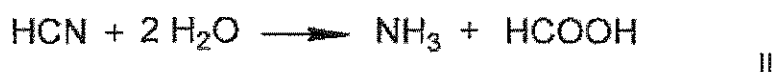


hydrolysis) can be operated continuously and integrated successively in a fully continuous process. How a column for hydrolysis of Met-hydantoin should be designed technically to minimize by-product formation has not yet been described in the prior art. Furthermore, there is no description of how the NH_3 circuit between hydantoin formation and hydantoin hydrolysis is to be operated loss-free at minimized energy expenditure. The hydrolysis process can be carried out in a steam-heated column, wherein the 5-(2-methylmercaptoethyl)-hydantoin solution is advantageously fed continuously to the top of the column at a rate such that the hydrolysis product, potassium methioninate solution, can accordingly be withdrawn at the bottom of the column after quantitative hydrolysis. The mother liquor can be reused after separating the methionine solids. The gaseous constituents (steam, ammonia and carbon dioxide) can be discharged at the top of the column and can be used for restoration of the aqueous ammonia/carbon dioxide solution for production of 5-(2-methylmercaptoethyl)-hydantoin. It is described as being especially advantageous, for avoiding by-products, to carry out the hydrolysis right at the start in the presence of alkali and carbon dioxide, i.e. in particular there is a mixture of alkali compounds, in particular alkali hydrogen carbonate, alkali carbonate, alkali hydroxide. To achieve complete conversion of the valuable starting material MMP to hydantoin, a molar ratio of 1.005 – 1.02 mol/mol HCN/MMP is employed in the method described. As a result of hydrolysis of unreacted hydrogen cyanide, in the column there is production of NH_3 by the following reaction (formula II):



The resultant formic acid is located as non-volatile potassium formate at the bottom of the reaction column; it thus displaces the required alkaline carbonates and therefore disturbs the alkaline potassium circuit.

The problem to be solved by the ~~disclosure~~invention was therefore to provide an improved method, relative to the prior art, for producing a methionine salt starting from the raw materials methylmercaptopropionaldehyde and hydrogen cyanide or starting from at least one compound that can be prepared from these raw materials, such as methylmercaptopropionaldehyde-cyanohydrin.

The problem is solved by the reaction system according to the ~~disclosure~~invention, use thereof according to the ~~disclosure~~invention and the method according to the ~~disclosure~~invention.

The reaction system has the following features:

1. Reaction system for production of a methionine salt, comprising a reactive-rectification column with a weir height of 100 mm or more
2. Reaction system according to 1, wherein
the weir height is in the range from 100 to 1000 mm, preferably in the range from 150 to 700 mm,
the plate spacing is in the range from 500 to 1000 mm,
the ratio of column diameter to weir length is in the range from 1.1 to 1.3,
the ratio of the cross-sectional area to the area through which gas flows is in the range from 1.5 to 2 and
the number of plates is in the range from 15 to 25, preferably in the range from 18 to 20.
3. Reaction system according to 1 or 2, wherein the reactive-rectification column is a sieve plate column, perforated plate column, valve plate column or bubble plate column.
4. Reaction system according to 3, wherein, in the reactive-rectification column
the ratio total area of all holes / area through which gas flows is in the range from 0.04 to 0.08 and
the diameter of the individual holes in the sieve plate is in the range from 5 to 10 mm.
5. Reaction system according to one of the above numbers, wherein the weir heights ensure an average residence time of the respective mixture of less than 0.5 min per plate.
6. Reaction system, wherein the reaction system further comprises at least one reactive absorber and optionally a second reactor for the production of 5-(2-methylmercaptoethyl)-hydantoin.

7. Reaction system according to 5, wherein the reactive absorber is a jet washer system.
8. Reaction system according to one of the numbers 1 to 7, wherein zirconium is used in the reactive-rectification column and/or in the reactive absorber and/or in the second reactor as material for parts in contact with the product.

