactam, a fraction comprising the lactam with high purity and a distillation bottom product comprising lactam and compounds with a higher boiling point than the lactam, characterized in that the process comprises a stage of treatment of said distillation bottom product which consists in recovering most of the lactam and in recycling said recovered lactam in the process for the purification of the lactam.

2. The process as claimed in claim 1, characterized in that the abovementioned treatment of the distillation bottom product is a process for the crystallization of the lactam from a solvent chosen from the group consisting of water, aqueous lactam solutions and lactams.

3. The process as claimed in claim 1, characterized in that the abovementioned treatment of

the distillation bottom product is a process for the thin-layer evaporation of the lactam.

4. The process as claimed in one of claims 1 to 3, characterized in that the abovementioned treatment comprises washing the abovementioned distillation bottom product with water, the lactam being recovered in the organic phase.

5. The process as claimed in one of the preceding claims, characterized in that it comprises a distillation of the lactam in an acidic medium, prior to the distillation in a basic medium.

6. The process as claimed in one of the preceding claims, characterized in that the amount of basic compounds which are added is between 0.05 g and 2 g of basic compounds per 1 kg of lactam.

7. The process as claimed in one of the preceding claims, characterized in that the basic compound is chosen from alkali metal hydroxides.

8. The process as claimed in claim 5, characterized in that the acidic compound is sulfuric acid.

9. The process as claimed in claim 5 or 8, characterized in that the amount of acidic compound added is between 0.5 and 1 mol per mole of free bases present in the lactam to be purified.

10. A plant for the implementation of a process for the purification of lactams by distillation

of said lactam, characterized in that it comprises three distillation columns mounted in series, the first (2, 22) being a dehydration column, the second column (6, 26) and the third column (11, 31) making it possible to separate either the compounds which are less volatile than the lactam or the compounds which are more volatile, the purified lactam being withdrawn from the third column (11, 31), and in that a basic compound is added to the lactam before feeding to at least one of the columns.

11. The plant as claimed in claim 10, characterized in that the basic compound is added to the lactam before feeding to the dehydration column (2, 22).

12. The plant as claimed in claim 11, characterized in that the compounds which are more volatile than the lactam are removed as top fraction (9) from the second column (6).

13. The plant as claimed in claim 12, characterized in that the compounds which are less volatile are separated as distillation bottom product (17) from the third column (11).

14. The plant as claimed in claim 12, characterized in that the distillation bottom product (17) from the third column (11) is subjected to a treatment in order to recover the lactam present and to

recycle this recovered lactam in the process for the purification of the lactam.

15 15. The plant as claimed in claim 10, characterized in that the compounds which are more volatile than the lactam are removed as top fraction from the third column (31) and the compounds which are less volatile than the lactam are removed as distillation bottom product from the second column (26).

10 16. The plant as claimed in claim 15, characterized in that the distillation bottom product from the second column (26) is subjected to a treatment in order to recover the lactam present and to recycle this recovered lactam in the lactam purification
15 process.

17. The plant as claimed in claim 15 or 16, characterized in that the basic compound is added to the lactam before it is fed to the dehydration column (22).

20 18. The plant as claimed in either of claims 15 and 16, characterized in that an acidic compound is added to the lactam before it is fed to the dehydration stage and in that the basic compound is added to the lactam before it is fed to the third column (31).

25 19. The plant as claimed in either of claims 14 and 16, characterized in that the treatment of the distillation bottom products comprising the compounds

which are less volatile than the lactam consists in separating the lactam by a process chosen from the group consisting of a thin-film or thin-layer evaporation, a crystallization and/or a washing with
5 water.

20. The plant as claimed in one of claims 10 to 19, characterized in that the basic compound is chosen from alkali metal hydroxides.

21. The plant as claimed in one of claims 10
10 to 20, characterized in that the amount of basic compounds which are added is between 0.05 g and 2 g of basic compounds per 1 kg of lactam.

22. The plant as claimed in claim 18,
characterized in that the acidic compound is sulfuric
15 acid.

23. The plant as claimed in either of claims 18 and 21, characterized in that the amount of acidic compound added is between 0.5 and 1 mol per mole of free bases present in the lactam to be purified.

20 24. The plant as claimed in one of claims 10 to 23, characterized in that the medium comprising the lactam to be purified is obtained by treatment through an ion-exchange resin of the aminonitrile hydrolysis reaction medium, after removal of ammonia.

25 25. The plant as claimed in one of claims 10 to 24, characterized in that the lactam is caprolactam.

26. The plant as claimed in one of claims 10 to 25, characterized in that the hydrolysis reaction medium is subjected to a hydrogenation, before removal of the ammonia.

5 27. The plant as claimed in one of claims 10 to 25, characterized in that the hydrolysis reaction medium is subjected to a hydrogenation, after removal of the ammonia.

28. The plant as claimed in one of claims 10
10 to 27, characterized in that the cyclizing hydrolysis is carried out in the vapor phase or in the liquid phase.

29. The plant as claimed in one of claims 10 to 27, characterized in that the cyclizing hydrolysis
15 is carried out in the liquid phase, in the presence of a solvent.