

Simultaneous preparation of caprolactam and hexamethylenediamine

Abstract

5

Caprolactam and hexamethylenediamine are prepared simultaneously starting from adiponitrile by a process in which

- 10 (a) adiponitrile is partially hydrogenated to give a mixture containing essentially 6-aminocapronitrile, hexamethylenediamine, ammonia, adiponitrile and hexamethyleneimine,
- 15 (b) the mixture obtained in (a) is subjected to a distillation to give ammonia as the top product and a bottom product I, the distillation being carried out at a bottom temperature of from 60 to 220°C and a pressure of from 10 to 30 bar in the presence of a compound A which is inert under the distillation conditions and boils at from 60 to 220°C at 18 bar, and the ammonia not being completely separated off,
- 20 (c) the bottom product I containing essentially 6-aminocapronitrile, hexamethylenediamine, adiponitrile, hexamethyleneimine, inert compound A and ammonia, the ammonia content being lower than that of the mixture used in stage (b), is
- 25 subjected to a second distillation to give a mixture comprising the inert compound A and ammonia as the top product and a bottom product II, the distillation being carried out at a bottom temperature of from 100 to 220°C and from 2 to 15 bar, with the proviso that the pressures in the first and
- 30 second columns are matched with one another so that a top temperature above 20°C is obtained at a bottom temperature of not more than 220°C in each case,
- 35 (d) the bottom product II containing essentially 6-aminocapronitrile, hexamethylenediamine, adiponitrile, hexamethyleneimine and inert compound A is subjected, in a third column, to a distillation to give the inert compound A as the top product and a bottom product III, the distillation being
- 40 carried out at a bottom temperature of from 100 to 220°C and from 0.1 to 2 bar, with the proviso that the inert compound A obtained as the top product is fed to the second column, and, if desired, the distillation being carried out in the presence of a compound B which is inert under the distillation conditions and boils at from 50 to 220°C at 0.3 bar,
- 45

- (e) the bottom product III containing essentially 6-aminocapronitrile, hexamethylenediamine, adiponitrile, hexamethyleneimine and, if desired, an inert compound B is subjected, in a fourth column, to a distillation to give a top product KP1, containing essentially hexamethyleneimine, if desired inert compound B and hexamethylenediamine, which is obtained at a bottom temperature of from 100 to 220°C and from 10 to 500 mbar, and a bottom product IV,
- (f) the top product KP1 is subjected, in a fifth column, to a distillation to give a top product KP2, which contains essentially hexamethyleneimine and, if desired, inert compound B and is obtained at a bottom temperature of from 100 to 220°C and from 50 to 2000 mbar, and a bottom product V containing essentially hexamethylenediamine in a purity of at least 95%, the top product KP2 being fed to the third column or, if desired, only some of the said top product being fed to the third column and the remainder being discharged, and
- (g) the bottom product IV containing essentially 6-aminocapronitrile and adiponitrile is subjected, in a sixth column, to a distillation to give 6-aminocapronitrile in a purity of at least 95% as a top product and adiponitrile as the bottom product, the distillation being carried out at a bottom temperature of from 100 to 220°C and from 1 to 500 mbar,

and the 6-aminocapronitrile thus obtained is then cyclized to give caprolactam.

30

35

40

45