

Title (en)

METHOD FOR THE SYNTHESIS OF 2,6-BIS (METHIONYL) -1,4-DIKETOPIPERAZINE

Title (de)

VEREINFACHTES UND SKALIERFÄHIGES VERFAHREN ZUR SYNTHESE VON 2,6-BIS(METHIONYL)-1,4-DIKETOPIPERAZIN

Title (fr)

PROCEDE DESTINE A LA SYNTHESE DE 2,6-BIS (METHIONYL)-1,4-DICETOPIPERAZINE

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Application

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Abstract (en)

[origin: WO2017005547A1] The invention relates to a method for producing 2,6-bis(methionyl)-1,4-diketopiperazine comprising the following steps: a) heating a reaction mixture containing methionine and a polar protic solvent at a temperature of 145.0 to 169.5 °C, wherein an inert gas flow is directed over or through the reaction mixture; b) obtaining 2,6-bis(methionyl)-1,4-diketopiperazine. The invention further relates to a method for isolating 2,6-bis(methionyl)-1,4-diketopiperazine from a reaction mixture, comprising the following steps: A1) diluting the reaction mixture containing 2,6-bis(methionyl)-1,4-diketopiperazine using a polar solvent and water, wherein 2,6-Bis(methionyl)-1,4-diketopiperazine crystallises; or A2) diluting the reaction mixture containing 2,6-bis(methionyl)-1,4-diketopiperazine using water only, without prior diluting with an organic solvent and with or without pressure control, wherein 2,6-bis(methionyl)-1,4-diketopiperazine crystallises; or A3) cooling the reaction mixture, or allowing same to cool, without the addition of substances until a temperature is reached, in particular of 135.0 to less than 145.0 °C, preferably 138.0 to 142.0 °C, at which the diketopiperazine begins to crystallise, and finally diluting by the addition of water and resultant cooling, wherein 2,6-bis(methionyl)-1,4-diketopiperazine crystallises; B) separating the crystallised 2,6-bis(methionyl)-1,4-diketopiperazine obtained as a solid in A1) or A2) or A3) and washing with a solvent, in particular ethylene glycol, acetone or methanol, and finally washing with water. The present invention relates in particular to a combination of a method for producing 2,6-bis(methionyl)-1,4-diketopiperazine and a method for isolating 2,6-bis(methionyl)-1,4-diketopiperazine from a reaction mixture.

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