In one aspect, there is provided a reaction system for production of a methionine salt, comprising a reactive-rectification column with a weir height in the range from 100 to 1000 mm wherein the plate spacing is in the range from 500 to 1000 mm, the ratio of column diameter to weir length is in the range from 1.1 to 1.3, the ratio of the cross-sectional area to the area through which gas flows is in the range from 1.5 to 2 and the number of plates is in the range from 15 to 25, preferably in the range from 18 to 20.

The method has the following features:

1. Method for continuous production of a methionine salt, wherein the following steps are carried out:
 - reaction of 3-methylmercaptopropionaldehyde and hydrogen cyanide or of a component that can be produced therefrom, wherein a solution containing 5-(2-methylmercaptoethyl)-hydantoin is obtained;
 - alkaline hydrolysis of the 5-(2-methylmercaptoethyl)-hydantoin obtained to a methionine salt in a reactive-rectification column, wherein only the solution containing 5-(2-methylmercaptoethyl)-hydantoin is fed on the topmost plate of the reactive-rectification column and an alkaline circulating solution is fed on a plate under that, preferably on the 2nd plate from the top.
2. Method according to 1, wherein the alkaline circulating solution contains an alkali carbonate, preferably potassium carbonate.
3. Method according to 1 or 2, wherein water, ammonia and CO₂ are removed from the top of the reactive-rectification column and the NH₃ removed is condensed completely or partially for use in the synthesis of 5-(2-methylmercaptoethyl)-hydantoin.

4. Method according to one of the numbers 1 to 3, wherein the concentration of ammonia in the bottom of the rectification column is less than 120 ppm, preferably less than 100 ppm and most preferably less than 80 ppm.
5. Method according to one of the numbers 1 to 4, wherein the conversion to 5-(2-methylmercaptoethyl)-hydantoin is carried out in a reactive absorber

and then in a second reactor, preferably in a second reactor designed as a flow tube.

6. Method according to one of the numbers 1 to 5, wherein the temperature of the reaction mixture at the outlet of the reactive-rectification column is in the range from 180°C to 190°C.
7. Method according to one of the numbers 1 to 6, wherein the temperature of the gas phase at the top of the reactive-rectification column is in the range from 160°C to 170°C.
8. Method according to one of the above numbers 1 to 7, wherein alkaline hydrolysis is carried out at a pressure in the range from 8 bar(excess) to 10 bar(excess).
9. Method according to one of the numbers 1 to 8, wherein steam is used as heating and stripping medium in the reactive-rectification column.
10. Method according to one of the numbers 1 to 9, wherein the method is carried out in a reaction system according to one of the numbers 1 to 8.

In another aspect, there is provided a method for continuous production of an methionine salt, wherein the following steps are carried out: reaction of 3-methylmercaptopropionaldehyde and hydrogen cyanide or of a component that can be produced therefrom, wherein a solution containing 5-(2-methylmercaptoethyl)-hydantoin is obtained; alkaline hydrolysis of the 5-(2-methylmercaptoethyl)-hydantoin obtained to a methionine salt in a reactive-rectification column as defined above, wherein only the solution containing 5-(2-methylmercaptoethyl)-hydantoin is fed on the topmost plate of the reactive-rectification column and an alkaline circulating solution is fed on a plate located under that, preferably on the 2nd plate from the top.

In another aspect, there is provided~~The invention also relates to the use of a~~ reaction system as defined above for the production of methionine.

Reference is made expressly to the preferred embodiments of the disclosure~~invention~~ presented in the subclaims.

