Title (en)

METHOD OF PREPARATION OF 4,1',6'-TRICHLORO-4,1',6'-TRIDEOXYGALACTOSUCROSE

Title (de)

VERFAHREN ZUR HERSTELLUNG VON 4,1',6'-TRICHLOR-4,1',6'-TRIDEOXYGALACTOSACCHAROSE

Title (fr)

PROCEDE DE PREPARATION DE 4,1',6'-TRICHLORO-4,1',6'-TRIDESOXYGALACTOSACCHAROSE

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Application

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Abstract (en)

[origin: WO9960006A1] The invention relates to the method of preparation 4,1',6'-trichloro-4,1',6'-trideoxygalactosucrose which can be used as a sugar ingredient (sweetener) of increased sweetness particularly in food and pharmaceutical industries. The most widely used sweetening agent is a natural carbohydrate known as sucrose. In accordance with the invention there is described a method of preparation of 4,1',6'-Trichloro-4,1',6'-trideoxygalactosucrose with sucrose as the base material consisting in selective acetylation of sucrose in pyridine, chlorination of monoacetylated product in Vilsmeier reagent, peracylation of the chloroderivative in pyridine with the deesterification and isolation of the desired product to follow. It has been found that the most preferred conditions for the acetylation stage are the following: the weight of the pyridine excess being 10- times as that of sucrose, conducting the reaction at room temperature and recurring back to the pyridine process with the pyridine removed from the reaction zone by an off-distillation with freezing. The preferred condition for preparation of Vilsmeier reagent is the reaction of phosphorus pentachloride with dimethylformamide (DMF) at the temperature of 0-25 DEG C in the absence of moisture. Chlorination is preferably conducted at gradual addition of sucrose 6- monoacetate solution in DMF to the prepared solution of Vilsmeier reagent, the temperature being maintained below 20 DEG C with the following heating of the reaction to 108-110 DEG C for min. 1,5 hours. Chlorination is conducted for the mixture of products received at the stage of direct acetylation without chromatographic separation of the mixture on ion- exchange resin. Peracylation is conducted by acetic anhydride in pyridine preferably at the temperature of 50 DEG C for 1,5-2,5 hours. Deacylation is provided by sodium methoxide in alcohol at room temperature for min. four hours at PH 8,8-9,0 with the following neutralization of the reaction by the ion- exchange resin in H-<+>. The total yield is 60 % at 85 % purity. The advantages of the method filed are provided due the reduction of synthesis stages and simplification of their conducting, considerable increase of the desired (end) product, fast and reliable control of all the stages of technological process by spectral analysis methods.

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