The ~~disclosure~~^{invention} relates to a method for continuous production of a methionine salt, wherein 3-methylmercaptopropionaldehyde and hydrogen cyanide (HCN) or a component that can be produced therefrom are reacted, wherein a solution containing 5-(2-methylmercaptoethyl)-hydantoin is obtained and wherein an alkaline hydrolysis of the 5-(2-methylmercaptoethyl)-hydantoin obtained to a methionine salt is carried out in a reactive-rectification column, wherein only the solution containing 5-(2-methylmercaptoethyl)-hydantoin is fed on the topmost plate of the reactive-rectification column and an alkaline circulating solution is fed on a plate located under that, preferably

Patent claims

1. Reaction system for production of a methionine salt, comprising a reactive-rectification column with a weir height in the range from 100 to 1000 mm
5 wherein
the plate spacing is in the range from 500 to 1000 mm,
the ratio of column diameter to weir length is in the range from 1.1 to 1.3,
the ratio of the cross-sectional area to the area through which gas flows is
in the range from 1.5 to 2 and
10 the number of plates is in the range from 15 to 25, preferably in the range
from 18 to 20.
2. Reaction system according to Claim 1, wherein the weir height is in the
range from 150 to 700 mm.
15
3. Reaction system according to Claim 1 or 2, wherein the reactive-rectification column is a sieve plate column, perforated plate column, valve plate column or bubble plate column.
- 20 4. Reaction system according to Claim 3, wherein in the reactive-rectification column
the ratio of total area of all holes / area through which gas flows is in the
range from 0.04 to 0.08 and
the diameter of the individual holes in the sieve plate is in the range from 5
25 to 10 mm.
5. Reaction system according to one of the preceding claims, wherein the weir heights ensure an average residence time of the respective mixture of
less than 0.5 min per plate.
30
6. Reaction system, wherein the reaction system further comprises at least one reactive absorber and optionally a second reactor for the production of
5-(2-methylmercaptoethyl)-hydantoin.
- 35 7. Reaction system according to Claim 5, wherein the reactive absorber is a jet washer system.

8. Reaction system according to one of Claims 1 to 7, wherein zirconium is used in the reactive-rectification column and/or in the reactive absorber and/or in the second reactor as material for parts in contact with the product.
9. Method for continuous production of a methionine salt, wherein the following steps are carried out:
- reaction of 3-methylmercaptopropionaldehyde and hydrogen cyanide or of a component that can be produced therefrom, wherein a solution containing 5-(2-methylmercaptoethyl)-hydantoin is obtained;
 - alkaline hydrolysis of the 5-(2-methylmercaptoethyl)-hydantoin obtained to a methionine salt in a reactive-rectification column according to one of Claims 1-8, wherein only the solution containing 5-(2-methylmercaptoethyl)-hydantoin is fed on the topmost plate of the reactive-rectification column and an alkaline circulating solution is fed on a plate located under that, preferably on the 2nd plate from the top.
10. Method according to Claim 9, wherein the alkaline circulating solution contains an alkali carbonate, preferably potassium carbonate.
11. Method according to Claim 9 or 10, wherein water, ammonia and CO₂ are removed from the top of the reactive-rectification column and the NH₃ removed is condensed completely or partially and used in the synthesis of 5-(2-methylmercaptoethyl)-hydantoin.
12. Method according to one of Claims 9 to 11, wherein the concentration of ammonia in the bottom of the rectification column is less than 120 ppm, preferably less than 100 ppm and most preferably less than 80 ppm.
13. Method according to one of Claims 9 to 12, wherein the reaction to 5-(2-methylmercaptoethyl)-hydantoin is carried out in a reactive absorber and then in a second reactor, preferably in a second reactor designed as a flow tube.

14. Method according to one of Claims 9 to 13, wherein the temperature of the reaction mixture at the outlet of the reactive-rectification column is in the range from 180°C to 190°C.
- 5 15. Method according to one of Claims 9 to 14, wherein the temperature of the gas phase at the top of the reactive-rectification column is in the range from 160°C to 170°C.
- 10 16. Method according to one of Claims 9 to 15, wherein alkaline hydrolysis is carried out at a pressure in the range from 8 bar(excess) to 10 bar(excess).
- 15 17. Method according to one of Claims 9 to 16, wherein steam is used as heating and stripping medium in the reactive-rectification column.
18. Method according to one of Claims 9 to 17, wherein the method is carried out in a reaction system according to one of Claims 1 to 8.
- 20 19. Use of a reaction system according to one of Claims 1 to 8 for the production of methionine.